Vlaamse Regering

Ministerial Decree approving the compendium of sampling and analysis methods under the Fertiliser Decree (BAM)

Legal grounds

This decision is based on:
— the Decree of 5 April 1995 laying down general provisions on environmental policy, Article 5.6.2, inserted by the Decree of 25 April 2014, 5.6.5, inserted by the Decree of 25 April 2014 and amended by the Decree of 8 December 2017;
— the Fertiliser Decree of 22 December 2006, Article 61 (8), inserted by the Decree of 12 June 2015;
— Article 45 (1) of VLAREL of 19 November 2010, replaced by the Flemish Government Decree of 1 March 2013 and amended by the Flemish Government Decree of 3 May 2019 and 6 December 2024, 49, amended by the Flemish Government Decrees of 1 March 2013, 3 May 2019 and 21 May 2021, 53/1, inserted by the Flemish Government Decree of 1 March 2013 and amended by the Flemish Government Decrees of 11 December 2015, 3 May 2019 and 21 May 2021, 58/4, inserted by the Flemish Government Decree of 3 May 2019 and amended by the Flemish Government Decrees of 21 May 2021 and 31 January 2025, 58/5, inserted by the Flemish Government Decree of 3 May 2019 and amended by the Flemish Government Decree of 31 January 2025.

Formal requirements

The following formal requirements are met:
— The Flemish Supervisory Commission for the Processing of Personal Data issued Opinion No XXXX/XXX on DATE;
— The Council of State gave its opinion XXXXX/XXX on DATE, pursuant to Article 84 (1), first subparagraph, point 2, of the laws on the Council of State, coordinated on 12 January 1973;
— This draft was notified to the European Commission on DATE, pursuant to 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 Mestbank made a proposal to amend the compendium of sampling and analysis methods under the Fertiliser Decree (BAM) on DATE.

THE FLEMISH MINISTER FOR THE ENVIRONMENT AND AGRICULTURE HEREBY DECIDES:

Article 1. The compendium of sampling and analysis methods under the Fertiliser Decree (BAM), and the corresponding table of contents, as set out in the Annex to this Decision, are approved.

Article 2. The Ministerial Decree of 16 July 2021 approving the compendium of sampling and analysis methods under the Fertiliser Decree (BAM) is hereby repealed.

Article 3. The compendium referred to in Article 1 shall enter into force on DATE.

Brussels,... (date).

The Flemish Minister for Environment and Agriculture,

JO Brouns

Annex to the Ministerial Decree of.... 2026 approving the compendium of sampling and analysis methods under the Fertiliser Decree (BAM)

Annex – Compendium of methods of sampling and analysis for manure, soil and feed (BAM)

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the Fertiliser Decree

Soil – Scope

The methods relate to the sampling and analysis of agricultural parcels as provided for in the Decree of 22 December 2006 concerning the protection of waters against pollution caused by nitrates from agricultural sources (hereinafter referred to as the Fertiliser Decree) and its implementing decrees with a view to:

- Determination of the nitrate residue
- Determination of ammoniacal and nitric nitrogen and carbon content in the context of a nitrogen fertilisation opinion
- Determination of the amount of plant-available phosphate for the purpose of determining the phosphate class
- Determination of phosphate saturation
- The determination of the texture for texture class change

The Executive Laboratory shall ensure that sampling and analysis is always carried out according to the methodology described below and shall be responsible for it.

Soil – Sampling

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1 PURPOSE AND SCOPE

This procedure describes the sampling of soil on agricultural parcels for the uses listed in BAM/part 1/00. The resulting laboratory sample shall be representative of the intended purpose over the entire field for the parameters to be analysed.

The implementing laboratory is responsible for the correct execution of the sampling.

2 SAMPLING STRATEGY

The sampling strategy to be applied depends on the purpose of sampling:

- A post-stratification method is used to determine the nitrate residue.
- For the determination for other purposes, samples may be taken from a grid.

2.1 SAMPLING AFTER STRATIFICATION

The points to be sampled on the parcel are provided via SMIL. These sampling points are determined by first dividing the plot into a fixed number of compact strata and then selecting one sampling point per stratum. In order to facilitate implementation on the ground, the shortest route between sampling points is also calculated. Each sampling point shall be numbered sequentially according to this shortest line.
The number of points to be sampled shall depend on the crop distinguishing between 'broad-sown crops' for which no effects of the seed or planting distance are expected on the distribution of nutrients in the soil and 'rows of crops' [1] for which this is the case.

- For broad-sown crops, 40 individual sampling points shall be selected.
- For crops in rows, 20 sampling points shall be selected and drilling shall be carried out at each point l2 along the transect perpendicular to the crop trij in accordance with the specified label (see 4.2). This divides the stitches between collecting the variance on the parcel and the variance on a transverse line in the direction of travel. Even when the crop has already been harvested, the stratification procedure for growing in rows is applied provided that the crops are still undisturbed and clearly visible (e.g. maize stubble).

Exceptions are provided for those parcels on which there is still a crop which makes it very difficult to navigate freely on the parcel (see 4.3.2).

[1] row crops are defined as those where the spacing between rows is at least ten times the diameter of the guts used. For example, although cereals are sown in rows, they are sampled as broad-sown crops, although here too the plants are mostly in rows.

2.2 Sampling by grid

The locations to be sampled are selected (visibly) by the sampler on a grid where the spacing between sampling points is determined by the size and shape of the plot:

- By default, at least 24 points in a 4-row grid x 6 sampling points shall be sampled over the entire plot. For long narrow plots, 3 rows x 8 sampling points may be used. 2 to 3 rows shall be crossed at each sampling point alternately to the left and to the right of the selected walking line. The walkways may be on the gridlines or in a double zigzag.
- Sampling points along the edges of the plot shall be taken no more than 10 metres from the side of the plot.
- There is no difference between wide-ranging crops or crops in rows.

In the case of samples taken as part of the preparation of a nitrogen fertilisation opinion, the sampling may be limited to the part of the parcel for which the opinion is intended. The sampled portion must then be clearly identified in the sampling report (see also 5.2 for documentation of sampling). The size of the sampled section does not affect the minimum number of drilling bits required.

2.3 Parcel area

The number of points to be sampled as provided for in points 2.1 and 2.2 shall apply for parcels with an area of 5 ha or less. Lines larger than 5 ha shall be divided into sub-parcels. The number of points to be sampled as set out in points 2.1 and 2.2 applies per 5 ha disc started.

Sub-parcels are always analysed and reported separately, no composite samples are taken.

For the determination of both nitrate residue and P-Al, in addition to the results of the sub-parcels, the average value per parcel is also reported. In these cases, care must be taken to ensure that both sub-parcels have the same area when sampling.

2.4 Sampling depth

The sampling depth depends on the purposes for which sampling is carried out:

- For the determination of nitrate in the context of a nitrate residue determination, sampling points with odd sequential numbers shall be sampled up to 90 cm in 3 layers up to 30 cm, 60 cm and 90 cm respectively; sampling points with even sequential numbers shall be sampled up to 60 cm in 2 layers up to 30 cm and 60 cm respectively.
- The phosphate saturation rate is always sampled to 90 cm in 3 layers to 30, 60 and 90 cm respectively.

- For the determination of P-Al, C and pH, sampling shall be carried out up to a depth of 30 cm. On arable land, the sampling depth may be limited to 23 cm; for grassland [2], the sampling depth may be limited to 6 cm depth. Sampling in the context of an erosion class reduction carried out on arable land that has been uncultivated for at least 5 years may be

carried out up to a depth of 10 cm provided that it is indicated in the report.Sampling for the determination of nitric and ammoniacal nitrogen carried out in the context of formulating a nitrogen fertilisation opinion shall be carried out up to a depth as indicated in Table 1.

	Cultivation to which the fertilisation advice relates	Sampling depth (cm)
1	Strawberries, basil, chives, chrysanthemums, courgettes, iceberg lettuces, herbs, paksoi, parsley, radish, rocket, lettuce, cut flowers, cut plants, spinach, lamb's lettuce, early leaf vegetables, early onions or winter flowering semi-bushes.	30
2	Potatoes Vegetables of Group I, Group II or Group III, other than those mentioned in 1 Grassland	60
3	Crops other than those mentioned in 1 or 2	60 or 90 depending on the rooting depth

Table 1: sampling depth for the determination of nitric and ammoniacal nitrogen carried out in the context of formulating a nitrogen fertilisation opinion

3 MATERIAL

a. A gut type earth boron with an inside diameter of at least 13 mm and an effective length of 6, 23, 30, 60 or 90 cm, as appropriate, with an indication per 10 cm. If samples are taken at specific depths, e.g. 23 cm, they must also be permanently marked. For sampling deeper than 30 cm or more, the guts must be fitted with a detonating head to allow the use of a hammer.
Mechanical sampling systems are permitted provided that a gut boron of minimum diameter as mentioned above is used.
b. If samples are taken for the determination of ammoniacal or nitric nitrogen, plastic bags of sufficient size and strength shall be provided and be capable of being sealed. For the determination of carbon, pH, plant-available phosphorus and phosphate saturation, samples may also be collected in other appropriate containers.
c. Hammer suitable for floating the guts in the ground. A returnless hammer is recommended.
d. A GPS module or application (smartphone, tablet, etc.) as referred to in BAM deel8/04.
e. A sharp, flat tool to scrape off the guts (palletmes type).
f. Supplies for refrigerated storage of the samples taken. The capacity must be sufficient for all samples to be taken before disposal at the lab or collection point. When a cooling box with cooling elements is used, at least 10 % of the volume of the box shall be provided with cooling elements. The cooling elements are stored in the freezer at -18 °C prior to use.

[2] permanent grassland with grass as the only crop in the year of sampling

Figure 1 Ground boron type guts

4 PRACTICAL IMPLEMENTATION

In case of stratification sampling, sampling shall be carried out by sampling points provided by SMIL one by one based on in situ GPS measurements. An example of a plot showing sampling points, calculated walking line and GPS track at sampling is shown in Figure 2.
When sampling in a grid, the sticks to be taken (on sight) are distributed over the entire parcel.

All sub-signs shall be collected in a separate container per layer sampled (and if applicable per sub-plot on a plot larger than 5 ha).

Shaking stations, access to the parcel, drinking points, surroundings of headfield storage, etc. are not avoided during sampling. Only when sampling is carried out as part of the preparation of a nitrogen fertilisation opinion may non-relevant zones be avoided. However, this does not change the sign to be taken and these areas must be indicated with reason (indicated) on the sampling report.

Figure 2: plot with sampling points and running line when sampling with stratification (left) and sampling points on a grid (right)

4.1 GENERAL – IMPLEMENTATION

Pitch drilling shall be carried out at the sampling points as described above. The ground is tightened slightly permanently on site at and around the site where the drilling will take place. Push the boron into the ground at right angles to the mowing field until the guts are fully filled. The gut boron shall be rotated at least 1 times fully to release the boron and then slowly raised. It is important that there is no loss of land. Using the spatula, the cut is scraped off, only the soil in the drilling body belongs to sample [3.] The soil is then moved out of the boron into the collection container.

Where, in the case of deeper sampling, multiple entries are made into the same borehole, the inclusion of soil from the side of the borehole in the sample shall be avoided. For this purpose, the top 2 centimetres of soil in the guts are removed and not added to the sample. It is permitted to use a different diameter for the different horizons provided that the same drilling diameter (s) are used over the entire plot.

During sampling, a GPS log shall be made in accordance with the provision of BAM part 8/04.

4.2 SAMPLING WITH STRATIFICATION OF CROPS IN ROWS

In case of sampling with stratification of crops in rows, in addition to the coordinate of the sampling point itself, a letter code of A, B or C shall be provided, referring to the places where drilling must take place along a transect perpendicular to the crops. The transect always starts between the plants of one row and runs to just before the plants of the next row.
Keep in mind, divide the line linking two rows of crops into two parts and each half into 3 equal parts. In each of the halves, the first location is labelled ‘A’, the second ‘B’ and the third ‘C’. The site is always crossed twice by SMIL, the distance between the two stings being equal to half the distance between the crops. See an example (Figure 3) for the case where location ‘A’ or ‘B’ was indicated in SMIL.

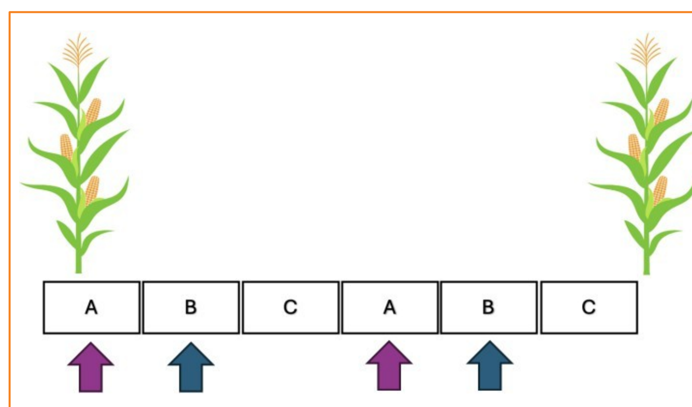

Figure 3: example for drilling sites “B” (blue arrows) and “A” (purple arrows).

3 in the case of very clayey or sandy soils, where some of the contents of the guts are often lost when scraped off, this operation may be omitted.

4.3 Sampling problems, deviations from the method

4.3.1 Places or depths not sampled

Where sampling cannot be carried out at all points or cannot be carried out at all appropriate depths, the following guidelines shall apply:

- Where sampling at a sampling point is not possible, e.g. due to the presence of welds, the point shall be replaced by choosing a location as close as possible to the specified sampling point.

- Where a contiguous zone (= three or more adjacent points) of a plot cannot be sampled, sampling points located in the zone shall not be replaced, up to a maximum of one quarter of the sampling points:
    - 10/40 points in the case of wide-ranging crops, if samples are taken using stratification.
    - 5/20 points for crops grown in rows in the case of stratification sampling.
    - 6/24 points in the case of sampling according to a grid.

  Where more than this number of points cannot be sampled, no sample representative of the entire plot can be taken. No (further) sampling is carried out.

- Where sampling cannot be carried out to the required depth, sampling at that point shall be limited to the achievable depth and the following shall apply:
    - A layer is accepted once half or more of the required stitch could be taken up to at least part of the required depth. When less stabling was possible, the field sample is removed.
    - Where it has not been possible to sample all the drilling operations to the required depth, the number of buds carried out and the average depth estimated to the nearest 10 cm must be indicated on the sampling report.

- Where it is a priori decided not to sample to the required depth due to concerns of the farmer for damage to drainage tubes, the sample after-median may be adjusted to the depth of the (drainage) tubes. This is always reported to VLM via SMIL and explicitly mentioned on the sampling report, regardless of whether the farmer has notified VLM in advance.

4.3.2 Presence of crops

Sampling by stratification can be seriously complicated if there is a crop in rows on the parcel that prevents the parcel from navigating freely. For example, in the case of maize, sprouts or potatoes, it is almost impossible to cross the plants if the crop is still present. In such cases, sampling may be carried out using an alternative method as described below:

- This method is an alternative to stratified sampling.
- The presence of the crop must be proven by a GPS localised photograph of the parcel.
- An orthogonal grid with 20 sampling points shall be used, with two drilling stitches taken at each point along the transverse line of the crop, cf. §4.2. For the location of these two drilling signs, the (a), (b) and (c) positions on the transect shall be sampled alternately as indicated in §4.2.
- Choose four rows proportionally distributed over the parcel along which the parcel can be crossed.

- Five points shall be selected for sampling over the length of each row, divided proportionally over the length.
- At these points, two to three crops are drunk alternately to the left and to the right. The two random samples are then taken at this location. See Figure 4.

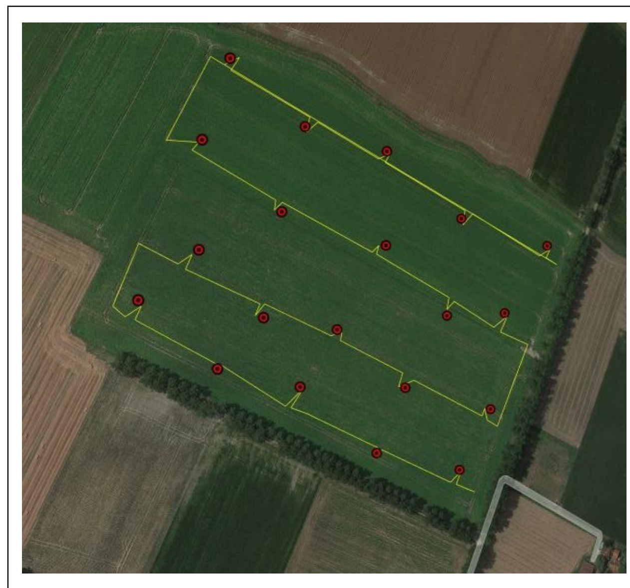

Figure 4: sampling pathway for the alternative method of sampling with stratification in the presence of degrading crops.

5 REPORTING OF SAMPLING, IDENTIFICATION OF SAMPLES

5.1 IDENTIFICATION OF SAMPLES

Each container shall bear a unique number, coding or other reference allowing a unique link to the sampling data and the SMIL steel sample number. It shall be affixed to the container in such a way as to be resistant to water and to allow samples to be frozen and thawed, and shall also be affixed to the sampling form.

The laboratory's sample management system must allow any information relating to an individual sample to be traced unambiguously afterwards.

5.2 REPORTING OF SAMPLING

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be recorded and reported:

(1) The SMIL steel sample number.
(2) Identification of the sampler (e.g. initials, identification code, SMIL steel nemer number).
(3) Date and time of start of sampling (SMIL start notification).
(4) The sampling protocol followed: with stratification or grid as a derogation from stratification in the presence of an impeding crop in nitrate residue determination (via SMIL start notification) or grid in other parameters.

(5) The depth (s) at which sampling was carried out.
(6) Where it has not been possible to sample all points to the desired depth, the number of drilling buds sampled and the average depth shall be accurate to 10 cm.
(7) Any deviations from the method, e.g. when only part of the plot could be sampled.
(8) The possible presence of drainage tubes and their depth.

The information may be on paper or digital medium or a combination thereof. If exceptional

circumstances arise, this should be indicated.

6 MONSTERCONSERVATION

In order to minimise the loss of ammoniacal N or microbiological conversions, in the case of determination of ammoniacal nitrogen and nitric nitrogen:

- Prevent an increase in the temperature of the sample. Therefore, immediately after sampling, pending and during transport to the sample storage place or laboratory, the sample should be kept refrigerated in a refrigerated box.
  It goes without saying that the cooling capacity of equipment taken into account during sampling (refrigeration boxes or refrigeration inside the wagon) cannot be sufficient to cool all soil samples to 3 ± 2 °C within a limited time interval. Therefore, the temperature of the samples should not be checked or secured by a measurement. However, the following minimum requirements are set for the equipment present at the time of sampling and after sampling:
  - Samples should be transferred immediately after sampling to a refrigerated box (or to an ice box if present in the vehicle). Only fixed cooling boxes (rigid plastic, piepfoam) are allowed, thermostatic bags may not be used.
  - The cooling boxes must be cooled with frozen cooling elements. This means that the cooling elements are stored in the freezer at -18 °C before use and for a sufficient period of time to ensure that they are completely frozen before use. The number of cooling elements used shall be at least 10 % of the volume of the cooling box.
  - If electrically cooled boxes are used, it shall be possible to prove that they are working properly; they shall be switched on at departure so that they are sufficiently cold when the first samples are placed in them.
- The sample is stored at a temperature of 3 ± 2 °C upon arrival at the laboratory or sample store. This temperature must be detectable when stored at the laboratory or sample store or when transported from a sample store to the laboratory.
- The analysis must begin within 72 hours of sampling. Prior to sampling, the necessary arrangements must be made with the laboratory to ensure this.
- If analysis cannot start within 72 hours, the storage period may be extended by freezing. The entire sample must then be frozen at a minimum of -24 °C within 18 hours of sampling.

For the determination of carbon, plant-available phosphorus and phosphate saturation, storage at room temperature is sufficient.

7 QUALITY CHECK

Both the sampler and the laboratory are responsible for the correct application of sampling. The laboratory must carry out an adequate check on this, which must include at least the following checks:

- Second-line check (re-sampling of parcels)
- Evaluation of GPS tracks
- Evaluation of sampling reporting
- Evaluation of species and prevention of deviations from the method.

Soil – Sample pre-treatment

1 PRINCIPLE

The preservation and preparation of soil samples is of great importance. It aims to minimise losses due to volatilisation (ammonia) or conversions.

For the determination of ammoniacal nitrogen and nitric nitrogen, the sample shall not be dried because it may give rise to losses due to volatilisation and conversions and the determination shall be carried out on field moist soil. Taking a representative sub-sample will be critical.

For the determination of pH, organic carbon, plant-available phosphorus and phosphate saturation, the sample should be dried under controlled conditions.

2 SOIL SAMPLE STORAGE FOR ANALYSIS

a. To avoid conversions in the soil samples, in case of ammoniacal nitrogen and nitrate nitrogen determination, they should always be kept cool (at a temperature of maximum 3 ± 2 °C).

b. In the case of ammoniacal nitrogen and nitrate nitrogen determination, the soil sample must be prepared for analysis no later than 72 hours after sampling. If this is not possible, the entire soil sample must be deep-frozen immediately (within 24 hours) and without undergoing any further processing at a temperature of at least -18 °C until it can be taken into processing. The frozen sample should be thawed at 3 ± 2 °C prior to analysis and the analysis should start the day after thawing has started. This must be demonstrable.

c. In the case of pH, organic carbon, plant-available phosphorus and phosphate saturation, samples may be stored in the laboratory at room temperature for a maximum of 5 days before processing.

Unlike the cooling boxes used on site, the temperature of the installations used for the storage of steel must be checked in accordance with normal practice under ISO 17025, as stated in BAM. This applies not only to the cold rooms or cabinets in the laboratories but also to the installations in the sample collection points when the samples are not taken directly to the laboratory by the steelmaker. Refrigerated transport from these assembly points to the laboratory must also be under temperature control, this must be traceable.

3 PREPARATION FOR DETERMINATION OF AMMONIACAL AND NITRIC NITROGEN ON VELDVOCHTIGE SOIL

The determination of ammoniacal nitrogen and nitric nitrogen shall be carried out on field moist soil. To this end, the entire soil sample (either moist in the field or thawed after deep-freezing) is homogenised.

In the case of frozen soil samples, the defrosting process should be carried out under controlled conditions in terms of both duration and temperature. To this end, the samples shall be defrosted in a refrigerator or cold room at a temperature of 3 ± 2 °C the evening before being taken into processing. The homogenisation of the soil sample may be carried out on the partially defrosted sample, provided that good mixing is possible at that time.

The entire field moist/defrosted sample is taken under processing. First, plant residues and stones are removed as much as possible. The whole sample must then be thoroughly homogenised. Homogenisation shall be carried out by thoroughly mixing the whole sample, either manually or mechanically using appropriate mixing devices, in such a way that the soil aggregates are reduced to particles of less than 5 mm. A representative sub-sample shall then be taken by manual or mechanical fractional sampling.

Manual fractional sampling (division into quarters): After thorough mixing (manual or mechanical) and reduction, the soil is spread out in a thin layer. This should be done on a pure substrate to minimise contamination. Divide the soil into 4 equal parts. The 2 diagonal portions are regrouped. Repeat this procedure until the desired sample quantity is reached.

Mechanical fractional sampling: The fractional sampling shall be carried out using an appropriate sample divider.

4 PREPARATION FOR DETERMINATION OF ORGANIC CARBON, PLANT-AVAILABLE PHOSPHORUS AND PHOSPHATE SATURATION ON PRE-DRIED SOIL

Where ammoniacal or nitric nitrogen is to be determined, a representative portion of the field moist sample shall be retained for the analysis of those parameters and for the determination of the moisture content of the field moist soil. Otherwise, the whole sample may be taken into working order.

For the determination of pH, organic carbon, plant-available phosphorus and phosphate saturation, the sample is either dried in air or under controlled conditions.

Drying under controlled conditions means drying in a oven with forced air circulation/ventilation, at a temperature of up to 45 °C for up to 48h until the residual moisture content of the dried soil is up to 1.5 %. The residual moisture shall be measured at random on 1 sample per day of measurement.

After drying, the sample is broken and then sieved at 2 mm. Only the sieved soil, free from stones, plant debris, etc., is used for further analysis.

5 QUALITY CHECK

As a quality control, at least 1 sample per day of measurement shall be analysed in duplicate for at least 1 parameter (excluding dry matter). For this purpose, 2 sub-samples are taken after sample pre-treatment and go through the entire analytical route.

Soil – Determination of moisture content — Gravimetric method

1 SCOPE

The moisture content should be determined to allow conversion to dry matter. In that procedure, the starting point may be either:

a. field damp soil (see BAM/part 1/02 point 3) resulting in W_{H_2O} in field damp soil
b. from soil after drying at 45 °C (see BAM/part 1/02 point 4) resulting in W_{H_2O} in pre-dried soil

Note: A pre-dried soil has dried a soil at up to 45 °C or air dry, with a residual moisture content of up to 1.5 %.

2 REMARK

In principle, for each air-dry sample, the residual moisture content should be determined according to that method. The determination of the residual moisture content may be omitted if a laboratory can demonstrate that the residual moisture content is always below 1.5 %. The residual moisture shall be measured at random on 1 sample per day of measurement.
However, for some applications it may still be necessary to determine the residual moisture content. In such a case, this is explicitly mentioned.

3 PRINCIPLE

Soil samples shall be dried at 105 ± 5 °C to constant weight.

4 MATERIAL

4.1 Oven set at a temperature of 105 ± 5 °C
4.2 Dried crucibles or shells
4.3 Desiccator
4.4 Balance accurate to 1 mg

5 PRACTICE

a. The drying crucibles are prepared by drying them at 105 ± 5 °C and cooling them in the desiccator.
b. The empty crucible is weighed: m_0
c. Place 10 to 15 g of field moist soil (or pre-dried soil) in the crucible and reweigh: m_1 (or $m_{1'}$)
d. Place the crucible with the sample in the preheated oven at a temperature of 105 ± 5 °C and dry to constant weight.
e. Remove Kroes from the oven and allow to cool to ambient temperature in the desiccator.
f. Reweigh to the nearest 1 mg: m_2 (or $m_{2'}$)

6 CALCULATION

6.1 METHOD OF CALCULATION FOR FIELD DAMP SOIL

$$W_{H_2O} \text{ IN FIELD DAMP SOIL} = \frac{M_1 - M_2}{M_2 - M_0} \text{ X } 100$$

with

W_{H_2O} in field moist soil moisture content on dry material in% (w/w) determined on field damp soil to the nearest 1 decimal place

m_0 mass of empty crucible in g

m_1 mass of crucible and field moist sample in g m_2
mass of crucible and dry sample (105 °C) in g

6.2 METHOD OF CALCULATION FOR PRE-DRIED SOIL

$$W_{H_2O} \text{ In pre-dried soil} = \frac{m_{1'} - m_{2'}}{M_{2'} - M_0} \text{ x } 100$$

with

W_{H_2O} in pre-dried soil moisture content on dry material in% (w/w) determined on pre-dried soil (i.e. residual moisture), up to 1 digit accurate after the decimal point

m_0 mass of empty crucible in g

$m_{1'}$ mass of crucible and pre-dried sample in g $m_{2'}$ mass
of crucible and dry sample (105 °C) in g

Note: ISO 11465 expresses the moisture content as the ratio of water to dry material. In order to correctly perform the conversion from moist to dry sample in the following procedures, that calculation method should be followed.

7 REFERENCE

ISO 11465: 1993 Soil quality – Determination of dry matter and water content on a mass basis – gravimetric method

Soil – Determination of nitric nitrogen

1 PRINCIPLE

For the determination of nitric nitrogen in the soil, an extraction with potassium chloride (KCl) should be carried out. Since the drying of soil samples may give rise to errors due to conversions, this extraction should be carried out on the field moist sample. The extraction procedure is analogous to that described in ISO 14256. The nitrate nitrogen concentration in the extract is determined spectrophotometrically (either manually or automatically). The concentrations are converted to dry matter. For this purpose, the moisture content in the soil sample shall be determined as described in BAM/Part 1/03.

2 SAMPLING AND SAMPLE PREPARATION

Soil sampling shall be carried out in layers of 30 cm. The determination of nitrate nitrogen is carried out in each layer separately. Eventually, where appropriate, the results of the different layers shall be combined to produce a total amount over the entire sampled depth.

For the implementation of sampling, reference is made to BAM/part 1/01.
The pre-treatment shall be carried out in accordance with BAM/part 1/02.

3 EXTRACTION PROCEDURE

3.1 Equipment and materials

3.1.1 Linear shaker or over-head mixer
3.1.2 Balance to weigh to 0.1 gram.

3.2 Reagents

3.2.1 Potassium chloride solution, 1 mol/l: 74.6 g/l KCl in water

3.3 Method for field damp soil

a. Weigh to the nearest 40 g, 0.1 g of the field moist homogenised sample into a container: m
b. Add 200 ml of KCl solution
c. Shake for 1h at constant temperature (20 ± 2 °C)
d. The extract is centrifuged or filtered. Rinse the filter with KCl solution before filtering the extract. Collect the remaining filtrate in a dry container.

4 DETERMINATION OF NITRATE NITROGEN IN EXTRACTS

After extraction with KCl, the relevant nitrogen fractions should be determined immediately, or at the latest 1 day after extraction. If this is not possible, the extracts may be stored in the refrigerator at 3 ± 2 °C for a maximum of 1 week or, if necessary, frozen at at at least – 18 °C.

The following spectrophotometric analytical methods may be used for the determination of nitrate in soil:

a. ISO/TS 14256-1: 2003 Soil quality – Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution – Part 1: Manual method.
b. ISO 14256-2: 2005 Soil quality – Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution – Part 2: Automated method with segmented flow analysis.
c. NBN EN ISO 13395: 1996 Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection (ISO 13395: 1996)
d. 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)

Note: for heavy load matrices, due consideration shall be given to the analysis to measure interference free

In order to carry out the analysis, reference is made to the above standard methods.

5 METHOD OF CALCULATION FOR VELDVOCHTIGE SOIL

The nitrate nitrogen concentration obtained is converted to a concentration of C_N (mg N/kg) in dry sample according to the following formula:

$$C_N \quad C_1 \, x \quad \lfloor \frac{V_{ext}}{m} \quad X \, 1 \, (\frac{W_{H_2O} \textit{ in field humid soil}}{100} \qquad \frac{W_{H_2O} \textit{ in field humid soil}}{100} \rfloor$$

with
C_N nitrate nitrogen concentration in dry sample in mg N/kg
C_l nitrate nitrogen concentration in the extract (if applicable after blank correction) in mg N/l
V_{ext} volume of extraction solvent in ml (normal 200 ml)
m weight of the field moist sample prepared for extraction in g (normally 40 g)
W_{H_2O} in field damp soil moisture content of the field wet soil determined according to BAM/part 1/03

6 REPORTING LIMIT

The maximum reporting limit is 4 kg $NO_{3\text{-}N}$/ha per soil layer.
When calculating the nitrate nitrogen over the nitrate residue determination profile, a measurement value < the reporting limit shall be set equal to half of the reporting limit (middle-bound approach) rounded to the nearest integer.

7 DETERMINATION OF NITRIC NITROGEN OVER THE PROFILE

7.1 GENERAL

The nitrate nitrogen concentration C_N is determined for each soil layer separately. This result shall be further converted using the soil density determined in accordance with BAM/part 1/09. The density varies according to the soil type and soil layer (see BAM/part 1/09). This calculation must therefore be made for each soil layer separately according to:

$$C \quad \frac{C_{N,i\,i} \; i \; D_{i} \; i \; 100}{}$$

with
C_i nitrate nitrogen content in soil layer i in kg NO3-N/ha, rounded to the nearest integer
$C_{N,i}$ nitrate nitrogen concentration in soil layer i in mg N/kg dry soil
ι density of soil layer i in kg/m^3
D_i height of soil layer i in metres (normally 0.3 m); if the height is different, the correct (average) height of the layer in question should be used here.

Finally, the results of the different soil layers are summed to a total content over the sampled profile. Therefore, when 3 layers were sampled, the total content is:

$$C_D = C_1 + C_2 + C_3$$

with
C_D total nitrate nitrogen up to depth D in kg $_{NO}$ 3-N/ha

7.2 REMARKS

a. If a **nitrate residue determination** did not allow sampling over the full depth of the profile (up to 90 cm), the total load up to a depth of 90 cm (C_{90}) shall be calculated and reported, together with a remark that this total load was obtained after calculation.

   1. Where only the 60-90 cm layer could not be sampled to the full depth, the total load C 90 is calculated as follows:

$$C_{90} = C_1 + C_2 + \frac{C_{laag3} \times 0.3}{D_{laag3}}$$

The calculated value C_{90} shall be rounded to the nearest integer.

2. When the layer 60-90 cm could not be sampled, the total load C_{90} is calculated from the value of layer 1 (0-30 cm) and layer 2 (30-60 cm) based on a multiple linear regression equation:

$$C_{90} = a + b \times C_1 + c \times C_2$$

The regression coefficients a, b and c to be used are determined by the crop group and the texture (Table 1.) Both characteristics of the parcel are made available through SMIL. The calculated value C_{90} shall be rounded to the nearest integer.

Table 1. Regression coefficients (a, b, c) for the different 'Non-sand' and 'sand' crop groups

Crop group	Non-sand			Sand		
	a	b	c	a	b	c
Corn	6.15	0.95	1.55	7.90	0.90	1.59
Cereals	5.68	0.91	1.56	10.96	0.78	1.69
Beets	3.12	1.00	1.50	4.14	0.97	1.62
Potatoes	12.10	0.91	1.51	17.28	0.79	1.59
Specific Cultivation	9.93	0.93	1.57	14.44	0.84	1.61
Other	8.42	0.91	1.59	9.64	0.81	1.67
Grass	2.10	1.01	1.59	5.21	0.90	1.66

If the 30-60 cm layer could not be sampled to the full depth, first C_2 is calculated as follows:

$$C_2 = \frac{C_{laag2} \times 0.3}{D_{laag2}}$$

The calculated value C_2 shall be rounded to the nearest integer before being used in the regression equation.

3. If only the 0-30 cm layer could be sampled, the total load C_{90} is calculated as follows:

$$C_{90} = C_1 \times \frac{1}{x}$$

The 'x' to be used is determined by the crop group and the texture (Table 2). Both characteristics of the parcel are made available through SMIL. The calculated value C_{90} shall be rounded to the nearest integer.

Table 2. Coefficient x% for the different growing groups for 'Non-sand' and 'sand'

Crop group	Coefficient x	
	Non-sand	Sand
Corn	0.42	0.38
Cereals	0.35	0.27
Beets	0.44	0.39
Potatoes	0.39	0.30
Specific Cultivation	0.35	0.29
Other Cultivation	0.35	0.31
Grass	0.48	0.39

b. For parcels (larger than 5 ha) where several sub-parcels were sampled separately, in case of nitrate residue determination, the **nitrate** nitrogen content of the entire parcel is calculated as follows:
   - For each sub-parcel, nitric nitrogen is determined up to a depth of 90 cm in accordance with point 7.1, in particular from 7.2 (a) if sampling has not taken place up to a depth of 90 cm.
   - The average nitrate nitrogen is then calculated up to a depth of 90 cm from the sub-parcels to determine the nitrate nitrogen content of the entire parcel. This average shall be rounded to the nearest integer.

8 REFERENCES

a. ISO/TS 14256-1: 2003 Soil quality – Determination of nitrate, nitrite and ammonium in field- moist soils by extraction with potassium chloride solution – Part 1: Manual method

b. ISO 14256-2: 2005 Soil quality – Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution – Part 2: Automated method with segmented flow analysis.

c. NBN EN ISO 13395: 1996 Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection (ISO 13395: 1996)

d. 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)

Soil – Determination of ammoniacal nitrogen

1 PRINCIPLE

For the determination of ammoniacal nitrogen in soil, an extraction with potassium chloride (KCl) should be carried out. Since the drying of soil samples may result in losses due to volatilisation or errors due to organic matter transformation, such extraction should be carried out on the field moist sample. In that extract, the concentration of ammoniacal nitrogen is determined spectrophotometrically. The concentrations are converted to dry matter. To this end, the moisture content in the soil moisture content in the field shall be determined as described in BAM/Part 1/03.

2 SAMPLING AND SAMPLE PREPARATION

Soil sampling shall be carried out in layers of 30 cm. The determination of ammoniacal nitrogen is carried out in each individual layer. Eventually, where appropriate, the results of the different layers shall be combined to produce a total amount over the entire sampled depth.

For the implementation of sampling, reference is made to BAM/part 1/01.
The pre-treatment shall be carried out in accordance with BAM/part 1/02.

3 EXTRACTION PROCEDURE

3.1 Equipment and materials

3.1.1 Linear shaker or over-head mixer
3.1.2 Balance to weigh to 0.1 gram.

3.2 Reagents

3.2.1 Potassium chloride solution, 1 mol/l: 74.6 g/l KCl in water

3.3 Practice

a. Weigh to the nearest 40 g, 0.1 g of the field moist homogenised sample into a container: m
b. Add 200 ml of KCl solution
c. Shake for 1h at constant temperature (20 ± 2 °C)
d. The extract is centrifuged or filtered. Rinse the filter with KCl solution before filtering the extract. Collect the remaining filtrate in a dry container.

4 DETERMINATION OF AMMONIACAL NITROGEN IN EXTRACTS

After extraction with KCl, the relevant nitrogen fractions should be determined immediately, or at the latest 1 day after extraction. If this is not possible, the extracts may be stored in the refrigerator at temperatures below 3 ± 2 °C for a maximum of 1 week.

The following spectrophotometric analytical methods may be used for the determination of nitrate in soil:

a. ISO/TS 14256-1: 2003 Soil quality – Determination of nitrate, nitrite and ammonium in field- moist soils by extraction with potassium chloride solution – Part 1: Manual method
b. ISO 14256-2: 2005 Soil quality – Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution – Part 2: Automated method with segmented flow analysis
c. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732: 2005)
d. 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)

Note: for heavy load matrices, due consideration shall be given to the analysis to measure interference free

In order to carry out the analysis, reference is made to the standard method above.

5 CALCULATION METHOD USED

The resulting ammoniacal nitrogen concentration is converted to a concentration of C_N (mg N/kg) in dry sample using the following formula:

$$C_N \quad C_l \quad X \frac{V_{ext}}{M} \quad X\,1 \left(\frac{W_{H_2O\ Infield\ humid\ soil}}{100} \qquad \frac{W_{H_2O\ Infield\ humid}\ soil}{100} \right]$$

with
C_N ammoniacal nitrogen concentration in dry sample in mg N/kg
C_l ammoniacal nitrogen concentration in the extract (if applicable after blank correction) in mg N/l V_{ext} volume of extraction solvent in ml (normal 200 ml)
m weight of the field moist sample prepared for extraction in g (normally 40 g)
W_{H_2O} in field damp soil moisture content of the field wet soil determined according to BAM/part 1/03

6 DETERMINATION OF AMMONIACAL NITROGEN OVER THE PROFILE

6.1 General

The determination of the ammoniacal nitrogen concentration C_N is carried out separately for each soil layer. This result shall be further converted using the soil density determined in accordance with BAM/part 1/09. The density varies according to the soil type and soil layer (see BAM/part 1/09). This calculation must therefore be made for each soil layer separately according to:

$$C \quad \underline{C_{N,i\,i\;i}\, D_{i}\, i\, 100}$$

with

C_i ammoniacal nitrogen content in soil layer i in kg NH_4– N/ha, rounded to the nearest integer

$C_{N,i}$ ammoniacal nitrogen concentration in soil layer i in mg N/kg dry soil

ı density of soil layer i in kg/m 3

D_i height of soil layer i in metres (normally 0.3 m); if the height is different, the correct (average) height of the layer in question should be used here.

Finally, the results of the different soil layers are summed up to a total content over the sampled profile:

$$C_D = C_1 + C_2 + C_3$$

with

C_D ammoniacal nitrogen up to depth D in kg NH_4– N/ha

7 REFERENCES

a. ISO/TS 14256-1: 2003 Soil quality – Determination of nitrate, nitrite and ammonium in field- moist soils by extraction with potassium chloride solution – Part 1: Manual method

b. ISO 14256-2: 2005 Soil quality – Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution – Part 2: Automated method with segmented flow analysis

c. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732)

d. 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)

Soil – Determination of phosphate saturation rate

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7 References **7**

1 PRINCIPLE

In acidic soil, phosphates react with iron and aluminium ions to poorly soluble compounds. The amount of phosphate that can be so captured depends on the amount and form of iron and aluminium ions present in the soil. The ‘active’ forms (the forms that bind the P_{ox}) in acidic sandy soils are the amorphous and microcrystalline forms of iron and aluminium and the ions of iron and aluminium bound to organic matter. Extraction of the soil with ammonium oxalate and oxalic acid allows the ‘active’ forms of iron (Fe_{ox}) and aluminium (Al_{ox}) to be determined separately.

The degree of phosphate saturation is expressed as the percentage of the amount of oxalate extractable phosphate in a soil in relation to the phosphate binding capacity.

$$FVG = \frac{P_{ox}}{FBV} X\ 100\ (\%)$$

with FVG phosphate saturation rate
FBV phosphate binding capacity
P_{ox} oxalate extractable phosphate

The oxalate extractable phosphate is determined by extracting a quantity of soil with a solution of oxalic acid and ammonium oxalate. After filtration, the amount of phosphate is determined in the filtrate. The phosphate binding capacity is determined by determining separately the soil extraction with oxalic acid and ammonium oxalate and then the ‘active’ forms of iron and aluminium.

2 SAMPLING AND SAMPLE PREPARATION

Soil sampling shall be carried out in layers of 30 cm. The determination of the phosphate-binding capacity and the oxalate extractable phosphate is carried out in each individual layer. Ultimately, the results of the different layers determine the profile average phosphate saturation rate.

For the implementation of sampling, reference is made to BAM/part 1/01.
The pre-treatment shall be carried out in accordance with BAM/part 1/02.

3 EXTRACTION

Extraction is carried out in a dark container.

a. weigh accurately 5.00 g air-dry soil (< 2 mm) into a dark polyethylene bottle;
b. add 100 ml extraction solvent (17.56 g oxalic acid and 28.40 g ammonium oxalate dissolve in 1 l ultra pure water);
c. shake for 2 hours shielded from light on a shaker at 20 °C;
d. filter the extract through an ash-free filter;
e. carry out the analysis on the filtrate.

4 DETERMINATION OF THE OXALAAT-EXTRAHEERBARE PHOSPHATE (POX) CONTENT OF AN ACIDIC SANDY SOIL

4.1 PRINCIPLE

The quantity of phosphate in the extract is determined colorimetrically after destruction (point 4.2) using the Scheel method or, where appropriate, after destruction with ICP-AES (NBN EN ISO 11885: 2009).
The colorimetric determination is based on the formation of a blue coloured phosphorus molybdenum complex within a certain pH range. The intensity of the blue colour is proportional to the concentration of phosphate in solution. However, blue colouring is prevented by the presence of oxalic acid. That oxalic acid must first be broken down by rendering. This involves converting the oxalate into $CO_2$by oxidation with concentrated H_4 $SO_{2\ and\ H}$ 2_{O2}.

4.2 DESTRUCTION OF OXALATE IN THE EXTRACT

a. 10 ml of concentrated H 100 SO 1 (minimum 95 %) is added to $_2$ml of extract in a $_4$ ml measuring cup.
b. On the hotplate, the cups are heated until the oxalic acid is completely broken down (the liquid is browsed and white fumes appear).
c. After cooling, 2 ml H_2O_2 (minimum 27 %) (Perhydrol, thermostable H_2O_2) is added and re-boiled. If precipitation occurs, eventings must be boiled with ultra pure water.
d. After cooling, the destruate is transferred quantitatively into 50 ml flasks and diluted with ultra pure water to the mark.

4.3 REAGENTS

4.3.1 Scheel 1
Dissolve 1 g 4-methylaminophenol sulphate ($(CH_3NHC_6H_4OH)_2.H2SO_4$), 5 g anhydrous sodium sulphite (Na_2SO_3), 75 g sodium disulphite ($Na_2S_2O_5$) and 200 mg lauryl sulphate, stirring up to 1 l with ultra pure water and homogenising.

4.3.2 Scheel 2
Dissolve 50 g of ammonium molybdate $(NH_4)_6\ Mo_7O_{24.4H\ 2}$ O in approximately 250 ml of H_2O, add 140 ml of concentrated $H_2\ SO_4$, cool, dilute to 1 l with ultra pure water and homogenise.

4.3.3 Scheel 3
Dissolve 340 g sodium acetate ($CH_{3COONa.3H\ 2}$ O) and make up to 1 l with ultra pure water.

4.3.4 Prepare a standard solution of 25 mg P/l

4.4 PRACTICE

a. For the standards, 0, 1, 2, 3, 4, 5 and 6 ml of 25 mg P/l stock solution are pipetted into a 50 ml flask. This corresponds to a concentration of 0; 0.5; 1; 1.5; 2; 2.5 and 3 mg P/l
b. The amount of destruate should be varied according to concentration. Place this quantity in a 50 ml flask.
c. Add 5 ml of Scheel 1 (4.3.1), shake; add 5 ml of Scheel 2 (4.3.2) and shake.
d. After 15 minutes, add 10 ml of Scheel 3 (4.3.3) and rinse the 50 ml volumetric flasks with ultra pure water to the mark.
e. Homogenise and leave to rest for 15 minutes
f. Measure the absorption at 662 nm.

4.5 CALCULATION

Using a calibration graph, the phosphate concentration (expressed as phosphorus) in the 50 ml flask is obtained. To convert it to the concentration in the destruate (C_l), account must be taken of the dilution used (50/volume taken for photometric determination).
This concentration should be further converted from mg P/l to mmol P/kg air dry soil. This is done according to:

$$P_{OX} = C_1 \times 0.1 \times 5 \times \frac{1000}{30.97 \times M} = \frac{C_1}{M} \times 16.14 \quad \text{(mmol P/kg air dry soil)}$$

with
P_{ox} extractable phosphorus concentration in soil in mmol P/kg soil, rounded to one decimal place
C_1 phosphorus concentration in the undiluted destruate in mg P/l
0.1 volume of extract in l
5 dilution propeller used in oxalate destruction (50/10)
30.97 Atomic mass of phosphorus in g/mol
m weight of soil taken for extraction in g (normally 5.00 g)

5 DETERMINATION OF PHOSPHATE BINDING CAPACITY (FBV)

5.1 ANALYTICAL DETERMINATION

For the determination of the phosphate binding capacity, the iron and aluminium concentration in the extract is determined using ICP-AES (NBN EN ISO 11885: 2009) or AAS (atom absorption spectrophotometer). This determination is carried out directly on the extract, after appropriate dilution with ultra pure water, or after destruction of the oxalate in the extract in accordance with point 4.2.

5.2 CALCULATIONS

$$FE_{OX} = \frac{C_{1,FE}}{M} \times 8.95 \quad \text{(mmol Fe/kg air dry soil)}$$

with
FE_{ox} extracted iron concentration in soil in mmol Fe/kg soil, rounded to the integer

$C_{1, Fe}$ iron concentration in the undiluted extract in mg Fe/l
m weight of soil prepared for extraction in g (normally 5.00 g)

$$Al_{ox} = \frac{C_{1, Al}}{m} \times 18.53 \text{ (mmol Al/kg air dry soil)}$$

with
Al_{ox} extracted aluminium concentration in soil in mmol Al/kg soil, rounded to the integer
$C_{1, Al}$ aluminium concentration in the undiluted extract in mg Al/l
m weight of soil prepared for extraction in g (normally 5.00 g)

FBV = 0.5 x (Fe_{ox} + Al_{ox}) (mmol P/kg air dry soil) with
0.5 proportionality factor determined experimentally
FE_{ox} oxalate extractable iron (mmol per kg air dry soil)
Al_{ox} oxalate extractable aluminium (mmol per kg air dry soil)

6 DETERMINATION OF PHOSPHATE SATURATION RATE (FVG)

To determine the phosphate saturation rate of the soil, the FBV and the oxalate extractable phosphate in the 3 layers are determined separately. The average values of FBV and P_{ox} are calculated over the profile, from which the profile average FVG is calculated.

6.1 B EPALING OF THE AVERAGE FBV AND P_{OX}

6.1.1 IF SAMPLED OVER THE FULL DEPTH (UP TO 90 CM IN 3 LAYERS):

$$FBV = \frac{FBV_1 + FBV_2 + FBV_3}{3}$$

$$P_{OX} = \frac{P_{OX,1} + P_{OX,2} + P_{OX,3}}{3}$$

6.1.2 IF SAMPLED TO A DEPTH OF BETWEEN 0 AND 30 CM (1ST LAYER INCOMPLETELY SAMPLED):

$$FBV = FBV_1 \text{ AND } \quad P_{OX} = P_{OX,1} =$$

6.1.3 IF SAMPLED TO A DEPTH OF BETWEEN 30 AND 60 CM (2ST LAYER INCOMPLETELY SAMPLED):

$$FBV = \frac{(FBV_1 \times 30) + (FBV_2 \times (\text{SAMPLING DEPTH} - 30))}{\text{SAMPLING DEPTH}}$$

$$P_{OX} = \frac{(P_{OX,1} \times 30) + (P_{OX,2} \times (\text{SAMPLING DEPTH} - 30))}{\text{SAMPLING DEPTH}}$$

6.1.4 If sampled to a depth of between 60 and 90 cm (3st layer incompletely sampled):

$$FBV = \frac{(FBV_1 \times 30) + (FBV_2 \times 30) + (FBV_3 \times (SAMPLING\ DEPTH - 60))}{SAMPLING\ DEPTH}$$

$$P_{OX} = \frac{(P_{OX,1} \times 30) + (P_{OX,2} \times 30) + (P_{OX,3} \times (SAMPLING\ DEPTH - 60))}{SAMPLING\ DEPTH}$$

6.2 Calculation of profile average phosphate saturation rate

6.2.1 General

The profile average phosphate saturation rate is calculated according to:

$$FVG = \frac{P_{OX}}{FBV} \times 100\ (\%)$$

with
FVG % phosphate saturation rate, whole numbers
P_{ox} the profile mean value of the oxalate extractable phosphorus. FBV
the profile mean value of the phosphate binding capacity

7 REFERENCES

a. NBN EN ISO 11885: 2009 Water quality – Determination of selected elements by Inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885: 2007)

Soil – Minerable specific weight (density)

1 SCOPE

The apparent specific weight (density) is used to convert the concentrations to kg N/ha when determining ammonium and nitric nitrogen.

2 APPARENT SPECIFIC WEIGHT

2.1 B ODEMLAAG 1 (0-30 CM)

A fixed soil density is assumed for the density of the building for (0-30 cm) for the loam and sandy loam soils (Alfisol soil order) on the one hand, and the soils in sandy Flanders and the Kempen (Spodosol soil order) together with the polders and dunes rounds (Entisol soil order) on the other.

a. For the loam and sandy loam soils (order Alfisol), a fixed average density of 1450 kg/m³ is proposed.
b. No distinction is made between the Spodosol and Entisol soil orders. For this reason, a fixed average density of 1250 kg/m³ is proposed for the soils of sandy Flanders and the Kempen and the polder and dunes rounds.

The type of soil can be consulted on the soil map of Geopunt Vlaaanderen showing the agricultural areas (http://www.geopunt.be/). The texture feature made available through SMIL can also be used.

2.2 B ODEMLAAG 2 AND 3 (30-90 CM)

For the underlying soil layers (deeper than 30 cm), a fixed density of 1500 kg/m³ is proposed.

Soil – Determination of organic carbon content

1 PURPOSE AND SCOPE

This procedure describes two methods for the determination of total organic carbon (TOC) in soil, one using the indirect method (Method A) and the other using the direct method (Method B).

The procedure described in NBN EN 15936: 2022 applies subject to the following additions/adaptations.

2 DEFINITIONS

- TC (total carbon): content of carbon present in the sample in the form of organic, inorganic and elemental carbon
- TIC (total inorganic carbon): content of carbon exempted as CO_2 by acid hydrolysis
- TOC (total organic carbon): content of carbon converted to CO_2 by combustion and not exempted as CO_2 by acid hydrolysis (i.e. the difference between TC and TIC).

3 PRINCIPLE

For the indirect method (Method A), the TOC content is calculated from the difference of the analytical results of the TC and TIC content. The TC content is determined by measuring the CO_2 exempted by burning the dried sample in an oxygen-containing gas stream free of CO_2. Catalytic converters/modifiers may be added to achieve complete combustion. The released amount of CO_2 is measured by infrared spectrometry, thermal conductivity detection, flame ionisation detection after reduction to methane, or by gravimetry, coulometry, conductometry after absorption. The TIC is determined individually on another sub-sample by acidification of the sample in which the inorganic carbon is removed by purging and the CO_2 gas formed is measured by one of the above techniques.

Under the direct method (Method B), the carbonates present in the sample are pre-removed by acid treatment of the sample. The amount of CO_2 exempted from subsequent combustion is measured by one of the above techniques and is a direct measure of the TOC content.

ADDITIONS NBN AND 15936

- Paragraph 6 Reagents: Other reagents and/or concentrations may be used provided they are appropriate for this use.
- Paragraph 8 Sample preparation: Sample preservation is described in BAM/part 1/01 and sample preparation in BAM/part 1/02.

- Paragraph 9 Procedure – Method A (indirect method):
  - Paragraph 9.1.3 of the TIC last paragraph:
    The treatment of samples with heated acid is not applicable.
  - § 9.2 calibration:
    In order to perform the daily analyses, calibration should be checked (e.g. with the highest standard) at least for each measurement series and should comply with the criterion established by the laboratory. Whether or not a correction is applied shall be recorded by the laboratory.
  - Paragraph 9.3 Monitoring measurements:
    The recovery rate for the TC and TIC content of control sample A shall be between 80 and 120 % of the appropriate value.
  - Paragraph 9.4 Calculations:
    Method A is applicable at a TIC/TOC ratio < 10; if not fulfilled, this shall be noted as a comment in the report.
    When calculating the TOC content, the measured values of TC and TIC (ignoring the limit of quantification) shall always be used.
- § 10 procedure – Method B (direct method)
  - § 10.2 calibration:
    In order to perform the daily analyses, calibration should be checked (e.g. with the highest standard) at least for each measurement series and should comply with the criterion established by the laboratory. Whether or not a correction is applied shall be recorded by the laboratory.
  - Paragraph 10.3 Monitoring measurements:
    The recovery rate for the TOC content of control sample B shall be between 80 and 120 % of the correct value.

Note 1: The laboratory must have the necessary data to demonstrate that the acid treatment is effective in removing the carbonates present for the type of samples analysed by the laboratory. This should be demonstrated on at least 1 sample per acid treatment/sample series.

Note 2: Control samples other than those specified in EN 15936 may be used as long as their accuracy and precision can be verified. The recovery rate for the TOC content of this control sample shall be between 90 and 110 % of the correct value.

4 CALCULATIONS

The TOC result is expressed in m/m% C dry matter.

5 REFERENCES

- NBN EN 15936: 2022 Soil, waste, treated biowaste and sludge – Determination of total organic carbon (TOC) by dry combustion.

Soil – Determination of phosphate extractable in soil with ammonium lactate – acetic acid buffer (P-AL)

1 PRINCIPLE

This standard describes a method for the determination of the plant-available P content in soil, extractable with a solution of ammonium lactate acetic acid buffered at pH = 3.75. This method can be used on pre-dried soil samples sieved through a 2 mm sieve.

The pre-dried soil samples are extracted at a ratio of 1: 20 (mass/volume) with a solution of ammonium lactate-acetic acid buffer at pH 3.75. That extraction solvent extracts the calcium phosphate compounds present and part of the iron and aluminium compounds present. Part of the clear filtrate is analysed for phosphate according to existing analytical methods.

Prior to this measurement, the pH value shall be determined and reported. For soil samples with pH-KCl > 7, a comment is added to the report.

2 SAMPLING AND SAMPLE PREPARATION

Soil sampling for the determination of the plant-available P shall be carried out in accordance with BAM/part 1/01.
The pre-treatment shall be carried out in accordance with BAM/part 1/02.

3 REAGENTS AND SOLUTIONS

3.1 Reagents

3.1.1 Water, use for all water solutions according to NEN-EN-ISO 3696.
3.1.2 Sodium hydroxide solution, c (NaOH) = 0.1 mol/l.
3.1.3 Hydrochloric acid solution, c (HCl) = 0.1 mol/l.
3.1.4 Phenolphthalein indicator solution obtained by dissolving 1 g of phenolphthalein in 100 ml of pure ethanol (approximately 96 %).
3.1.5 Methyl red indicator solution obtained by dissolving 0.1 g of methyl red in 100 ml of ethanol (approximately 60 %).
3.1.6 Lactic acid (ρ = 1.21 g/cm^3)
3.1.7 Note: The solution has a shelf life of 5 years.

3.2 Solutions

3.2.1 Lactic acid solution (about 3 mol/l).
Obtained by diluting 500 ml of lactic acid with 1 l of water in a pyrex bottle and subjected to the following operations. Cover the bottle with a watch glass and place it for 48 h in a oven at 95 °C to hydrolyse the lactic acid. Leave to cool.

Determine the concentration of the solution as follows. Pipette 10.0 ml of the solution into a 100 ml graduated flask and make up with water. Homogenise and pipette 10.0 ml of this diluted solution and titrate with a 0.1 mol/l NaOH solution using phenolphthalein as an indicator. Calculate the concentration (= A) of the lactic acid solution.

3.2.2 Concentrated acetic acid (about 16 mol/l).
Determine their exact concentration as follows. Pipette 10.0 ml of acetic acid into a 500 ml volumetric flask containing 400 ml of water. Make up to volume with water and homogenise. Pipette 10.0 ml of that diluted solution and titrate with a 0.1 mol/l NaOH solution using methyl red as an indicator. Calculate the exact concentration (= B) of the concentrated acetic acid.

3.2.3 Concentrated ammonia solution (about 13 mol/l).
Determine their exact concentration as follows. Pipette 10.0 ml of ammonia into a 500 ml volumetric flask containing 400 ml of water. Make up to volume with water and homogenise. Pipette 10.0 ml of that diluted solution and titrate with a 0.1 mol/l HCl solution using methyl red as an indicator. Calculate the exact concentration (= C) of the concentrated ammonia.

Note: The set value of solutions 3.2.1 to 3.2.3 is valid for 1 day. If solution 3.2.4 is made from non-fresh solutions, those solutions should be reinstated.

3.2.4 Concentrated extraction solution.
Take a 1-litre volumetric flask already containing 300 ml of water and add: 1000/A ml of lactic acid solution, 4000/B ml of concentrated acetic acid and 1000/C ml of concentrated ammonia, mix after each addition. Allow to cool and make up with water and homogenise.

Note: The solution has a shelf life of 1 years.

3.2.5 Diluted extraction solution.
Dilute 500 ml of the concentrated extraction solution (3.2.4) to 5 litre with water. The pH of the solution must be: 3.75 ± 0.05.

Note: This solution is stable for 5 days.

4 EQUIPMENT

4.1 Usual laboratory glassware.
4.2 100 ml wide aperture flasks.
4.3 Shaking machine (180 strokes per minute).
4.4 Hard paper filters that are phosphate-free and do not adsorb phosphate.
4.5 pH meter

5 PROCEDURE

Weigh 2.5 g ± 0.05 g of pre-dried soil sample into a shake-flask and add 50 ml of diluted extraction solution (3.2.5). Take two blank samples. Shake for 4 h at 20 ± 2 °C. Filter the suspensions and blank samples. Check the filtrates for clarity. If necessary, filter a second time.

6 ANALYSIS

Within 24 hours of extraction, if necessary at a dilution, determine the P-concentration of the filtrates by an appropriate analytical method:

a. NBN AND ISO 15681-1: 2005 water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 1: Method by flow injection analysis (FIA) (ISO 15681-1: 2003)
b. NBN AND ISO 15681-2: 2019 water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2: 2018)
c. NBN AND ISO 6878: 2004 water quality – Determination of phosphorus – Ammonium molybdate spectrometric method (ISO 6878: 2004)
d. NBN EN ISO 15923-1: 2024 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection (ISO 15923-1: 2013)
e. Scheel colorimetric method
f. NBN AND ISO 11885: 2009 water quality – Determination of selected elements by Inductively coupled plasma optical emission spectrometry (ICP-OES)(ISO 11885: 2007).

Note: Standard series should be made in the same medium (3.2.5) as the ground extracts.

7 CALCULATION

7.1 GENERAL

The extractable phosphate content according to the P-AL method is expressed in mg P per 100 g air dry soil, using the following formula:

$$P\ AL\ \frac{(a\ b)\ x\ f}{m}\ x\ 5$$

where:

P – AL	is the extractable phosphate content in soil samples, in mg P per 100 g air-dry soil;
a	is the concentration of phosphate in the soil extract, in mg/l P;
b	is the mean concentration of phosphate in the blank samples, in mg/l
P; m	is the mass of the dry air sample in grams;
f	is dilution factor.

7.2 REMARKS

a. Note: 1 mg P per kg = 0.229 mg P_2O_5 per 100 g
b. For parcels (larger than 5 ha) where several sub-parcels were sampled separately, the extractable phosphate content of the entire parcel is calculated as the average of the sub-parcels using the P-AL method and reported.

8 REFERENCES

a. H. Egner, H. Riehm and W.R. Domingo, Untersuchungen über die chemische Bodenanalyse as Grundlage für die Beurteilung des Nährstoffzustandes der Böden. II Chemical Extraktionsmethodszur Phosphor- und Kaliumbestimmung, Kungl. Lantbrukshögskolans Ann. 26, 199-215, 1960.
b. NEN 5793: 2013 soil – Determination of phosphate extractable in soil with ammonium lactate-acetic acid buffer (P-AL).

Soil – Determination of rapidly released organic nitrogen

1 PRINCIPLE

The organic matter for study is incubated in a reference soil under controlled conditions of temperature, moisture content and density. Samples are taken at regular intervals to determine the amount of mineral N in the soil. The time series of the mineral N content in the soil then allow the mineralisation (possibly immobilisation) of N from the organic matter to be determined.

Note: The total N content must be known. This determination was performed by a VLAREL accredited laboratory according to the methods described in BAM and/or CMA (depending on the matrix type).

2 PRACTICE

2.1 SOIL PRETREATMENT

Reference soil [1] is used. The reference soil is dried to air dry and sieved on a 2 mm sieve. The soil is then stacked in a container at a density of 1.4 Mg m^{-3} and wetted to a moisture content of 35 % of pore volume filled with water [2]. The soil is thus incubated for one week at a temperature of 15 ± 2 °C.

2.2 THE ORGANIC MATTER APPLIED

After pre-incubation, the organic matter should be administered fresh to the reference soil as it will be used in practice. In most cases, the material to be incorporated is fine particles. It must be highly homogenised before incorporation. In the case of coarse material (e.g. plants), it must be further cut or chopped into particles of a size of 0.25 to 0.5 cm^2. The desired predetermined amount of organic matter is mixed intensively with a certain amount of reference soil (sufficient to fill one incubation container: the material should therefore not be mixed in bulk with the total amount of soil in order to minimise variability). The dose used in practice will guide the determination of the amount of organic matter to be administered.

2.3 INCUBATION

---

[1] the reference soil has an initial mineral N < 20 mg N-NO_3/kg content and a low mineralisation potential, so that mineralisation from the added organic matter is well traceable. The texture of the reference soil shall be loamy sand, light sandy loam, sandy loam or loam with a pH KCl between 5 and 7.5 and an organic carbon content of less than 1.5 %.

[2] the residual voids volume is calculated as 1 – (Dry Density bodem/2.65). In this case 1 – (1.4/2.65) = 47.2 %. A moisture content of 35 % water-filled pore volume corresponds to 16.5 volume% moisture per volume of unit dry soil. Taking into account the density of dry soil, this is to add 118 ml water to 1 kg dry soil.

PVC cylinders with a length of 0.18 m and an internal diameter of 0.046 m are used as incubation containers, with a well-fitting cap at the bottom. These incubation containers are filled up to a height of 10 cm with the mixture of the organic matter with the soil. The apparent density of the soil in the container is brought to a predetermined value by pressing the mixture. This pressure must be applied evenly during the filling of the container in order to ensure a homogeneous density throughout the length of the container. Particular care should be taken to ensure that the soil surface is not obscured when adjusting density, as this may adversely affect mineralisation. For a filling height of 10 cm and an apparent density value of 1.4 Mg m^{-3}, weigh 233 g of dry reference surface inside the container. When weighing the soil, the moisture content of the soil after pre-incubation should of course be taken into account. After filling, the moisture content of the mixture in the incubation containers is adjusted to 50 % of the pore volume filled with water, taking into account of course the moisture content in the applied organic matter and the moisture already present in the soil. The containers are then sealed with a parafilm, which minimises moisture losses during incubation but still allows gas exchange. The weight of the filled containers shall be determined. This weight is recorded and checked regularly during incubation to ensure that no excessive loss of moisture occurs. If the moisture content in an incubation container falls by more than 1 % (absolute) during incubation, the moisture content should be adjusted by adding ultra pure water. The soil is incubated at a constant temperature of 15 °C. Containers are also incubated with only the soil, i.e. without added organic matter, which should allow the determination of the net N mineralisation (= blank samples).
The total duration of incubation is 4 months.

2.4 Sampling

9 samples are provided during incubation. This number is necessary to overcome the high variability that is inevitable when working with fresh organic matter. The duration of incubation is 4 months. At predetermined times, a number of incubation containers (both containers with and without added organic matter) are sampled for destructive determination of the mineral N content in soil. Sampling is carried out on day 0 and every 14 days thereafter until the end of incubation (days 14, 28, 42, 56, 70, 84, 98, and 112). Sampling shall be carried out at least in triplicate, i.e. a minimum of three containers with and three containers without added organic matter shall be analysed per sampling time.

2.5 Analysis

Based on the recommended dose of the sample expressed in tonnes per ha, a surface maintenance equivalent is calculated for inclusion in the incubation container (m_{sample}).

A moisture content is determined on both the reference soil and the sample in order to adjust the moisture content in the incubation container to 50 % of the pore volume filled with water.

From the total N content in the sample [N total sample] $_{and}$ the amount of sample added per incubation container, the net amount of additional total N added per incubation container can be calculated ([N_{sample}]).

At the start of incubation, at 6 intermediate times and after 4 months, an analysis of mineral N (NO_3^--N + NO_2^--N + NH_4^+-N) is performed on the mixture and the reference soil (blank). The incubation containers are emptied, their contents are mixed intensively and a fresh subsample (30 grams) is immediately extracted with a KCl solution (150 ml; 1 m) for the determination of the amount of mineral N in the extract (NO_3^--N + NO_2^--N + NH_4^+-N)

Another sub-sample (30 grams) is used to determine the moisture content of the soil in the container (drying at 105 °C to constant weight).
On the basis of the extracts analysed, the following contents can be calculated at any time:

a. [No_3, soil]: nitrate content in reference soil expressed in mg of N-NO_3 per incubation container
b. [NH_4, soil]: ammonium content in reference soil expressed in mg of N-NH_4 per incubation container
c. [No_3, mixture]: nitrate content in mixture expressed in mg of N-NO_3 per incubation container
d. [NH_4, mixture]: ammonium content in mixture expressed in mg of N-NH_4 per incubation

container

For incubation

a. 2 total N determinations (sample only)
b. 4 determination of moisture content (2 reference base, 2 sample)

During incubation (9 times)

a. 54 KCl extractions
b. 54 provisions of NO3--N + NO2 – N
c. 54 provisions of NH4 ± N
d. 9 determination of moisture content

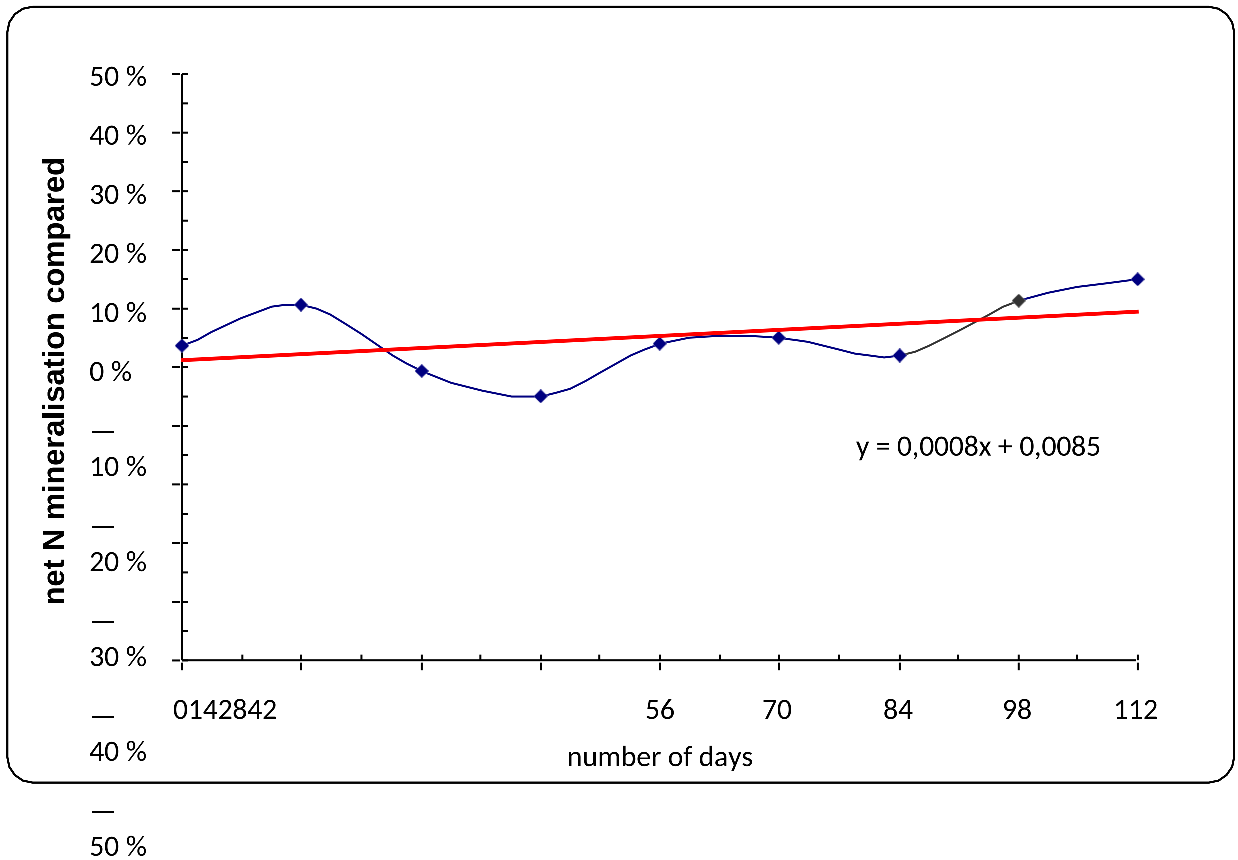
3 CALCULATIONS

The net amount of mineralised N is calculated by subtracting from the mineral N contents determined in the treatments with added organic matter the mineral N amounts measured in the blank treatment. The amount of easily mineralisable N from the organic material is then calculated by expressing the amount of mineral N exempted after 4 months on the total amount of N (organic + mineral N) present in the material.

The percentage net nitrogen mineralisation (% N mineralisation) expressed as total additional total nitrogen added (from sample) is calculated per time point as:

$$\% N_{minus\ eralisation} \quad \frac{No_{3,\,mixture} \quad NO_{3,\,bottom} \quad NH_{4,\,mixture} \quad NH_{4,\,bottom}}{N_{sample}}$$

The percentage content of rapidly released organic nitrogen is calculated by performing linear regression at the 9 points and calculating the percentage net nitrogen mineralisation after 4 months (example presentation below, the percentage content of rapidly released organic nitrogen is 9.8 %).


net N mineralisation compared
50 %
40 %
30 %
20 %
10 %
0 %
— 10 %
— 20 %
— 30 %
— 40 %
— 50 %
y = 0,0008x + 0,0085
0142842
56
70
84
98
112
number of days

Soil – Determination of pH

1 PURPOSE AND SCOPE

This procedure describes the determination of pH in a 1: 5 (volume fraction) suspension of soil in 1 M KCl.

The procedure described in NBN EN ISO 10390: 2022 shall apply subject to the following adaptations:

- Paragraph 1 Scope: extraction solvent is 1M KCl;
- Paragraph 4 Principle: extraction solvent is 1M KCl
- Paragraph 6.3 Equipment: combined glass electrode: a maximum deviation of 0.30 pH units or ± 18 mV is proposed for the zero point of the glass electrode; the value of the slope shall be between 95 % and 102 % of the theoretical slope;
- Paragraph 7 Sample preparation: Sample preservation is described in BAM/part 1/01 and sample preparation in BAM/part 1/02. The analysis is carried out on the sample dried at max. 45 °C and sieved through a 2 mm sieve.

  Note: the pH determination may also be carried out on an air-dried soil sample.

- Paragraph 8.1 Preparation of the suspension: extraction solvent is 1M KCl
  In addition to mechanical shaking for 60 minutes, mechanical shaking for a shorter period and manual shaking of the suspension is permitted for the purpose of thoroughly suspending the soil in the extraction solution. A minimum contact time between soil and KCl solution of 2 hours is necessary. (Reference: Vito report 2007/MIM/R/023);
- PARAGRAPH 8.2: calibration of the pH meter and the pH measurement itself shall be carried out in accordance with NBN EN ISO 10523: 2012 *Water quality – Determination of pH*;
- PARAGRAPH 8.2: 20 °C ± 5 °C;
- PARAGRAPH 8.2: calibration is checked by measuring an independent buffer solution. In the range between 4 and 10, the measured pH value will not differ by more than 0.1 pH units from the theoretical buffer value;
- PARAGRAPH 8.3: 20 °C ± 5 °C.

2 REFERENCE

- NBN EN ISO 10390: 2022 Soil, treated biowaste and sludge – Determination of pH (ISO 10390: 2021)
- NBN AND ISO 10523: 2012 water quality – Determination of pH (ISO 10523: 2008)
- C. Vanhoof, B. Van Hasselt, K. Duyssens and K. Tirez, Determination of pH in soil, VITO report 2007/MIM/R/023, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/rapport_pH_2006.pdf
- C. Vanhoof, H. Van den Broeck, S. Hofman, Groep AN and K. Tirez, Comparative measurements of pH in soil, VITO report 2009/MANT/R/068, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/rapport_pH_2009_final.pdf

Soil – Determination of soil texture by manual method

1 PURPOSE AND SCOPE

The granulometric composition (grain distribution) of a soil is an important element, as it significantly influences both the physical and chemical properties of a soil. A soil consists of a mixture of grains of different sizes. The grain size can be broken down into different fractions:

- the fraction > 2 mm is called the coarse or gravel fraction and as such is not included in a granulometric analysis;
- the fraction < 2 mm is called fine earth or fine soil and this fraction is then subdivided into further sub-fractions:
  - sand fraction (50-2 000 µm);
  - loam fraction (2-50 µm);
  - clay fraction (< 2 µm).

The texture of a soil is named in function of the granulometry, which is the content of clay (0-2 µm), loam (2-50 µm) and sand (50-2 000 µm). For the exact determination of the texture, it is necessary to calculate the percentage of each of the fractions present in the mixture; the texture can then be deduced using a triangular graph.
The Centre for Soil Mapping (RijksUniversiteit Gent) proposed a texture triangle for all Belgian soils around 1950 (Figure 1 Belgian texture triangle). The zoning and designation is a compromise between all the terms used by the different mapping leaders.

The demarcation of the different zones is as follows:

- Sand
  1. sand
  2. clayey sand
  3. loamy sand
- Sandy loam
  4. light sandy loam
  5. sandy loam
  6. heavy sandy loam
- Loam
  7. very light loam
  8. light loam
  9. heavy loam
- Clays
  10. loamy clay
  11. light clay
  12. clays
  13. sandy clay
  14. heavy clay
  15. very heavy clay
  16. heavy sandy clay

A soil expert with field experience is able to distinguish these soil texture classes by kneading and crushing the soil between the fingers. However, it remains appropriate to regularly confront these estimates in the field with the laboratory’s analytical data.

Texture provisions in the context of soil protection require only a distinction in the following 4 main textures: sand, sandy loam, loam and clay.

Classification in the main textures may be carried out by manual texture determination or by granulometric texture determination (Robinson-Köhn pipette method).

This procedure describes guidelines for manual texture determination and BAM/Part 1/15 describes the procedure for granulometric texture determination.

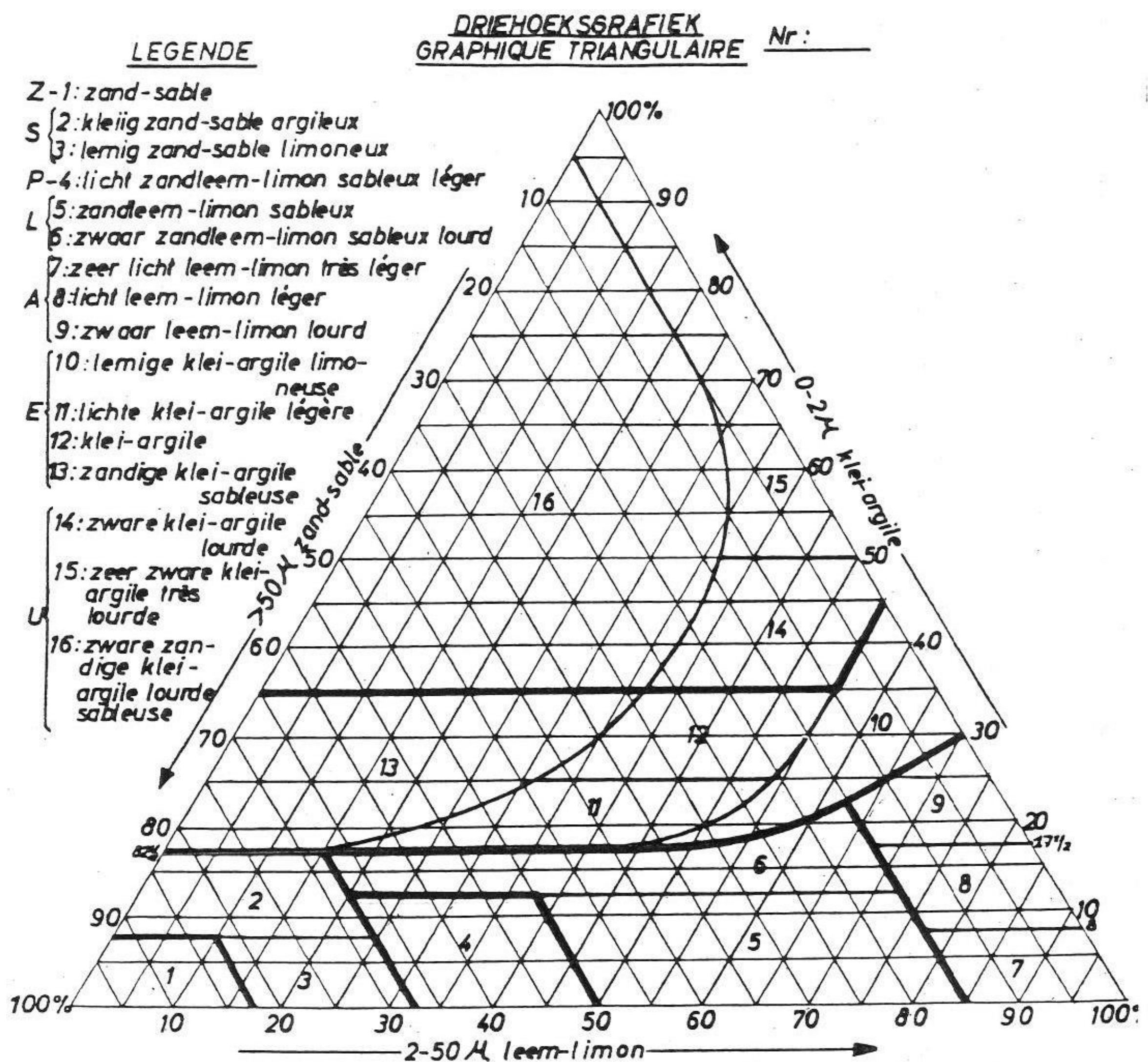


Figure 1: Belgian texture triangle

2 MANUAL TEXTUURBEPALING

The texture of the soil can be estimated manually and an experienced soil expert can achieve a high degree of accuracy. The estimation is done by kneading and crushing the soil between the fingers. A number of guidelines, drawn up by the University of Ghent, are described below to enable this manual texture estimate to be carried out. Some physical

characteristics shall be described and, to the extent possible, the different textures shall be described on the basis of these characteristics.

Each soil expert may apply his or her own specific method. Experience is by far the most important characteristic for correctly estimating the texture.

3 PHYSICAL PROPERTIES

STICKINESS

This property is estimated by compacting the soil between thumb and index finger and then removing the two fingers from each other. Depending on the amount of soil that remains attached to the fingers and depending on whether the soil expands when the fingers are removed, the stickiness of the material is indicated. Usually sand is not sticky, as when released almost nothing remains attached to the fingers. Loam is slightly sticky: it sticks, but it has little resistance at the point of discharge. A heavy clay is very sticky, as the material adheres strongly to the fingers and stretches out when they are removed from each other.

PLASTICITY

Plasticity is estimated by the extent to which the material can be rolled into a wire or rod between thumb and index finger and by the extent to which the wire or rod can be processed. Usually sand is not plastic (no wire can be rolled). Sandy loam is not very plastic (a short roller can be rolled but tears in the roller). Loam is slightly plastic (a roller can be rolled, but the roller breaks when attempting to fold it into a horseshoe shape). Clay is very plastic (the roll turns into a horseshoe shape and, in the case of heavy clay, even into a circle).

MALLEABILITY

The malleability is determined by the pressure required to knead a soil mass with a moisture content between air dry and field capacity. Sand is loose to very crumbly, loam is crumbly and heavy clay is rigid to extremely rigid.

CONSISTENCY

The consistency is determined in the dry state by breaking a soil mass between thumb and index finger. No general behaviour of the different textures can be mentioned in this regard. For example, sand can be loose or extremely hard in the dry state.

4 DESCRIPTION OF THE DIFFERENT TEXTURES

SAND

Sand consists of loose grains that can be individually felt and generally seen. If a handful, more or less cohesive and fairly dry sand is taken, it can be dispersed into loose grains with very slight pressure. In the damp state, the material is somewhat coherent, although it will disintegrate due to weak mechanical action (pressure, shock, etc.). Sand lacks any plasticity and cohesion.

SANDY LOAM

Sandy loam already contains a very small to small amount of clay, so the grains are slightly more cohesive. As a result, in the dry state, the material is less easily dispersed into loose grains. However, individual sand grains can still be seen. In the dry state, the nozzles are easy to break, while in the humid state they can already be manipulated with caution (e.g. kneaded into a cylinder).

LOAM

Loam has a low to moderate clay-sand content. This material therefore forms lumps or crumbs in the dry state, which can be broken and crushed by hand. The lower the presence of sand, the softer the material feels. The more the clay content increases, the more difficult it is to break down the dry aggregates. Loamy material feels floral (meel-like). Both dry and wet form aggregates or lumps that are easy to treat. However, in the wet to wet state, kneading between thumb and index finger can only produce a short thread which breaks down rapidly due to its own weight. If a damp block is printed by hand, it breaks down into fragments.

CLAYS

Clay, in the dry state, forms aggregates or lumps that can no longer be broken by hand. They are very hard. If the aggregates are moist to wet, the material is plastic (e.g. modelling clay), usually rigid (it requires considerable strength to knead clay) and often slightly sticky. By kneading between thumb and index finger, a long, flexible thread can be formed. The material feels greasy in a more wet state.

The above differences do not reflect all the nuances that a specialist feels between the different textures. The whole becomes even more complex when the soil contains organic matter and $CaCO_3$. A rather general rule here is that due to a higher content of well-digested organic matter sandy soils appear more clayey, while heavy clay soils will be more clayey and less rigid than their clay content would suggest. Large amounts of $CaCO_3$ feel soft on what is a characteristic of loamy soils. In addition, the sand content will appear higher as the grains are large. Even a slightly contiguous flask of sandy grains may exaggerate the sandy assessment of a soil.

Soil – Determination of the soil texture using the Robinson-Köhn pipette method

1 PURPOSE AND SCOPE

The clay content is determined by a partial texture analysis. The texture analysis concerns the separation of the mineral soil into granulometry fractions, in particular sand, loam and clay, as well as the determination of the proportions of the fractions. The analysis is carried out on the fine earth (< 2 mm), after separation of the coarse elements. In order to obtain a good dispersion of the clay fraction, all cementing materials such as organic matter, carbonates and dissolved salts present should be removed.
The fine fractions (loam and clay) are separated from the sand by wet sevening on a 50 µm sieve. The clay content is determined by a pipette of Robinson-Köhn after dispersion of the colloidal fraction with a dispersive substance. The time and depth of the pipetop (10 cm) are derived from the Stokes Act.

The procedure described in ISO 11277: 2020 and ISO 11277: 2020/Amd 1: 2024 shall apply with the following additions/adaptations.

2 ISO 11277: 2020 AND ISO 11277: 2020/AMD 1: 2024 SUPPLEMENTS

- Paragraph 7 Sample preparation: Sample preservation is described in BAM/part 1/01 and sample preparation in BAM/part 1/02. The analysis is carried out on the sample dried at max. 45 °C and sieved through a 2 mm sieve.
- PARAGRAPH 9.2.2. Intrusion depth: The uptake of clay is based on the relationship between sediment velocity and particle size of the particles and can occur either at a varying insertion depth (10 ± 0.5 cm) at a predetermined time point at a specified temperature or at a fixed insertion depth (10 ± 0.1 cm) at a varying time point at a specified temperature (see Table 3 in ISO standard).
- § 9.2.10 drying oven set at a temperature of 105 °C ± 5 °C
- § 9.6 removal of organic matter (mandatory)
  - Washing to conductivity < 40 mS/m is optional. A washing step is performed after the removal of the carbonates and thus before the dispersion step.
  - After removal of the organic matter, the supernatant fluid 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 any)
  - The presence of carbonates can be tested by adding a few drops of HCl to a small amount of the sieved sample.
  - Waxes to conductivity < 40 mS/m
- § 9.9 removal of iron oxide: not applicable

§ 9.11 NAT sieves at 63 µm: replaced by sieves at 50 µm

3 CALCULATION

The% sand,% loam and% clay are calculated.

Based on the soil texture triangle, the main texture class (sand, sandy loam, clay and loam) is determined.

4 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

Soil – Reporting

1 GENERAL

The reporting shall be carried out in accordance with BAM/part 8/20.

The sampling data recorded cf. BAM/part 1/01 – point 5.2 shall be added to or incorporated into the analysis report.

Without prejudice to the provisions of BAM/part 8/20, the analytical report shall include:

a. Laboratory letterhead paper with at least name, address, telephone, e-mail
b. Unique report number
c. Unique sample number and, if applicable, SMIL steel composition number (s)
d. Date of sampling
e. Identification of the sampler (e.g. initials, identification code, SMIL steel nemer number). If the sample has not been taken by a sampler registered with the laboratory, this should be explicitly mentioned in the analytical report.
f. Sampling depth (s)
g. Date of receipt of the sample by the laboratory
h. Date of preparation of sample for analysis
i. Date on which the analytical report was sent
j. Name and signature of the person in charge of the laboratory (possibly digitally)
k. Name and address of the person to whom the report is delivered

2 NAME OF WEBSITE NOTIFICATION (SMIL)

Data on sampling, analysis results and GPS data catalogues are reported to the Flemish Land Agency via the SMIL application in accordance with the provisions in BAM/part 8/03.

Feeding-stuffs – Scope

The methods relate to the sampling and analysis of livestock feed as provided for in the Decree of 22 December 2006 concerning the protection of waters against pollution caused by nitrates from agricultural sources (hereinafter referred to as the Fertiliser Decree) and its implementing decrees:

The Executive Laboratory shall ensure that sampling and analysis is always carried out according to the methodology described below and shall be responsible for this.

The methods are taken from the European Directives on sampling and analysis methods for the official control of feedingstuffs and supplemented by methods developed within CEN/TC 327 and ISO TC 34/SC 10 *Animal feeding stuffs*.

Fodder – Sampling

CONTENT

1 Principle 3

2 Sampling of concentrates 3

3 Sampling of coarse feedingstuffs 3

3.1 Material 3

3.2 Practical implementation 4

4 Identification of samples 4

5 Sample preservation during transport 4

1 PRINCIPLE

Sampling must be carried out in such a way that a representative sample is obtained.

2 SAMPLING OF CONCENTRATES

Sampling may be carried out at the premises of the user or at the premises of the supplier or producer of the power plant.

Sampling can easily be carried out by collecting the subsamples with a scoop or container.

For the sampling of a batch of concentrated feed, a minimum of 5 subsamples shall be taken throughout the batch. In the case of packaged concentrates, the subsamples shall be collected from individual packages.

The subsamples shall be at least 500 g and shall be mixed into a composite sample. From this composite sample, a laboratory sample of minimum size is prepared by quartering.
500 g. The laboratory sample shall be collected in a glass or plastic container, well sealed.

3 SAMPLING OF COARSE FEEDINGSTUFFS

3.1 MATERIAL

The sampling of coarse fodder, whether stored or transported, shall be carried out using a dry and clean pitch consisting of a sample tray and a sample lid (Figure 1) or a coarse coarse fodder boron consisting of a hollow tube with a cutting head and an inner rod.

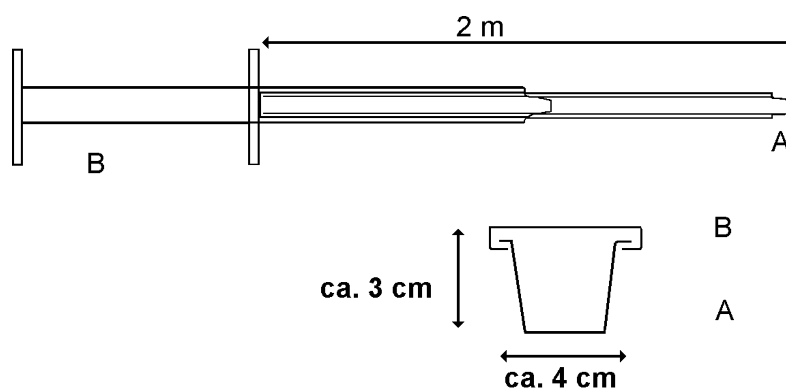


Figure 1 Sampling slurry for coarse fodder

3.2 Practical implementation

With the pitch or coarse fodder boron, at least 10 pitch samples over the full depth shall be taken proportionally over the load or the stack of stored coarse fodder.

A random sample with the pitch is taken by pushing the channel as deep as possible into the coarse fodder. The lid is then placed over the channel and the entire pitch is pulled out of the cargo of roughage. The channel is emptied in a dry and clean bucket, tray or crusher.

The random samples taken with the pitch shall be thoroughly mixed. A sample of about 1 litres is then taken from that mixture and placed in a dry and clean container (plastic or glass) that can be closed well.

Sampling takes place during the loading or unloading of a cargo.

4 IDENTIFICATION OF SAMPLES

The tag (number, barcode...) of the sample must be unambiguous so that no afterwards misunderstandings may arise as to the origin of the sample.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be provided on the sampling form accompanying the sample:

a. client;
b. client or third parties present at the sampling (Y/N);
c. type of concentrate (+ supplier or producer with their approval number or registration number assigned by FASFC) or type of coarse fodder;
d. animal category for which the feed is intended, if known;
e. method of sampling (for example, landing samples) + number of subsamples;
f. estimated volume of stored concentrate/coarse fodder;
g. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number);
h. place and date of sampling;
i. description of sampling location (e.g. shed, packaging, transport ...);
j. analyses to be carried out.

The laboratory's sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

5 MONSTERCONSERVATION DURING TRANSPORT

For moisture-rich feeds (e.g. coarse feeds and bowls), the sample is kept refrigerated pending and during transport to the laboratory.

Animal feed – Sample pre-treatment

CONTENT

1 Preparation of samples for analysis 3

1.1 Goal 3

1.2 Precautions 3

1.3 Practice 3

1.3.1 Feedingstuffs which can be ground as such 3

1.3.2 Feed that can be ground after drying 4

1.3.3 Liquid or semi-liquid feedingstuffs 4

1.3.4 Other feed 4

1.4 Retention of samples 4

2 Quality check 4

1 PREPARATION OF SAMPLES FOR ANALYSIS

1.1 D OEL

The procedures described below relate to the preparation for analysis of samples as described in Annex II to Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed.

These samples shall be prepared in such a way that the quantities weighed for carrying out the methods of analysis are homogeneous and representative of the final samples.

Representative sub-samples shall be provided for the determination of:
- moisture content at 105 °C, crude protein: fresh sample;
- total phosphorus: fresh sample or sample dried at 105 °C.

Note: The test sample for the determination of moisture content may continue to be used for the determination of total phosphorus on a dried sample.

1.2 PRECAUTIONS

All necessary operations must be carried out in such a way as to avoid, as far as possible, contamination of the sample and changes in composition. Grinding, mixing and sieving shall be carried out as quickly as possible with minimal exposure of the sample to the air and light. Mills and grinders likely to appreciably heat the sample shall not be used. For feed which is particularly sensitive to heat, manual grinding is recommended. Care should also be taken to ensure that the grinder itself is not the cause of contamination with trace elements.
If preparation cannot take place without significant changes in the moisture content of the sample, the moisture content must be determined before and after preparation using the method laid down in BAM/Part 2/03.

1.3 PRACTICE

Mix the final sample thoroughly, either mechanically or manually. Divide the sample into two equal portions (if possible by the four-part method). Keep one of the portions in a suitable clean, dry container, fitted with an air-tight stopper, and prepare the other portion or a representative part of it, of at least 100 g, as indicated below.

1.3.1 FEEDINGSTUFFS WHICH CAN BE GROUND AS SUCH

Unless otherwise specified in the methods of analysis, sieve the whole sample through a sieve with apertures of 1 mm (in accordance with recommendation ISO R.565). Repeat as necessary. Avoid any over grinding.

Mix the sieved sample and collect it in a suitable clean, dry container fitted with an air-tight stopper. Mix again, immediately before weighing out the amount for analysis.

1.3.2 FEED THAT CAN BE GROUND AFTER DRYING

Unless otherwise specified in the analytical methods, dry the sample so that its moisture content is reduced to 8-12 %, in accordance with the preliminary drying method, see BAM/deel2/03. Then proceed as indicated in section 1.3.1.

1.3.3 LIQUID OR SEMI-LIQUID FEEDINGSTUFFS

Collect the sample in a suitable clean, dry container, fitted with an air-tight stopper. Mix thoroughly, just before weighing the quantity for analysis.

1.3.4 OTHER FEED

Samples which cannot be prepared by one of the above methods must be treated by another method which ensures that at least the quantities weighed for analysis are homogeneous and representative of the final samples.

1.4 RETENTION OF SAMPLES

Keep the samples at a temperature that will not affect their composition. Samples intended for the analysis of vitamins or products particularly sensitive to light shall be stored in brown glass vessels.

2 QUALITY CHECK

As a quality control, at least 50 duplicate samples shall be analysed for each parameter per day or per batch of 1 samples started. For this purpose, 2 sub-samples are taken after sample pre-treatment and go through the entire analytical route.

Feeding-stuffs – Feed content

CONTENT

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2 Principle______3

3 Material______3

4 Implementation______4

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4.1.1 Feedingstuffs other than those listed in points 4.1.2 and 4.1.3______4

4.1.2 Cereals and Grütze______4

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4.2 Drying 5

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4.2.2 Cereals, flour, groats and meal______5

4.2.3 Compound feedingstuffs containing more than 4 % sugars derived from sucrose or lactose and the following straight feedingstuffs: Straight feedingstuffs such as locust beans, hydrolised cereal products, malt seeds, dried beet chips, fish and sugar solubles; compound feedingstuffs containing more than 25 % of mineral salts including water of crystallisation.____5

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1 PURPOSE AND SCOPE

This method makes it possible to determine the moisture content of feedingstuffs. It does not cover the analysis of milk products as feed materials, the analysis of mineral substances and mixtures composed predominantly of mineral substances, the analysis of animal and vegetable fats and oils or the analysis of the oil seeds and oleaginous fruit. The determination of moisture content in animal and vegetable fats and oils is described in Annex III (B) to Regulation (EC) No 152/2009.

2 PRINCIPLE

The sample is dried under certain conditions depending on the nature of the feed. Weight loss is determined by weighing. In the case of solid feedingstuffs with a high moisture content, drying must be carried out first.

3 MATERIAL

3.1 Windmill

Mill of a material that does not absorb moisture, that is easy to clean and that allows rapid and even grinding without producing significant heat, that is as close as possible to the outside air and that meets the requirements set out in point
4.1.1 and 4.1.2 (for example, microcross impact, water cooled, demountable ball, slow-moving ball and toothed disc mills).

3.2 Analytical balance

Analytical balance with a sensitivity of 0.5 mg or more.

3.3 Soccer boxes

Soak boxes made of stainless metal, glass or aluminium with airtight sealing lids and having a working surface allowing the sample to be divided at a rate of approximately 0.3 g per cm^2.

3.4 Ear cream receptacles

For example, 20 × 12 cm aluminium tray with 0.5 cm rim or other suitable containers.

3.5 Electric drying oven

Electrically heated oven with thermostat (± 5 °C), allowing rapid temperature control and good ventilation. [1]

3.6 Vacuum oven, electric

Vacuum oven, electrically heated, with thermostat and oil pump, fitted with a device for supplying dried hot air or fitted with a desiccant (for example, calcium oxide).

3.7 Desiccator

Desiccator with a thick perforated metal or porcelain plate and an effective desiccant.

4 IMPLEMENTATION

N.B.: The operations described in this section must be carried out immediately after opening the packages of samples. The analyses must be carried out at least in duplicate.

4.1 PREPARATION

4.1.1 FEEDINGSTUFFS OTHER THAN THOSE LISTED IN POINTS 4.1.2 AND 4.1.3

Take at least 50 g. Fine appropriately, if necessary, in such a way as to avoid any change in moisture content.

4.1.2 CEREALS AND GRÜTZE

Take at least 50 g of particles of such size that at least 50 % pass through a 0.5 mm sieve and no more than 10 % remain on a 1 mm round-meshed sieve.

4.1.3 LIQUID OR GREY FEEDING-STUFFS, FEEDING-STUFFS CONSISTING ESSENTIALLY OF FAT

Take about 25 g weighed to the nearest 10 mg, add an appropriate quantity of anhydrous sand weighed to the nearest 10 mg and mix until a homogeneous product is obtained.

[1] for drying cereals and by-products of cereal processing, the heating capacity of the oven must be such that, if it is pre-set at a temperature of 131 °C, it will return to that temperature within 45 minutes of the maximum number of samples being applied. Ventilation shall be such that when all samples of common wheat, which may contain the oven, are dried for 2 hours at the same time, the results obtained are less than 0.15 % of those obtained after drying for 4 hours.

4.2 Drying

4.2.1 Feedingstuffs other than those listed in points 4.2.2 and 4.2.3

Weigh a container with its lid to the nearest 0.5 mg. Weigh into the weighed container to the nearest 5 mg about 1 g of the sample and spread evenly. Place the container without lid in a oven preheated to 105 °C. To prevent the temperature from falling too much, the container must be placed in the oven as soon as possible. Leave to dry for 4 hours reckoned from the time when the oven temperature returns to 105 °C.

After opening the oven, seal the container with its lid, remove it from the oven, leave to cool for 30 to 45 minutes in the desiccator and weigh to the nearest 1 mg.

Samples consisting mainly of fat shall be dried again in the oven at 105 °C for 30 minutes.

The difference between the two weighings must not exceed 0.1 % of moisture.

4.2.2 Cereals, flour, groats and meal

Weigh a container with its lid to the nearest 0.5 mg. Weigh to the nearest 5 mg approximately 1 g of the crushed sample into the weighed container and spread evenly. Place the container without lid in a oven preheated to 130 °C. To prevent the temperature from falling too much, the container must be placed in the oven as soon as possible.

Leave to dry for 2 hours reckoned from the time when the oven temperature returns to 130 °C.

After opening the oven, seal the container with its lid, remove it from the oven, leave to cool for 30 to 45 minutes in the desiccator and weigh to the nearest 1 mg.

4.2.3 Compound feedingstuffs containing more than 4 % sugars derived from sucrose or lactose and the following straight feedingstuffs: Straight feedingstuffs such as locust beans, hydrolised cereal products, malt seeds, dried beet chips, fish and sugar solubles; compound feedingstuffs containing more than 25 % of mineral salts including water of crystallisation.

Weigh a container with its lid to the nearest 0.5 mg. Weigh to the nearest 5 mg approximately 1 g of the crushed sample into the weighed container and spread evenly. Place the container without lid in the vacuum oven preheated to between 80 °C and 85 °C. To prevent the temperature from falling too much, the container must be placed in the oven as soon as possible. Set the pressure to 10 cm of mercury and dry the sample for 4 hours at that pressure, either in a dry warm air supply or using a desiccant (about 300 g for 20 samples). In the latter case, the connection to the vacuum pump is broken when the required pressure is reached. Calculate the drying time from the time when the oven temperature returns to 80 to 85 °C. After the end of the drying period, gently return the pressure in the oven to that of the outside air.

After opening the vacuum oven, close the container with its lid, remove it from the oven, leave to cool for 30 to 45 minutes in the desiccator and weigh to the nearest 1 mg. Leave to dry for 30 minutes under vacuum in the oven at 80 to 85 °C and weigh

again. The difference between the two weighings must not exceed 0.1 % of moisture.

4.3 Pre-stirring

4.3.1 Feedingstuffs other than those listed in point 4.3.2

Solid feedingstuffs with a high moisture content, which are difficult to crush, are pre-dried as follows.

Weigh, to the nearest 50 mg, 10 g of the unground sample (pressed feed or feed in lumps if necessary) into a suitable pre-drying container. Dry in an oven at a temperature of 60 to 70 °C until the moisture content has been reduced to between 8 % and 12 %. Remove the receptacle from the oven and allow to cool uncovered for 1 hours in the laboratory; weigh to the nearest 10 mg. Crush immediately as indicated in 4.1.1 and dry as indicated in 4.2.1 or 4.2.3 according to the nature of the feed.

4.3.2 G recognises

Grain with a moisture content of over 17 % must be subjected to preliminary drying as follows:

Weigh, to the nearest 50 mg, 10 g of unground grain into a suitable pre-drying container. Dry in an oven for 5 to 7 minutes at 130 °C.

Remove the receptacle from the oven and allow to cool uncovered for 2 hours in the laboratory; weigh to the nearest 10 mg. Fine immediately as described in 4.1.2 and dry as described in 4.2.2.

5 CALCULATING OF RESULTS

The moisture content as a percentage of the sample is given by the following formulae:

5.1 Drying with preliminary drying

$$\% \text{ MOISTURE} = \frac{(M - M_0)}{M} \times 100$$

with:
m: initial weight, in grammes, of the test sample,
m_0: weight, in grammes, of the dry test sample.

5.2 Drying by pre-drying

$$\% \text{ MOISTURE} = \left[\frac{(M_2 - M_0) \times M_1}{M_2} + M - M_1\right] \times \frac{100}{M} = 100 \times \left(1 - \frac{M_1 \times M_0}{M \times M_2}\right)$$

with:
m: original mass of the sample (g) m_1: mass of the sample after pre-stirring (grams) m_2: mass of the sample after crushing (grams) m_0: mass of the dried sample (grams)

6 REMARK

If the sample has to be crushed and if this results in a change in the moisture content of the product, then the analytical results, which relate to the constituents of the feed, must be converted to the moisture content of the original sample.

Feeding-stuffs – Total phosphorus

CONTENT

1 Principle 3

2 Unsealing methods 3

3 Reporting 4

4 Reference 4

1 PRINCIPLE

The analyses shall be carried out on the refined sample (< 1 mm) obtained after sample pre-treatment as described in BAM/Part 2/02.

For the determination of phosphorus in feed, the sample is first destroyed. The phosphorus content in the solubilising solution is then determined.

The following methods of analysis have been developed within CEN/TC 327 and ISO TC 34/SC 10 *Animal feeding stuffs* and may be used for the determination of phosphorus in feed:

- **a.** ISO 6491: 1998 Animal feeding stuffs – Determination of phosphorus content – Spectrophotometric method: this method describes a digestion either by dry digestion at 550 °C and digestion with HCl and HNO_3 on a heating plate (organic feedingstuffs free of phosphates) or by wet digestion with H_2SO_4 and HNO_3 (especially in the case of mineral substances and liquid feedingstuffs). The digestion solution is treated with vanadate molybdate reagent. The optical density of the yellow-coloured solution formed is measured by means of a spectrophotometer at 430 nm;
- **b.** NBN EN 15510: 2008 Animal feeding stuffs – Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, Molybdenum, arsenic, lead and cadmium by ICP-AES (idem ISO 27085: 2009): this method describes a digestion either by dry digestion at 450 °C and digestion with HCl on a heater plate (organic feedingstuffs free of phosphates) or by wet digestion with HCl on a heater plate (mineral substances and organic feedingstuffs containing phosphates). The phosphorus content is then measured using ICP-AES;
- **c.** NBN EN 15621: 2012 Animal feeding stuffs – Determination of calcium, sodium, phosphorus, magnesium, potassium, sulphur, iron, zinc, copper, manganese and cobalt after pressure digestion by ICP-AES: this method describes a pressurised digestion using a microwave oven or a high-pressure gas. The acid mixture used is HNO_3/h_2O_2 or HNO_3. The phosphorus content is then measured using ICP-AES.

2 ONTSLUTION SMETHODES

The opening methods described in the standard methods above apply. In addition, the following breakdowns may also be used.

a. Digestion with $HNO_{3/HCl}$, with a heatable rendering block with rendering tubes fitted with a compact condenser.

Note: Alternatively, a watch glass or a shut-off cap (tightening and half turn back) may be used for the condenser.

Weigh to the nearest 1 mg approximately 1 g of fresh or dried test sample into a rendering tube. Gradually add 4 ml of 14M HNO_3 and 12 ml of 12M HCl.

Place the condenser on the rendering tubes. Leave rendering tubes at room temperature to allow a slow reaction of the organic matter. Then carry out the rendering programme with incremental warming, for example:

a. warm up in 20 minutes to 45 °C, 5 minutes at 45 °C;

b. warm up in 10 minutes to 65 °C, 10 minutes at 65 °C;
c. warm up to 105 °C for 120 minutes at 105 °C.

Make up to 50 ml with ultra pure water.

The phosphorus content in the solubilising solution is measured with ICP-AES according to EN 15510 or EN 15621.

b. Digestion by ashing at 550 °C and digestion with HNO_3 in a hot water bath (organic feeding-stuffs, phosphate-free)

Weigh, to the nearest 1 mg, 2,5 to 1 g (m) of fresh or dried sample.

Ash that sample at (550 ± 25) °C for 4 hours. The axle shall be grey white. If the ash is not white: add a few drops of 14M HNO_3 and ash again for 1 hours.

Quantitatively transfer the ash into a 100 ml beaker containing 20 ml 1M HNO_3 1M. Leave to distil for one hour on a heater or in a hot water bath.

Filter and collect filtrate in a 100 ml volumetric flask and rinse well with 1M HNO_3. Make up to 100 ml with 1M HNO_3.

The phosphorus content in the solubilising solution is measured with ICP-AES according to EN 15510 or EN 15621.

3 REPORTING

The total phosphorus content shall be reported in% P on fresh sample.

4 REFERENCE

a. Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed, Annex III, Part P, https://eur-lex.europa.eu/legal-content/NL/TXT/PDF/?uri=CELEX:32009R0152&from=nl
a. ISO 6491: 1998 Animal feeding stuffs – Determination of phosphorus content – Spectrophotometric method.
b. NBN EN 15510: 2008 Animal feeding stuffs – Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, Molybdenum, arsenic, lead and cadmium by ICP-AES (idem ISO 27085: 2009).
c. NBN EN 15621: 2012 Animal feeding stuffs – Determination of calcium, sodium, phosphorus, magnesium, potassium, sulphur, iron, zinc, copper, manganese and cobalt after pressure digestion by ICP-AES.
d. C. Vanhoof, F. Beutels, K. Duyssens and K. Tirez, *Evaluation of the method for determining phosphorus in feed*, VITO report 2012/MANT/R/104.

Feeding-stuffs – Crude protein

CONTENT

1 **Principle** 3

2 **Reagents** 3

3 **Equipment** 3

4 **Practice** 4

4.1 Dilated 4

4.2 Distillation 4

4.3 Titration 4

4.4 Blank test 4

5 **Calculating of results** 4

6 **Reference** 5

1 PRINCIPLE

The crude protein content of feedingstuffs is determined on the basis of the nitrogen content according to the kjeldahl method.

The analyses shall be carried out on the refined sample (< 1 mm) obtained after sample pre-treatment as described in BAM/Part 2/02.

The sample is digested by sulphuric acid in the presence of a catalyst. The acid solution is made alkaline with sodium hydroxide solution. The ammonia is distilled and collected in a suitable absorbent, depending on the method of determination chosen. The determination of the ammonium content may be carried out:

a. by backtitration with a standard solution of sodium hydroxide;
b. according to NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection;
c. in accordance with ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method;
d. in accordance with ISO 15923-1: 2013 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection.

This procedure describes the titrimetric method.

2 REAGENTS

a. potassium sulphate, K_2SO_4;
b. catalytic converter: cupric oxide (CuO) or cupric sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$). Other commercially available catalytic converters suitable for this determination are also permitted;
c. zinc grains;
d. sulphuric acid 20 = 1.84 g/ml;
e. sulphuric acid c (H_2SO_4) = 0.5 mol/l;
f. sulphuric acid c (H_2SO_4) = 0.1 mol/l;
g. methyl red indicator: Dissolve 300 mg methyl red in 100 ml ethanol, = 95-96 % (v/v);
h. sodium hydroxide solution (m/v: 40 %), technical quality is sufficient, = 40 g/100 ml (m/v: 40 %);
i. sodium hydroxide solution, c = 0.25 mol/l;
j. sodium hydroxide solution, c = 0.1 mol/l;
k. granules of pumice stone washed and annealed with hydrochloric acid;
l. acetanilide (sm.p. = 114 °C; N = 10.36 %);
m. sucrose (nitrogen free).

3 EQUIPMENT

Equipment suitable for carrying out digestion, distillation and titration by the kjeldahl method.

4 PRACTICE

4.1 DILATED

Weigh, to the nearest 1 g, 0,001 g of the sample and place it in the receptacle of the digestion apparatus. Add 15 g of potassium sulphate, an appropriate quantity of catalyst (0,3 to 0.4 g of cupric oxide or 0,9 to 1.2 g of cupric sulphate pentahydrate), 25 ml of sulphuric acid (20) and a few granules of pumice stone; mix the whole. First heat the container gently under occasional stirring, if necessary, until the mass is carbonised and the foam has disappeared; heat more vigorously until the liquid boils regularly. Heating is sufficient if the boiling acid condenses against the wall of the container. Make sure that the wall does not overheat and that no organic matter is attached to it. Boil for two hours after the solution has become clear and light green. Leave to cool.

4.2 DISTILLATION

Add carefully enough water to ensure complete dissolution of the sulphates. Leave to cool, add a few granules of zinc.

Place in the receiver of the distillation apparatus an accurately measured quantity of 25 ml of sulphuric acid (0.5 mol/l or 0.1 mol/l) depending on the expected nitrogen content. Add a few drops of methyl red.

Connect the digestion container to the condenser of the distillation apparatus and ensure that the end of the condenser tube is at least 1 cm below the surface of the liquid in the collecting flask. Slowly pour 100 ml sodium hydroxide solution (40 %) into the digestion container, without loss of ammonia.

Heat the container until all ammonia is overdistilled.

4.3 TITRATION

Titrate the excess sulphuric acid in the collecting flask again with sodium hydroxide solution (0.25 mol/l or 0.1 mol/l) depending on the concentration of the sulphuric acid used, until the end point is reached.

4.4 BLANK TEST

Carry out a blank test (digestion, distillation and titration) using, for example, 1 g of sucrose instead of the sample.

5 CALCULATING OF RESULTS

Calculate the crude protein content in% on fresh sample using the following formula:

$$\frac{(R_0 \quad V_1)\ c\ 0{,}014\ 100\ 6{,}25}{m}$$

with:

V_0: volume (ml) of NaOH (0.25 mol/l or 0.1 mol/l) consumed in the blank test;
V_1: volume (ml) of NaOH (0.25 mol/l or 0.1 mol/l) consumed during sample titration; c: concentration (mol/l) of sodium hydroxide (0.25 mol/l or 0.1 mol/l);
m: mass (g) of the sample.

6 REFERENCE

a. Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed, Annex III, Part C, https://eur-lex.europa.eu/legal-content/NL/TXT/PDF/?uri=CELEX:32009R0152&from=nl
b. ISO 5983-1 Animal feeding stuffs – Determination of nitrogen content and calculation of crude protein content – Part 1: Kjeldahl method.
c. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection.
d. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method.
e. ISO 15923-1: 2013 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection.
f. NEN 6604: 2007 Water – Determination of ammonium, nitrate, nitrite, chloride, orthophosphate, sulphate and silicate content using a discrete analytical system and spectrophotometric detection.

Feed – Reporting

1 GENERAL

The reporting shall be carried out in accordance with BAM/part 8/20. The sampling report drawn up on the basis of the field records (sampling form) shall be added to the analysis report or incorporated into the analysis report.

Without prejudice to the provisions of BAM/part 8/20, the analytical report shall include the following information:

a. laboratory letterhead paper with at least name, address, telephone, e-mail;
b. unique report number;
c. unique sample number and, if applicable, sample number assigned by the manure bank (if applicable);
d. date of sampling;
e. Identification of the sampler (e.g. initials, identification code, SMIL steel nemer number). If the sample has not been taken by a sampler attached to the laboratory, this should be explicitly mentioned in the analytical report;
f. client present at sampling (Y/N);
g. description of the place of sampling (e.g. shed, packaging, transport, etc.);
h. the type of concentrated feed (+ supplier or producer with their approval number or registration number assigned by FASFC) or type of coarse fodder;
i. animal category for which the feed is intended, if known;
j. date on which the sample was received by the laboratory;
k. the date on which the sample was taken for analysis;
l. date on which the report was sent;
m. name and signature of the person in charge of the laboratory (possibly digitally);
n. name and address of the person to whom the report is delivered.

2 UNITS

Moisture content: in%
Protein content: in%
Total phosphorus: in%

Liquid livestock manure – Scope

The methods relate to the sampling and analysis of liquid livestock manure as provided for in the Decree of 22 December 2006 concerning the protection of waters against pollution caused by nitrates from agricultural sources (hereinafter referred to as the Fertiliser Decree) and its implementing decrees.

'Livestock manure' means both livestock excrements (whether or not mixed with litter) and all intermediate or final products resulting from a physical, chemical or microbiological (production) process in which livestock excrements (whether or not mixed with litter) are involved, irrespective of their proportion.

The raw, untreated excrements of livestock (whether or not mixed with litter) are hereinafter referred to as 'manure'. All final and intermediate products resulting from a physical, chemical or microbiological (production) process in which manure was a raw material are hereinafter referred to as 'treated manure'.

"Liquid" livestock manure means:

1. liquid manure with a dry matter content of less than 30 %;
2. liquid treated manure with a dry matter content of less than 15 %.

For sampling of liquid manure from manure cellars and for (simulation of) manure transport, the methods described in BAM/part 3/01- A and BAM/part 3/01-B shall apply. For the sampling of liquid treated manure and liquid manure from an external manure storage (pools, lagoons, silos...), the methods described in CMA [1] or WAC [2]shall be applied taking into account BAM/part 3/01-C.

For sample preparation of liquid manure, the methods described in BAM apply. Samples with a dry matter content between 15 and 30 % can be classified with both liquid and solid manure. The subdivision of the laboratory sample delivered into the relevant matrix type and the related sample pre-treatment can be carried out based on the estimated dry matter content combined with a visual assessment. However, the physical state based on the visual observation is determinative for carrying out sample pre-treatment.

For the sample preparation of liquid treated manure, the methods described in CMA apply, taking into account BAM/part 3/02.

For the analysis of liquid manure, the methods described in BAM apply. For the analysis of liquid treated manure, both BAM and CMA methods may be used. For the analysis of liquid streams with a dry matter content < 2 % (such as effluents, effluents, biopurification effluents, washing waters and grease waters), BAM/deel3/07 shall apply. If the CMA methods of analysis are used, the results should be converted to the units as prescribed in the corresponding BAM methods.

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[1] compendium for sampling and analysis in implementation of the Materials Decree and the Soil Decree (https://emis.vito.be/nl/erkende-laboratoria/bodem-en-afvalstoffen-ovam/compendium-cma)

[2] compendium for water sampling, measurement and analysis (https://emis.vito.be/nl/erkende-laboratories/water gop/compendium wac)

Note: For BAM, in the case of liquid streams, a visual assessment may be used to estimate whether it is a product with a dry matter content greater or less than 2 % in order to determine whether it is used as ‘manure’ steel or as ‘water’ steel. A prior determination of the dry matter content for the choice of the analytical method or a re-analysis with the other method if the dry matter content is found to be above 2 % is not necessary

The following table gives an overview of the methods to be used for sample preparation and analysis in function of the matrix:

Parameter	**Liquid manure methods**	**Liquid treated manure methods**	**Methods for samples with a dry matter content < 2 %**
Sample preparation	BAM/part 3/02	CMA/5/B.1 and BAM/part 3/02	BAM/part 3/02
Dry matter content	BAM/part 3/03	BAM/part 3/03 (or CMA/2/IV/1)	BAM/part 3/07
Total phosphorus	BAM/part 3/04	BAM/part 3/04 (or CMA/2/IV/19)	BAM/part 3/07
Ammoniacal nitrogen	BAM/part 3/05	BAM/part 3/05 (or CMA/2/IV/6 § 5.8 + CMA/2/IV/7)	BAM/part 3/07
Total nitrogen	BAM/part 3/06	BAM/deel3/06 and CMA/2/IV/4	BAM/part 3/07

The implementing laboratory must ensure that sampling or analysis is always carried out in accordance with the methodology described and is responsible for this.

Liquid manure – Sampling of manure cellars and in simulated manure transport

CONTENT

1 Principle and scope 3

2 Hygiene measures 3

3 Equipment and materials 3

4 Sampling of a manure cellar 4

4.1 Scope 4

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5 Sampling in simulated manure transport 7

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1 PRINCIPLE AND SCOPE

This procedure describes the sampling of liquid manure from manure cellars and when simulating a transport by pumping/applying manure. Its purpose is to obtain a representative laboratory sample.

The following observations should be taken into account:

a. a sampling of liquid pig manure or mixture of liquid pig manure may only be carried out in the case of manure transport as described in BAM/Part 3/01-B The procedure for sampling in the case of simulation of a manure transport (tap sample) as described in point 5 may be used as an alternative. However, that procedure may only be applied by a VLAREL accredited laboratory in the manure discipline for the M-M1 package;
b. sampling of liquid pig manure from the manure cellar (well sample) in accordance with point 4 shall only be allowed in case of a stocktaking;

2 HYGIENE MEASURES

In the case of sampling, the sanitary rules in force at the farm must be complied with at the request of the client (e.g. boots by disinfecting bath, use of overalls on site, showering, etc.).

Both the protective clothing (overalls, footwear, etc.) and all sampling material must be clean when entering the farm in order to avoid cross-contamination from previously visited farms.

3 EQUIPMENT AND MATERIALS

Equipment and supplies shall, as far as possible, consist of materials which are inert to the component (s) to be analysed. They must be well maintained and clean so that the representativeness of the sampling is not adversely affected. Equipment and supplies must be cleaned regularly either mechanically or chemically. The appearance of, for example, doffy or discoloured spots may indicate that the device is no longer suitable for sampling.

Required material:

a. GPS logger or other device with built-in GPS function to record coordinates in WGS84 format, in decimal degrees to 5 decimal places;

b. leak-proof laboratory sample receptacles with a minimum volume of:
   - 500 ml in the case of sampling in accordance with point 4 (well sample);
   - 0,8-1 litres in the case of sampling according to paragraph 5. (tapping sample). If the sampler generates a tap sample of more than 650 ml, the volume of the receptacle shall be adjusted so as to provide approximately 20 % headspace. Exceptions may be made to this rule in the case of automatic samplers working with specific containers. Sample reduction is not permitted when taking a tap sample;
c. personal protective equipment;
d. buckets in PE or RVS as collection bucket;
e. pollen spoon;
f. refrigerated boxes with sufficient refrigeration elements or equipment to ensure refrigerated transport of samples;
g. specifically for the sampling of manure cellars as described in point 4: liquid layer sampler with a minimum internal diameter of 2.5 cm and a minimum length of 2 m;
h. specifically for sampling in simulated manure transport as described in paragraph 5.: a sampling device validated in accordance with BAM/Part 8/01. The correct operation of the sampler shall be checked at least every six months by carrying out the weighing test described in BAM/Part 8/01 § 3.3.1.1. The variation of the grip size expressed as the coefficient of variation calculated over a minimum of five grips shall not exceed 0,075 (7.5 %) as stated in § 3.2 of BAM/Part 8/01.
   Taps on filling pipes are not permitted.

4 SAMPLING OF A MESTKELDER

4.1 Scope

This method is only allowed for the sampling of bovine manure and in case of sampling in the framework of a pig manure stock determination.

4.2 Safety

During the storage of liquid livestock manure in the manure cellar, a decomposition layer occurs in the case of cattle manure, while in the case of pig and chicken manure a decomposition layer is formed. In order to obtain a homogeneous batch of liquid livestock manure, stirring or mixing plants are sometimes used. In this case, the duration of mixing prior to sampling must be recorded. Certainly during mixing, but also in stables above unmixed manure pits, gases may be released that are formed in the manure during storage. These gases may accumulate in less ventilated areas in the house and some of them are toxic (H_2S, NH_3) or flammable (CH_4). A risk of asphyxiation or explosion would then arise. Maximum ventilation is therefore absolutely necessary. During mixing, do not stay close to the pump hole or poorly ventilated areas in the house. This risk also exists in the case of non-mixing; please also note that some gases are heavier than air and are present in higher concentrations at low altitudes.

4.3 DISTRIBUTION OF SAMPLING POINTS

Samples are always taken at the rate of one laboratory sample per cellar. Therefore, when sampling, no composite samples may be taken or made up, nor may more than one cellar be sampled within one sample.

In order to overcome the heterogeneity of the lot, at least 10 random samples must be taken throughout the cellar. Mechanical mixing of the cellar prior to sampling is recommended.

The following guidelines shall be taken into account when selecting the places where a random sample is to be taken:

a. if several species or weight classes are present in the house, the number of stitches must be distributed in such a way that their relative proportions are reflected in the sample;
b. two random samples shall be taken in the service corridors between the compartments if they are submerged and accessible;
c. random samples should not be taken at the level of drinking nipples, brush wells, pump holes, feeding troughs or other locations where the composition may not be representative due to: infiltrating water, additional aeration and sweeper.

An example of a correct distribution of sampling points is given in Figure 1.

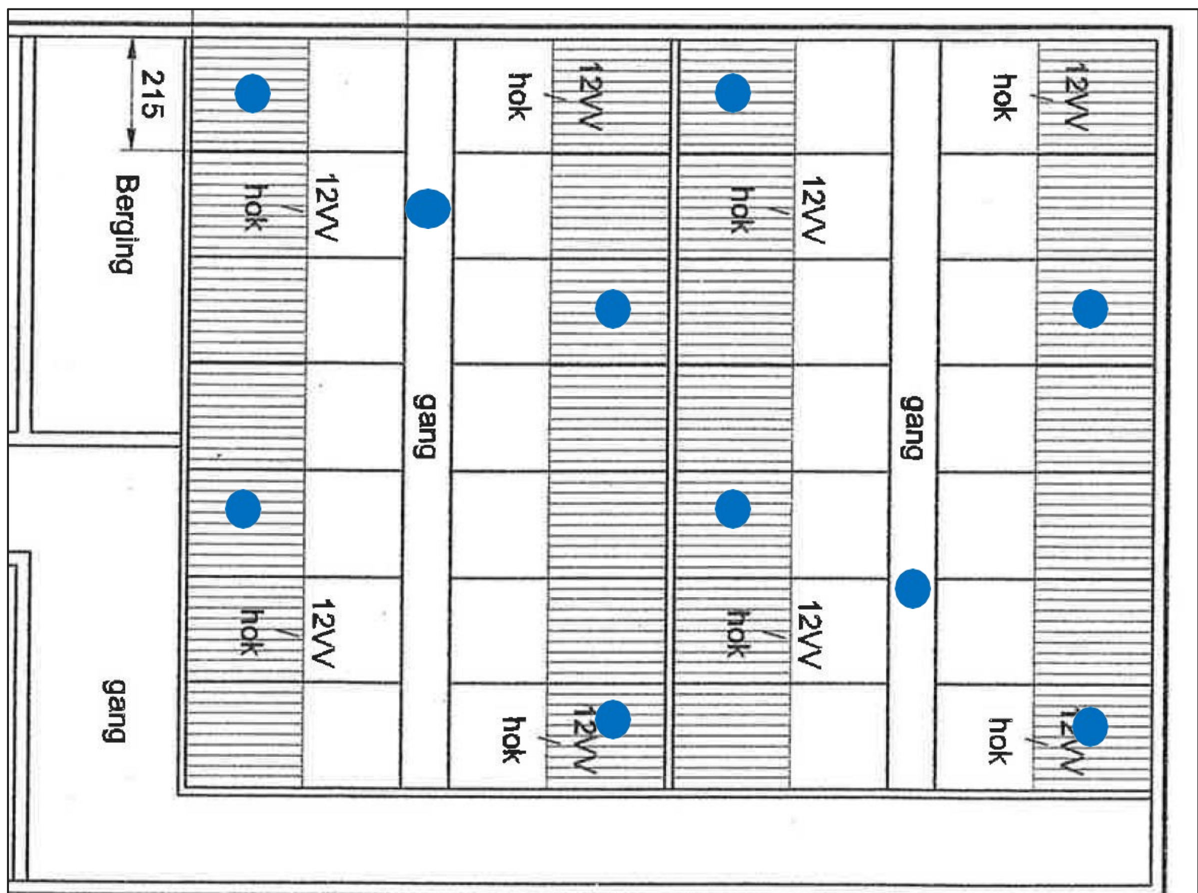

Figure 1: example of a distribution of sampling points across the house

4.4 IMPLEMENTATION

Sampling shall be carried out by the grilles or, if the grilles are not sufficiently wide, after lifting the grilles or by special openings in the grilles. In no case shall a rise tube of a smaller diameter be used or samples be taken from the pump hole.

Sometimes, when a manure cellar is emptied, the more solid settling layer is not removed. In such cases, they should not be included in the sampling. If the settling layer is removed, it should also be sampled with it. Whether or not the lower layer is to be sampled must be agreed with the client prior to sampling and recorded on the sampling form.

For the sampling of the manure in the manure cellar, the unsealed rising tube (see Figure 3) is placed on the bottom of the well. This should be done slowly so that all layers in the well are sampled. If the lower layer is not to be sampled, raise the tube to the required height. Close the tube by pulling the stopper into the tube aperture. Pull the tube out of the well and empty in a collecting bucket. Where appropriate, a multisampler [2] may also be used for sampling in a manure cellar.

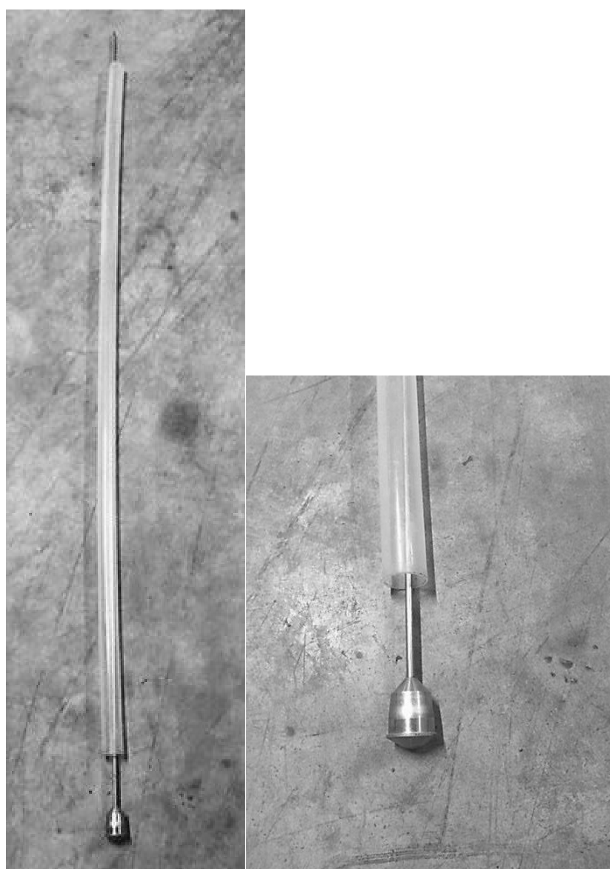

Figure 2: steam-operated liquid layer sampler

[2] see CMA/1/A.16 sampling techniques liquids (https://emis.vito.be/nl/referentielabo-ovam)

If desired, a sample reduction to about 500 ml may be carried out. Due attention shall be paid to the mixing of the field sample in order to avoid bagging during sub-sampling.

5 SAMPLING IN SIMULATED MANURE TRANSPORT

5.1 SCOPE

It is permissible to simulate transport by pumping the manure or redirecting it to another manure storage facility via an eel ton, such as, for example, an external tank, another manure cellar or another compartment in the house. That procedure may be used only with the express consent of the contracting authority. The client must also determine to which other manure storage the manure is to be transferred.

The manure shall under no circumstances be returned to the same manure storage facility as the one from which it is pumped during the simulation.

When simulating transport, at least 2 tapping samples shall be taken. The first tapping sample may be taken only after a minimum of 20 m^3 of manure has been pumped out. Between 2 tap samples, at least 20 m^3 of manure must also be pumped before taking a second tap sample. If the manure is directly pumped to another storage facility, the different partitions forming one tap sample must be taken over a period of time corresponding to pumping at least 20 m^3 of manure. If the adjustment to the other storage is carried out by means of an eel ton, the various partitions making up one tap sample shall be distributed regularly over the total loading time needed to adjust at least 20 m^3.

5.2 PRACTICAL IMPLEMENTATION

Sampling shall be carried out by manual or automated taking of a tap sample using a sampling device. The sampling is described below with a manual side-tube device. For other appliances, reference is made to the manufacturer's manual.

The side tube apparatus (Figure 3) consists of two closely fitting and partially open tubes. The sample chamber is bounded at the top by the pressure bar and at the bottom by a valve. Due to a rotating movement, the hollow, partially open tube takes a portion of manure from the manure stream. After opening the valve at the bottom of the tube, the pressure bar presses the manure in the sample inoculum.

When a sample is taken manually, the control levers are set to the initial position, which is pulled up the pressure bar, the side tube is turned off with the manure flow opening and the ball tap closed. A sample sampler shall be placed under the outlet. The side tube is rotated completely against the direction of flow of the manure. The ball cock is opened and the pressure bar is fully moved downwards. The expression bar is then pulled up and the ball tap closed.

The tap sample is taken by at least five times an amount of manure, distributed regularly over the time required to pump/reset at least 20 m^3 of manure (see Figure 4).

draining manure. During sampling, all other inlet or outlet outlets are necessarily closed. The tapping sample is collected in a dry, clean, empty and sufficiently large sample inoculum. The whole tag sample shall be considered to be a laboratory sample. No sample reduction shall be carried out. The minimum sample volume is 650 ml. If necessary, the number of sub-interventions should be increased. Where the amount of manure is applied to more than one tonne of eel, 3 tap samples shall be taken from each tonne of eel, evenly distributed over the application. The eel tonnes used must be of the same size.

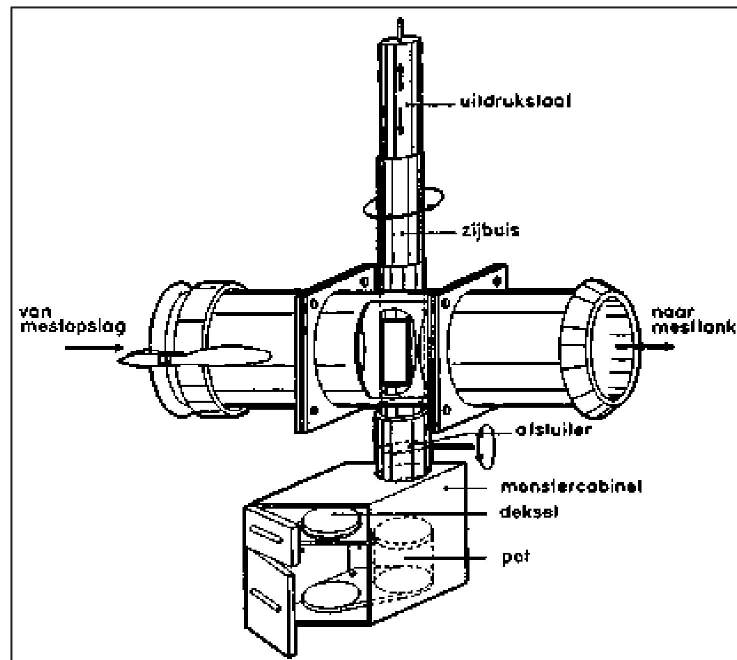

Figure 3: side-tube apparatus for manure sampling during transport

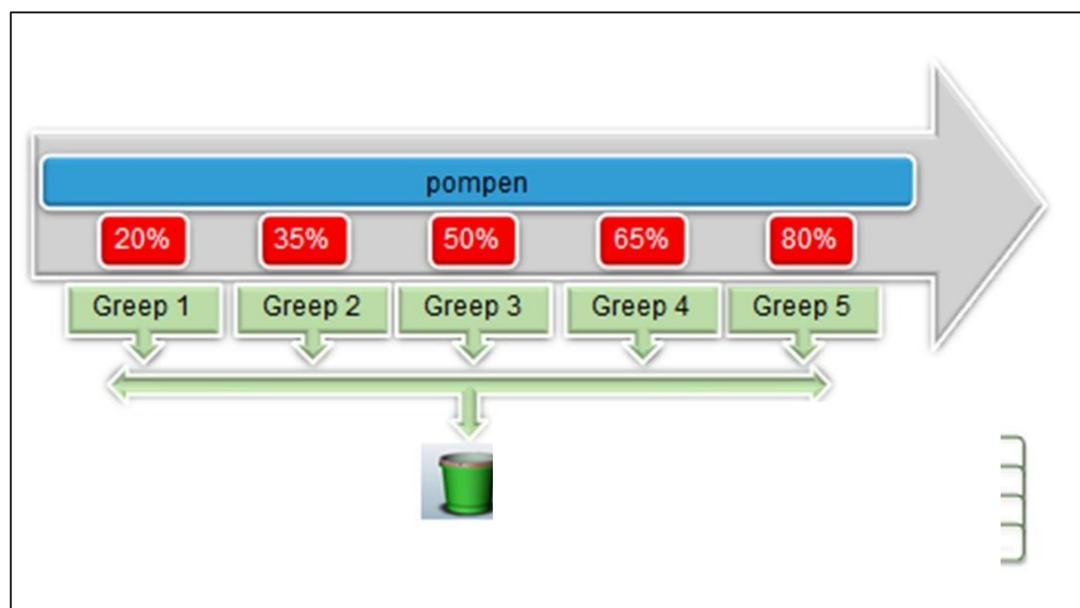

Figure 4: sampling in simulated transport: distribution of grips over the time needed to pump/reset at least 20 m^3 of manure

6 IDENTIFICATION OF SAMPLES

The tag (number, barcode, etc.) of the laboratory sample must be unambiguous so that no misunderstanding can subsequently arise as to the origin of the sample.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be indicated on the (digital) sampling form accompanying the sample:

a. client, address, agricultural number and operation number;
b. client or third parties present at the sampling;
c. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number;
d. date and time of sampling;
e. own sample number or sample coding;
f. type of manure (e.g. sow slurry, pig meat slurry, calf slurry...). The manure codes used by the Flemish Land Agency and included in SMIL must be used for this purpose;
g. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the house or manure storage. Those coordinates shall be determined locally by a GPS device;
h. description of the manure storage sampled (e.g. manure cellar, manure silo...);
i. the estimated volume of manure in the manure cellar/manure storage. Alternatively, for the purpose of estimating the volume of a manure cellar, the height of the manure in the manure cellar may also be determined using a probe and recorded together with the depth of the manure cellar;
j. the sampling equipment used (liquid layer sampler, multisampler, etc.). If a (automatic) side-tube device is used, this shall be indicated with its individual identification;
k. in case of sampling of a manure cellar:
   - sketch showing distribution of sampling points across basement if sampling was not carried out in accordance with the prescribed procedure;
   - volume of the field and laboratory sample if sample reduction was applied in situ;

   in case of simulation of manure transport:
   - or by pumping or resistance to a eel ton.
   - in the case of pumping, the volume after which the plug was taken shall be recorded.
   - In case of resistance with an eel ton, the volume of the eel ton, the number of tons moved, the tons sampled and the number of grips per ton are also recorded.
   - A description, including GPS coordinates, of the basement or storage to which the displaced manure was transported. The volume of storage and the amount of manure present before and after the simulation shall be reported.
   - the number of fractional bars needed to obtain the minimum required sample volume of 650 ml;
l. significant remarks and/or deviations that may affect the interpretation of the analytical result.

The laboratory's sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

7 MONSTERCONSERVATION

- The sample is stored chilled (5 ± 3) °C immediately after sampling. All transports must be refrigerated (with refrigeration box or refrigeration inside the wagon).
- Refrigeration must be traceable during storage.
- The sample must be prepared for analysis no later than the seventh day after sampling.

Liquid manure – Sampling in manure transport

CONTENT

1 **Principle and scope** 3

2 **Hygiene measures** 3

3 **Equipment and materials** 3

4 **Sampling during transport** 4

5 **Identification of samples** 6

6 **Sample preservation** 6

1 PRINCIPLE AND SCOPE

This procedure describes the sampling of liquid manure in the case of manure transport actually carried out. Its purpose is to obtain a representative laboratory sample.

The following observations should be taken into account:

- **a.** sampling of liquid pig manure or mixture of liquid pig manure may only be carried out in manure transport as described in this procedure. The procedure for sampling in simulation of a manure transport, as described in BAM/Part 3/01-A, point 5, may be used as an alternative. However, that procedure may only be applied by a VLAREL accredited laboratory in the manure discipline for the M-M1 package;
- **b.** a turbocharger shall not be used if the load is sampled;

2 HYGIENE MEASURES

In the case of sampling, the sanitary rules in force at the farm must be complied with at the request of the client (e.g. boots by disinfecting bath, use of overalls on site, showering, etc.).

Both the protective clothing (overalls, footwear, etc.) and all sampling material must be clean when entering the farm in order to avoid cross-contamination from previously visited farms.

3 EQUIPMENT AND MATERIALS

Equipment and supplies shall, as far as possible, consist of materials which are inert to the component (s) to be analysed. They must be well maintained and clean so that the representativeness of the sampling is not adversely affected. Equipment and supplies must be cleaned regularly either mechanically or chemically. The appearance of, for example, doffy or discoloured spots may indicate that the device is no longer suitable for sampling.

Required material:

- **a.** GPS logger or any other device with built-in GPS function to record coordinates in WGS84 format, in decimal degrees to 5 decimal places;
- **b.** leak-proof laboratory sample receptacles with a minimum volume of 0,8-1 litres. If the sampler generates a tap sample of more than 650 ml,

the volume of the container shall be adjusted to accommodate approximately 20 % headspace. Exceptions may be made to this rule in the case of automatic samplers working with specific containers. Sample reduction is not permitted when taking a tap sample;

c. personal protective equipment;
d. refrigerated boxes with sufficient refrigeration elements or equipment to ensure refrigerated transport of samples;
e. a sampling device validated in accordance with BAM/Part 8/01. The correct operation of the sampler shall be checked at least every six months by carrying out the weighing test described in BAM/Part 8/01 § 3.3.1.1. The variation of the grip size expressed as the coefficient of variation calculated over a minimum of five grips shall not exceed 0,075 (7.5 %) as stated in § 3.2 of BAM/Part 8/01.
   Taps on filling pipes are not permitted.

4 SAMPLING DURING TRANSPORT

Sampling may only be carried out when loading a load where a load consists of at least 20 m^3 of manure.

Sampling shall be carried out by manual or automated taking of a tap sample using a sampling device. The sampling is described below with a manual side-tube apparatus. For other appliances, reference is made to the manufacturer's manual.

The side tube apparatus (Figure 1) consists of two closely fitting and partially open tubes. The sample chamber is bounded at the top by the pressure bar and at the bottom by a valve. Due to a rotating movement, the hollow, partially open tube takes a portion of manure from the manure stream to the transport tank. After opening the valve at the bottom of the tube, the pressure bar presses the manure in the sample inoculum.

When a sample is taken manually, the control levers are set to the initial position, which is pulled up the pressure bar, the side tube is turned off with the manure flow opening and the ball tap closed. A sample sampler shall be placed under the outlet. The side tube is rotated around the direction of flow of the manure. The ball cock is opened and the pressure bar is fully moved downwards. The expression bar is then pulled up and the ball tap closed.

The tapping sample is taken by draining at least five times a quantity of manure, distributed regularly over the loading time of the tanker (see Figure 2). During sampling, all other inlet or outlet outlets are necessarily closed. The tapping sample is collected in a dry, clean, empty and sufficiently large sample inoculum. The whole tag sample shall be considered to be a laboratory sample. No sample reduction shall be carried out.

The volume of cargo, the number of partitions and the total loading time are recorded on the sampling form. The minimum sample volume is 650 ml. If necessary, the number of sub-interventions should be increased.

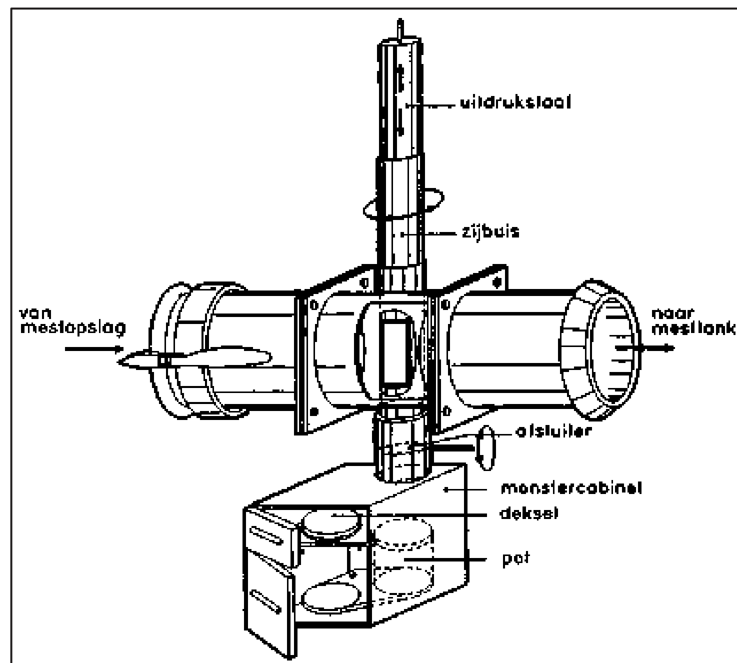

Figure 1: side-tube apparatus for manure sampling during transport

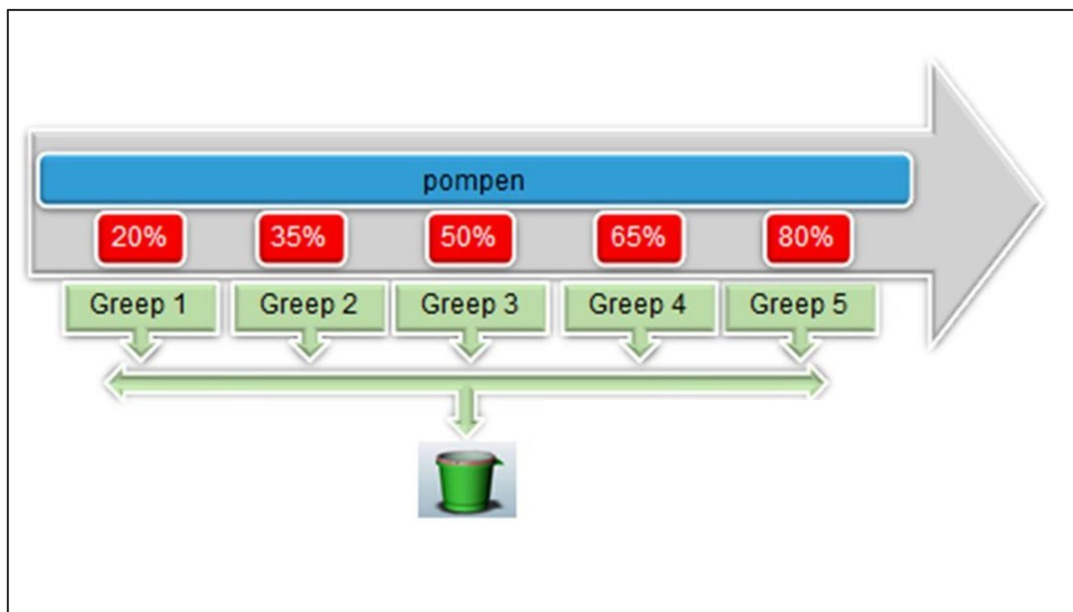

Figure 2: transport sampling: example of distribution of the grips over the entire load

5 IDENTIFICATION OF SAMPLES

The tag (number, barcode...) of the sample must be unambiguous so that no misunderstandings can subsequently arise as to the origin of the sample.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be indicated on the (digital) sampling form accompanying the sample:

a. client, address, farmer number and operator number;
b. client and/or third parties present at the sampling;
c. reference of MAD/Neighbouring scheme BR manure disposal document in case of cargo sampling;
d. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number);
e. date and time of sampling;
f. own sample number or sample coding;
g. type of manure (e.g. sow slurry, pig meat slurry, calf slurry...). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. The same description shall be used as the one used on the MAD, if applicable;
h. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the loading and sampling point. Those coordinates shall be determined locally by a GPS device;
i. description of the manure storage sampled (e.g. manure cellar, manure silo...);
j. The (automatic) side-tube device used shall be indicated with its individual identification;
k. the estimated volume of manure in the manure cellar/manure storage. Alternatively, for the purpose of estimating the volume of a manure cellar, the height of the manure in the manure cellar may also be determined using a probe and recorded together with the depth of the manure cellar;
l. ranking of the sampled cargo in the series of transported loads if several loads are transported over a period of one or more (consecutive days) from the same manure storage (based on the information provided by the client);
m. the number of fractional bars needed to obtain the minimum required sample volume of 650 ml;
n. significant remarks or deviations that may affect the interpretation of the analytical result

The laboratory's sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

6 MONSTERCONSERVATION

- The sample is stored chilled (5 ± 3) °C immediately after sampling. All transports must be refrigerated (with refrigeration box or refrigeration inside the wagon).
- Refrigeration must be traceable during storage.

- The sample must be prepared for analysis no later than the seventh day after sampling.

Liquid manure and liquid treated manure – Sampling of manure storage

CONTENT

1 Scope 3

2 Hygiene measures 3

3 Sampling of a vertical storage 4

4 Sampling at a tap 5

5 Sampling of lagoons 6

6 Sampling of manure bags 7

7 Identification of samples 7

8 Sample preservation 8

1 SCOPE

For the sampling of liquid treated manure (digestate streams, effluents, thin fraction after separation...) and of liquid manure from a manure store other than a manure cellar under the house, such as ponds, silos, lagoons, collecting basins..., reference is made to CMA/1/A.16 (liquid sampling techniques) and CMA/1/A.18 (on site sample pre-treatment) of the Compendium for sampling and analysis in implementation of the Materials Decree and the Soil Decree (https://emis.vito.be/nl/referentielabo-ovam) and to WAC/I/A/002 (instantaneous sampling (tap) of water) and WAC/I/A/003 (instantaneous sampling (landing sample) of water) of the Compendium for sampling, measuring and analysis of water (https://emis.vito.be/nl/lne-erkenningen-water), in particular in compliance with the provisions set out in this procedure.

The following deviation from CMA applies: if only the dry matter, nitrogen and phosphorus parameters are to be determined on the sample, the volume of the lab sample may be limited to 0,5 to 0.8 l.

Sampling of liquid pig manure or mixture of liquid pig manure may only be carried out in manure transport as described in BAM/Part 3/01-B The procedure for sampling in simulation of a manure transport as described in BAM/deel3/01-A, point 5 may be applied as an alternative;

For liquid streams with a dry matter content < 2 % (such as effluents, effluents, effluents from biological treatment, washing water, grease water), preservation applies when samples are taken:

- Total N: 100 ml container (P or G)
    - acidification to pH 1-2 with H_2SO_4[1] or with HCl[2], 1 month
- NH_4– N: 100 ml container (P or G)
    - Immediately after sampling, 1 day
    - Acidification to pH 1-2 with H_2SO_4, 1 month
- Total P: 100 ml container (P or G)
    - Acidification to pH 1-2 with HNO_3, 1 month

Note: Depending on the nature of the sample (e.g. samples with high organic carbon) acidification may result in a severe reaction. For safety reasons, it may then be decided, on the basis of expert judgement (visual assessment), not to carry out on-site preservation. In case of doubt, acidification is left to the laboratory.

[1] acidification to pH < 4 may be carried out using the oxidative digestion method cfr WAC/III/D/032.
[2] if Chemiluminiscence method cfr WAC/III/D/033 is used.

2 HYGIENE MEASURES

In the case of sampling, the sanitary rules in force at the farm must be complied with at the request of the client (e.g. boots by disinfecting bath, use of overalls on site, showering, etc.).

Both the protective clothing (overalls, footwear, etc.) and all sampling material must be clean when entering the farm in order to avoid cross-contamination from previously visited farms.

3 SAMPLING OF A VERTICAL STORAGE

The sampling of tanks and large liquid storage units of liquid manure or liquid treated manure shall be carried out by taking multiple samples at equal distance from each other and evenly distributed over the total liquid height.

For this purpose, the sampler must be capable of being shut down in the liquid. In the case of storage of (treated) liquid manure, the sampling device shall be of sufficient severity or capable of being pressed into the (treated) manure.

In practice, this limits the choice of sampling device to:
- a suitably heavy sampling bottle or bottle;
- a rod operated multisampler.

Working method:

a. Determine the fluid height: for this purpose, use a probe or sufficiently heavy probe. To avoid errors, the measurement should preferably be performed in duplicate. Always clean the probe/probe for the next measurement.
b. Samples shall be taken at 3 depths (upper, middle and lower): at 80 %, 50 % and 20 % altitude of the total liquid height.
c. Calculate the depths at which the grabs are to be taken relative to a fixed reference point (liquid surface or edge storage) and mark them on the cable or bar of the sampler. Take into account the depth at which the suction orifice will be located.
d. Always start sampling the top layer so that the underlying layers are disturbed as little as possible.
e. Where 3 fully filled samplers do not provide sufficient field steel to form the necessary laboratory samples, several handles shall be taken at each depth. The same number of steps shall be taken at each depth.
f. Allow the closed sampling device to sack to 80 % of the total fluid height ('top') and open the sampling device. When using a sampling bottle, wait long enough before collecting the sampling bottle so that it can be fully filled.
g. Collect the sampling device and check that it is fully filled. Transfer the contents of a fully filled sampler into a pure bucket. Note that the material on the outside of the bottle does not end up in the bucket. If the sampling device is not fully filled, remove the sample and repeat sampling at the same depth.
h. Repeat points (f) and (g) at the same depth if necessary (see point (e))

i. Repeat points (f) to (h) for the sampling levels at 50 % ('centre') and 20 % ('below') of the fluid height. Any deviation or limitation of this sampling strategy in a vertical sense (i.e. deviation at one or more of the sampling levels imposed) shall be documented and reported.
j. The grips of the upper and middle samples may be pooled in situ into a composite sample provided that the bottom and middle samples all contain the same volume of liquid and complete grips are pooled at all times.
k. Repeat this until sufficient field steel was collected to make the necessary lab samples.
l. Mix the field steel sufficiently and fill the necessary containers.

Sampling of the full fluid height by moving an open sample bottle through the fluid column (bags or pick-up) is not allowed.

4 SAMPLING AT A TAP

The sampling of liquid manure or liquid treated manure may be carried out at a tap which is mounted on the line through which the (treated) manure passes or which is mounted on an above-ground storage tank. This method may only be used where the lot cannot be sampled by any other means.

The following considerations shall be taken into account when starting sampling and selecting the sampling point:

a. Select the sampling point at a location where it is representative of the flow to be sampled.
b. Sampling should preferably take place from a sampling point with flowing (treated) manure. The flow and turbulences in the flow will ensure homogeneous distribution of particles and/or contaminants. In the case of stationary (treated) manure, this is not the case and heterogeneity will occur mainly in a vertical direction.
c. Preferably use existing sampling points to allow comparison of results, unless the existing sampling point is not compatible/suitable for the intended purpose of sampling and/or suitable for the parameter (s) to be analysed.
d. Avoid sampling points on horizontal pipes, or where there is a loss of liquids.
e. Avoid dirty, contaminated or corroded taps and cocks that are too close to the ground (contamination risk), or leaking connections above the sampling point/tap.
f. Avoid small taps that do not allow representative sampling of floating parts. The diameter of the pipe to the tap and of the tap is at least 3 times the size of the largest floating parts.

For sampling at a tap, single sampling shall be applied when the tap is mounted on:

- A piping circuit with flowing liquid (treated) manure
- A storage tank whose contents can be considered homogeneous or which can be homogenised by a blender or a rotary pump circuit. In the latter case, the mixing must be carried out for a sufficient period of time to permit the assumption that the contents are homogeneous.

If the contents of a storage tank cannot be regarded as homogeneous and cannot be homogenised, simple sampling at the tap is not representative of the

sample guarantee and sampling may only take place at the tap if the storage tank has no manholes that allow sampling. In this situation, immediate sampling no longer guarantees the representativeness of the sample in relation to the entire storage tank. The words ‘instantaneous sampling at tap – unrepresentative’ shall be added to the sampling form and report.

Working method:

a. Document and describe the product sampled, the sampling situation (line circuit, stationary, flowing, tank (storage) (wagon), blender/mixer, round pump circuit/circulation facility and the selected sampling point (address/local or location in production, flows, sketch, GPS coordinates,...) unequivocally on the sampling form.
b. Remove all devices and/or connectors (if any) that can be easily detached manually or by means of a tongue/key (only to be carried out in consultation with the person responsible for the production!).
c. Remove visibly sticky dirt.
d. Open the tap (preferably and if it is safe to do so at the same rate as the flowing (treated) manure in the pipe) and rinse for at least 1 minute (if the tap is installed directly on the pipe circuit with flowing (treated) manure), or at least 3 times the dead volume of the mouth pipe, up to a maximum of 50 litres. Collect this volume.
e. Adjust the tap at half flow rate (if possible) and collect at least 1/3 of the amount of (treated) manure needed to fill the different containers. Preferably use an engraved container (e.g. measuring cup) and transfer the (treated) manure into a pure bucket.
f. Repeat steps d. and e. twice with 5 minutes each time between sampling and the next wash of the dead volume. Collect the same amount of manure each time.
g. Mix the sample of the field sufficiently and fill the necessary containers.
h. If necessary, mount the removed pads and/or couplings.

5 SAMPLING OF LAGOONS

When liquid manure or liquid treated manure is stored in lagoons (here mainly effluents), the sample is taken from the perimeter of the lagoon.

The creator stick is usually the most suitable for this method. Alternatively, a sampling bottle or cage may be used, provided that local conditions allow sub-sampling at specific depths.

If the lagoon sediment is also to be sampled, a multisampler, piston boron, Beekersampler, peat boron, certain types of ground drilling, etc. may also be used.

<u>Sampling procedure for liquid fractions (e.g. effluents):</u>

a. Determine the depth of the batch. This is necessary to (i) determine the sample after-median values for the sub-samples and (ii) calculate the volume of the lot.
b. Compile a composite sample from partial samples taken at a minimum of 4 locations spaced along the perimeter perimeter. Avoid locations along the perimeter that could affect the steel (e.g. inflow of fresh material, inflow of water,...). Non-accessible parts

of the perimeter may affect the analytical result of the sampling and therefore you need to document it.

c. At each of the 4 locations, take a first sub-sample at a depth of 1-20 cm at a minimum of 30 metres from the side and a second sub-sample at half of the lot depth (at a minimum of 1 metres). Collect all subsamples in a collection container.
d. Divide the aggregate sample into the appropriate sub-containers under thorough mixing.

Method for sampling the sediment fraction

a. Assemble a composite steel from 4 sub-samples taken at a minimum of 4 locations spaced along the perimeter perimeter. Avoid locations along the perimeter that could affect the steel (e.g. inflow of fresh material, inflow of water,...). Non-accessible parts of the perimeter may influence the analytical result of the sampling and therefore you need to document it.
b. At each location, bring the multisampler or piston boron to the bottom of the basin. The piston keeps you more or less stationary at the top of the sediment fraction to be sampled, only the tube pushes you down. By pushing the tube down at the same time and raising the piston rope or rod, the tube is depressed to make it easier to take steel.
c. Remove the apparatus from the sediment layer and pour the stem into a pure collection container.
d. If the sediment thickness exceeds the length of the tube of the sampling device (typically 1 m, some devices have 2 m workable length), place the device in the same place in the manure layer so that the underlying layers are sampled in successive stitches of 1 m length each (e.g. 0-100, 100-200, etc.). You repeat this until you reach the soil in that place. Make sure that the sides of the pelvis are inclined. If possible, try to place the drilling parallel to the basin wall.
e. Combine the 4 sub-samples to form 1 field sample.

6 SAMPLING OF FERTILISERS

Liquid manure or liquid treated manure from manure bags may only be sampled at loading or unloading using a sampling device. For the sampling, reference is made to BAM/part 3/01-B
Two transports or at least two transports shall be sampled, distributed proportionally over the filling or emptying of the manure bag.

7 IDENTIFICATION OF SAMPLES

The tag (number, barcode, etc.) of the sample must be unambiguous so that no misunderstandings can subsequently arise as to the origin of the sample.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be indicated on the (digital) sampling form accompanying the sample:

a. client, address of the farm number of the holding;
b. client or third parties present at the sampling;
c. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number);
d. date and time of sampling;
e. own sample number or sample coding;
f. type of manure (e.g. sow slurry, pig meat slurry, calf slurry...) or type of treated manure (e.g. effluent of [type of digestate/type of manure], thin fraction after separation of [type of digestate/type of manure]...). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. If the sample is derived from treated manure, this must be stated unequivocally and explicitly;
g. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the sampled storage. Those coordinates shall be determined locally by a GPS device and stored electronically.
h. description of manure storage sampled (e.g. basin, silo...);
i. sampling description: technique/equipment used, sampling pattern,... sufficient to reproduce the sampling conditions
j. a photograph of the sampled storage
k. the estimated volume of manure in the manure storage; in the case of sampling in lagoons, map data may be used for the size of the lagoon.
l. volume of the field and laboratory sample and whether or not to carry out sample reduction in situ;
m. significant remarks and/or deviations that may affect the interpretation of the analytical result.

The laboratory's sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

8 MONSTERCONSERVATION

- The sample is stored chilled (5 ± 3) °C immediately after sampling. All transports must be refrigerated (with refrigeration box or refrigeration inside the wagon).
- Refrigeration must be traceable during storage.
- The sample must be prepared for analysis no later than the seventh day after sampling.

Liquid manure and liquid treated manure – Sample pre-treatment

CONTENT

1 Principle 3

2 Material 3

3 Reagents 4

4 Practice 4

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4.2 Samples with an estimated dry matter content of at least 15 % 4

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5.1 Samples with an estimated dry matter content of less than 15 % 4

5.2 Samples with an estimated dry matter content of at least 15 % 4

6 Quality check 5

7 Reference 5

The following method describes the procedure for homogenising samples of liquid manure prior to analysis. This is based on a laboratory sample with a volume of between 0.5 l and 1 l.

Note: The liquid samples with dry matter content < 2 % are homogenised by shaking or mixing. The volume of the laboratory sample shall be at least 100 ml per parameter.

Samples of liquid manure with a dry matter content between 15 and 30 % can be classified with both the liquid manure and the solid manure. The subdivision of the laboratory sample delivered into the relevant matrix type and the related sample pre-treatment can be carried out based on the estimated dry matter content combined with a visual assessment. However, the physical state based on the visual observation is determinative for carrying out sample pre-treatment.

Sample pre-treatment for liquid treated manure prior to analysis is described in the Compendium for Sampling and Analysis in implementation of the Materials Decree and the Soil Decree, and more specifically in CMA/5/B.1 Sample pre-treatment *of Fertiliser Soil Improvement Product* (https://emis.vito.be/nl/referentielabo-ovam).

For both liquid manure and liquid treated manure, the samples shall be:

a. be kept cool at a temperature of (5 ± 3) °C at all times to avoid conversions;
b. processed for analysis no later than the seventh day after sampling.

1 PRINCIPLE

In this procedure, after any addition of water, the sample is homogenised by a rapidly rotating knife of a construction such that optimal mixing is obtained. Homogenisation is carried out using a robust bar mixer with a variable rotational speed and a closed shaft (stator) in which the rotor moves. The dilution factor is determined.

Representative sub-samples shall be provided for the determination of:

a. dry matter at 105 °C, total nitrogen, ammoniacal nitrogen: fresh sample;
b. total phosphorus: fresh sample or sample dried at 105 °C.

Note: The test sample for dry matter determination may continue to be used for the determination of total phosphorus on a dried sample.

2 MATERIAL

The usual laboratory glassware and also:

a. bar mixer with a variable rotation speed of at least 10.000 revolutions per minute (e.g. ultra turrax);
b. lockable plastic bottle;
c. balance, capable of weighing to an accuracy of at least 0.1 g.

3 REAGENTS

Use only reagents of analytical grade.

4 PRACTICE

Take a sample of at least 500 ml. Remove non-manure objects.

4.1 SAMPLES WITH AN ESTIMATED DRY MATTER CONTENT OF LESS THAN 15 %

Place the monmortalities under the rod mixer, with the rotor blade located approximately 3 cm from the bottom of the monmortalities. Homogenise the sample at as high a rotational speed as possible, avoiding excessive foaming, taking into account the type of manure. To optimise homogenisation, the rod mixer can be moved vertically back and forth during homogenisation. Subsamples shall be taken immediately after homogenisation. After homogenisation, the oral bottle is closed.

4.2 SAMPLES WITH AN ESTIMATED DRY MATTER CONTENT OF AT LEAST 15 %

Weigh a plastic bottle to the nearest 0.1 g (mass m_0).

Quantitatively transfer the sample into the bottle with a quantity of water weighed to 0.1 g (m_1). Weigh the plastic bottle with the sample and the added water (m_2). Proceed as described above.

5 CALCULATION OF THE DILUTION FACTOR

For further determinations carried out on that sample, the dilution factor shall be included in the final calculations.

5.1 SAMPLES WITH AN ESTIMATED DRY MATTER CONTENT OF LESS THAN 15 %

Is the dilution factor F = 1.

5.2 SAMPLES WITH AN ESTIMATED DRY MATTER CONTENT OF AT LEAST 15 %

Calculate the dilution factor (F) from the equation

$$F = \frac{m_2 - m_0}{m_2 - m_1 - m_0}$$

with:
F: dilution factor;
m_0: mass of the empty plastic bottle in grams;
m_1: mass of water added, in grams;
m_2: mass of the plastic bottle with sample and water, in g.

Round the result to 3 decimal places.

6 QUALITY CHECK

As a quality control, at least 1 sample per day shall be analysed in duplicate for at least one relevant parameter. For this purpose, 2 sub-samples are taken after sample pre-treatment and go through the entire analytical route.

7 REFERENCE

NEN 7430: 1998 Animal manure and manure products – Sample pre-treatment by homogenisation – Fertiliser

Liquid manure and liquid treated manure – dry matter content

CONTENT

1 **Principle** 3

2 **Equipment and materials** 3

3 **Practice** 3

4 **Remarks** 3

5 **Calculations** 4

6 **Reference** 4

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 3/02.

The dry matter content (in relation to fresh material) must be determined to allow conversion to fresh material in the determination of total phosphorus.

The method consists of drying a predetermined quantity of homogenised sample at a temperature of 105 °C ± 5 °C for a specified time.

2 EQUIPMENT AND MATERIALS

a. drying crucibles or other suitable containers
b. drying oven, mechanically ventilated, set at a temperature of 105 °C ± 5 °C
c. desiccator
d. balance accurate to 0,001 g
e. 50 ml pipette with wide orifice or a 50 ml graduated pipette (the correct volume is not important)

3 PRACTICE

A test portion should be taken immediately after sample preparation so that a homogeneous sample can be taken.

Crosses are prepared by drying at 105 °C ± 5 °C and then cooled in a desiccator. The empty crucible is weighed (m_0).

50 ml of homogenised sample is placed in a weighed crucible by means of a measuring plate or a pipette with a wide orifice. Re-weigh (m_1).

Place Crosses in the pre-heated oven. Dry for 24 hours at 105 °C ± 5 °C. Remove Crosses from the oven and leave to cool in a desiccator to ambient temperature. Re-weigh (m_2).

4 REMARKS

a. Weighings shall be made to the nearest 1 mg.
b. The total P content can be performed on a test portion of the dried sample.

5 CALCULATIONS

$$DS\ F^{\,m_2\ \ m_{0\ 100\ m\ 0}}\ m_1\ \ m_0$$

with:
DS: dry matter content in kg/1000 kg
VM; F: dilution factor;
m_0: mass of empty crucible in grams;
m_1: mass of crucible + fresh sample in
g; m_2: mass of crucible + dry sample in
g.

Round the result to 2 decimal places for values ≤ 1, 1 decimal place for values > 1 and to one integer for values > 100.

6 REFERENCE

NEN 7432: 1998 Animal manure and manure products – Determination of dry matter and organic matter content – Gravimetric method

Liquid manure and liquid treated manure – Total phosphorus

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1 **Principle** 3

2 **Equipment and materials** 3

3 **Reagents** 3

4 **Unsealing methods** 3

4.1 Digestion with rendering and HNO_3 destruction 3

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5 **Analytical determination of phosphorus in the solubilising solution** 4

6 **Calculations** 5

6.1 Method of calculation for dried samples 5

6.2 Method of calculation for fresh samples 5

7 **References** 5

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 3/02.

For the determination of total P in liquid manure and liquid treated manure, the following methods of rendering and analysis may be applied:

- **a.** the dried sample is ashed at 550 °C, then the ash is dissolved in HNO_3. The determination of phosphorus in the solution is carried out spectrophotometrically or with ICP-AES;
- **b.** the fresh or dried sample is solubilised with an aqua regia (HNO_3: HCl) acid destruction. The determination of phosphorus in the solution is performed with ICP-AES.

For the determination of total P in liquid manure, the fresh sample may be solubilised with sulphuric acid, hydrogen peroxide and copper sulphate according to NEN 7433. The determination of phosphorus in the solution is carried out spectrophotometrically or with ICP-AES. On the same solubilising solution, it is possible to determine total N (= Kjeldahl-N).

2 EQUIPMENT AND MATERIALS

- **a.** crucibles for ashing
- **b.** oven set at 550 °C ± 25 °C
- **c.** desiccator
- **d.** heating plate
- **e.** ash-free filter paper
- **f.** acid-resistant rendering block, programmable to at least 105 °C
- **g.** disposable rendering tubes, 50 ml, acid-resistant
- **h.** compact condenser

3 REAGENTS

- **a.** HNO_3, 14 mol/l
- **b.** HNO_3, 1 mol/l
- **c.** HCl, 12 mol/l

4 ONTSLUTION SMETHODES

4.1 O NOTIFICATION WITH INCINERATION AND HNO_3 DESTRUCTION

Weigh to the nearest 1 mg, 2,5 to 1 g of dry sample (m).

Ash that sample at 550 °C for 4 hours. The axle shall be grey white. If the ash is not white, add a few drops of 14M HNO 3 and ash again for 1 hours.

Quantitatively transfer the ash into a 100 ml beaker containing 20 ml of 1M HNO_3. Leave to distil for one hour on a heater or in a hot water bath.

Filter and collect filtrate in a 100 ml volumetric flask and rinse well with 1M HNO_3. Make up to 100 ml with 1M HNO_3.

4.2 O NOTICE WITH HNO_3/HCL (AQUA REGIA)

The rendering may also be carried out in a heatable rendering block with rendering tubes fitted with a compact condenser.

Note: Alternatively, a watch glass or a shut-off cap (tightening and half turn back) may be used for the condenser.

Weigh to the nearest 15 mg (m) approximately 1 g of freshly homogenised or 1 g of dried sample ground in the mortar in a rendering tube. Gradually add 4 ml of 14M HNO_3 and 12 ml of 12M HCl.

Place the condenser on the rendering tubes. Leave rendering tubes at room temperature to allow a slow reaction of the organic matter. Carry out the rendering programme with incremental warming, for example:

a. warm up in 20 minutes to 45 °C, 5 minutes at 45 °C;
b. warm up in 10 minutes to 65 °C, 10 minutes at 65 °C;
c. warm up to 105 °C, 120 °C for 105 minutes.

Filter the sample after destruction. Make up to 50 ml with ultra pure water.

5 ANALYTICAL DETERMINATION OF PHOSPHORUS IN THE ONLY OPINION

The analytical determination of phosphorus in the solubilising solution may be carried out according to:

a. NBN EN ISO 11885: 2009 Water quality – Determination of selected elements by Inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885: 2007).

The analytical determination of phosphorus in the solubilising solution can be performed spectrophotometrically according to:

a. NBN AND ISO 6878: 2004 water quality – Determination of phosphorus – Ammonium molybdate spectrometric method;
b. NBN AND ISO 15681-1: 2005 water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 1: Method by flow injection analysis (FIA) (ISO 15681-1: (2003);
c. NBN AND ISO 15681-2: 2005 water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2: (2003);
d. ISO 15923-1: 2013 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection.

Note: For the spectrophotometric methods, the phosphorus content is determined in a five-fold dilution of the solubilising solution.

6 CALCULATIONS

6.1 METHOD OF CALCULATION FOR DRIED SAMPLES

The measured phosphorus concentration shall be converted to a concentration C_P (kg P_2O_5/1000 kg) in fresh material according to the following formula:

$$C_P = \frac{C_1}{M} \times F \times V \times DS \times 2.29 \times \frac{1}{1000}$$

with:
C_P: phosphorus concentration in the original sample in kg P_2O_5/1000 kg VM;
C_1: measured phosphorus concentration in mg P/l;
f: dilution factors, if any;
V: volume of solubilising solution in litres;
DS: dry matter content in kg/1000 kg VM as determined in BAM/deel3/03; m: mass of dry sample taken under processing in grams.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

6.2 METHOD OF CALCULATION FOR FRESH SAMPLES

The measured phosphorus concentration shall be converted to a concentration C_P (kg P_2O_5/1000 kg) in fresh material according to the following formula:

$$C_P = \frac{C_1}{M} \times F \times V \times 2.29$$

with:
C_P: phosphorus concentration in the original sample in kg P_2O_5/1000 kg VM;
C_1: measured concentration in mg P/l;
f: dilution factors, if any;
V: volume of solubilising solution in litres;
m: mass of fresh sample taken under processing in g.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

7 REFERENCES

a. NEN 7433: 1998 Animal manure and manure products – Sample pre-treatment for the determination of nitrogen, phosphorus and potassium – Dissolution with sulphuric acid, hydrogen peroxide and copper sulphate
b. NEN 7435: 1998 2nd draft Animal manure and manure products – Determination of phosphorus content in destruates
c. NBN EN 13650: 2001 Soil improvers and growing media – Extraction of aqua regia soluble

elements

d. NBN EN ISO 6878: 2004 Water quality – Determination of phosphorus – Ammonium molybdate spectrometric method

e. NBN EN ISO 15681-1: 2005 Water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 1: Method by flow injection analysis (FIA) (ISO 15681-1: 2003)
f. NBN EN ISO 15681-2: 2005 Water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2: 2003)
g. NBN EN ISO 11885: 2009 Water quality – Determination of selected elements by Inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885: 2007)
h. ISO 15923-1: 2013 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection
i. C. Vanhoof, A. Cluyts, K. Duyssens, E. Poelmans, Wendy Wouters and K. Tirez, *Sustainability of N parameters and destruction of P in manure samples*, VITO report 2011/MANT/070, https://reflabos.vito.be/onderzoeksrapporten/rapport_mest_N_en_P_2011.pdf
j. C. Vanhoof and K. Tirez, *Evaluation of methods of analysis for the determination of inorganic parameters in digestates*, VITO report 2012/MANT/R/005, https://reflabos.vito.be/onderzoeksrapporten/Rapport_2011_digestaten_finaal.pdf
k. C. Vanhoof and K. Tirez, *Harmonisation of inorganic analytical methods for the analysis of compost*, *manure and various digestate* streams, VITO report 2012/MANT/R/086, https://reflabos.vito.be/onderzoeksrapporten/2012_Bodemverbeterende_middelen_finaal.pdf

Liquid manure and liquid treated manure – Ammonium nitrogen

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3.2.4 Ammoniacal nitrogen with a discrete analytical system (spectrophotometric detection) 6

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4 References 7

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 3/02.

The determination of the ammonium content of liquid manure or liquid treated manure may be carried out directly on the homogenised sample using the titrimetric method after steam distillation according to:

a. ISO 5664: 1984 water quality – Determination of ammonium – Distillation and titration method.

If the following methods are used, leaching of the sample is required:

a. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method;
b. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection;
c. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li^+, Na^+, NH_4^+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998);
d. NOT AN 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).

Note: for heavy load matrices, due consideration shall be given to the analysis to measure interference free.

2 DETERMINATION OF AMMONIUM AFTER STEAM DISTILLATION

2.1 PRINCIPLE

Ammonium in a solution containing alkali-labile nitrogen components is exempted by the addition of MgO. The resulting ammonia is released by steam distillation and collected in excess acid. The amount of ammonium is determined by return titration.

Sodium hydroxide is not used during distillation and the duration of distillation is kept as short as possible to avoid the determination of alkali-labile organic nitrogen compounds.

2.2 PROCEDURE

The procedure described in ISO 5664: 1984 shall apply with the following additions:

a. § 2.3 sensitivity: not applicable;
b. Paragraph 4 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
c. Paragraph 6 Sampling: for the preservation and handling of the samples reference is made to BAM/part 3/02;
d. § 7.1 selection of test portion volume: other volumes may be used if they are appropriate for this application;
e. Paragraph 7.2.3: other endpoint detections are also possible.

2.3 CALCULATIONS

The pre-treatment of samples shall be taken into account.

The result is expressed as nitrogen concentration C_N (kg N/1000 kg) in fresh material using the following formula:

$$C_N = M_N \times \frac{(R_1 - V_0) \times C_{HCL}}{M} \times F$$

with:
C_N: concentration of ammonium in the original sample in kg N/1000 kg VM; M_N:
is the molar mass of nitrogen (14.007 g/mol);
V_1: volume at titration of the sample in ml; V_0: volume at titration of the blank in ml;
m: mass, in grams, of the sample taken under processing; C_{HCl}:
concentration of hydrochloric acid in mol/l,
F: dilution factor.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

3 DETERMINATION OF AMMONIUM AFTER LEACHING

The determination of ammonium may be carried out in leaching using one of the following methods:

a. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method;
b. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection;
c. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li^+, Na^+, NH_4^+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998);
d. 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).

3.1 EXTRACTION PROCEDURE

Weigh to the nearest 5 mg a certain quantity of freshly homogenised sample (± 1 g). A representative sub-sample can be taken by using a pipette with a wide outlet or a graduated disc.

This sub-sample is diluted with water at a ratio of 1/100 (m/v) in a graduated flask. Shake well. The volume of the volumetric flask is V_{ext}.

Centrifuge or filter the solution. Rinse the filter with sample solution and discard the first part of the filtrate. The rest of the filtrate is collected in a dry container.

Carry out further analysis immediately after filtration.

Note: The water used for the dilution of the manure can be slightly acidified with HCl to avoid ammonia volatilisation. This should only be done if it does not affect the method of determination.

3.2 Measurement of ammoniacal nitrogen in leaching

3.2.1 Ammoniacal nitrogen by manual spectrophotometric method

The procedure described in ISO 7150-1: 1984 shall apply with the following additions:

a. § 1.5 sensitivity: the minimum absorption shall be checked, but the concentration used and the procedure used may differ from the described ISO procedure;
b. Paragraph 4 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
c. Paragraph 6 Sampling: for the preservation and handling of the samples reference is made to BAM/part 3/02;
d. Paragraph 7.3: other relevant concentration levels may be used if they are appropriate for this application. The same procedure shall be applied for samples and standards;
e. § 7.5 calibration: the methodology may deviate from the described procedure if the calibration line is established with at least 5 calibration solutions and complies with this application.

3.2.2 Ammoniacal nitrogen with continuous flow analysis (CFA) using spectrophotometric detection

The procedure described in NBN EN ISO 11732: 2005 shall apply with the following additions:

a. § 3 determination of ammoniacal nitrogen with flow injection analysis (FIA) and spectrophotometric determination: not applicable;
b. Paragraph 4.3 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
c. Paragraph 4.4.3 Sampling: for the preservation and handling of the samples reference is made to BAM/part 3/02;
d. Paragraph 4.5.2 Control performance tool: the minimum absorption shall be checked, but the concentration used and the procedure used may differ from the described ISO procedure;
e. § 4.5.3 blank control reagents: blank control of reagents is optional.

3.2.3 Ammoniacal nitrogen with ion chromatography

The procedure described in NBN EN ISO 14911: 1999 shall apply with the following additions:

a. Paragraph 6 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
b. § 8 quality requirements for separation column: other concentrations may be used to evaluate separation conditions;
c. Paragraph 9 Sampling: for the preservation and handling of the samples reference is made to BAM/part 3/02.

3.2.4 AMMONIACAL NITROGEN WITH A DISCRETE ANALYTICAL SYSTEM (SPECTROPHOTOMETRIC DETECTION)

The procedure described in NBN EN ISO 15923-1: 2024 shall apply with the following additions:

a. § 5 other reagents and concentrations may be used if they are appropriate for this use;
b. § 7 for the preservation and handling of the samples reference is made to BAM/part 3/02;
c. Annex B to H: deviations from the implementation of the described methods 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;
d. § 8.1 and § 8.2: Additional quality control for the determination of parameters ammonium, nitrate and nitrite in leaching. The analysis of these samples must include at least 1 of the following quality checks:
    1) analysis of the sample by at least 1 degrees, with a bias of not more than 10 % relative to the theoretical value;
    2) at least 2 measurements of the same sample with a dilution factor differing by at least a factor of 2, resulting in 2 measurement results within the measuring range differing by not more than 10 %.

Note: False negative results may occur in the determination of ammonium at high concentrations. The quality checks referred to above are intended to remedy this situation.

Note: Ammonium and nitrate can be determined in leaching of liquid manure samples and samples that can be used in or as fertilisers or soil improvers. Those samples should normally be strongly diluted to eliminate matrix interference.

3.3 CALCULATIONS

Determine the concentration of ammonium in leaching, taking into account any dilutions.

The result is expressed as nitrogen concentration C_N (kg N/1000 kg) in fresh material using the following formula:

$$C_N = \frac{C_l \cdot V_{ext}}{m} \cdot F$$

with:

C_N: concentration of ammonium in the original sample in kg N/1000 kg VM; C_1:
concentration of ammonium in the extract in mg N/l;
m: mass of sample extracted in grams; V_{ext}: total
volume of extract in l;
F: dilution factor.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

3.4 REPORTING LIMIT

The reporting limit is ≤ 0.2 kg N/1000 kg VM.

4 REFERENCES

a. ISO 5664: 1984 water quality – Determination of ammonium – Distillation and titration method
b. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method
c. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection
d. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li+, Na+, NH_4^+, K+, Mn 2 +, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998)
e. 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)
f. NEN 6604: 2007 Water – Determination of ammonium, nitrate, nitrite, chloride, orthophosphate, sulphate and silicate content using a discrete analytical system and spectrophotometric detection
g. C. Vanhoof, A. Cluyts, E. Poelmans, W. Wouters and K. Tirez, *Evaluation of discrete analyser for the determination of nitrate and ammonium in soil and manure*, VITO report 2012/MANT/R/04, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/2011_r apport_discrete_analyser_VLM.pdf

Liquid manure and liquid treated manure – Total nitrogen

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3 Reagents 4

4 Practice 4

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4.2 Destruction 4

4.3 Determination 5

5 Remarks 5

6 Calculations 5

7 Reporting limit 6

8 References 6

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 3/02.

The determination of the total N content in liquid manure and liquid treated manure shall be carried out according to the following methods:

a. NBN AND 13654-2: 2001 soil improvers and growing media – Determination of nitrogen – Part 2: Dumas method;
b. NBN EN 16168: 2012 Sludge, treated biowaste and soil – Determination of total nitrogen using dry combustion method;
c. NBN EN 13654-1: 2001 Soil improvers and growing media – Determination of nitrogen – Part 1: Modified Kjeldahl method;
d. sum of Kjeldahl-N, nitrate and nitrite nitrogen.

The Kjeldahl-N method is described in:

a. NBN EN 16169: 2012 Sludge, treated biowaste and soil – Determination of Kjeldahl nitrogen;
b. NEN 7437: 1998 Animal manure and manure products – Determination of total nitrogen content.

Alternative to Kjeldahl-N method:

a. NEN 7433 Animal manure and manure products – Sample pre-treatment for the determination of nitrogen, phosphorus and potassium – Dissolution with sulphuric acid, hydrogen peroxide and copper sulphate.

It is assumed that liquid manure does not contain nitrate or nitrite. The same applies to the thin fraction obtained after separation of liquid manure. The determination of total nitrogen in liquid manure or the liquid fraction obtained after separation of liquid manure is therefore limited to Kjeldahl nitrogen. The determination of Kjeldahl nitrogen includes a rendering with H_2SO_4 and a catalyst mixture which converts organic nitrogen compounds into ammonium. After destruction, ammonia is exempted by the addition of sodium hydroxide and distilled in a suitable absorbent. Ammonium is then determined in the distillate by titration or spectrophotometric determination.

Alternatively, for Kjeldahl nitrogen determination, the sample may be solubilised with sulphuric acid, hydrogen peroxide and copper sulphate according to NEN 7433, followed by titrimetric or spectrophotometric determination of ammonium content. On the same solubilising solution, it is possible to determine total P spectrophotometrically or with ICP-AES.

If the analysis is carried out on liquid treated manure (e.g. effluent from biology, effluents...), except for the thin fraction obtained after separation of liquid manure, it should not be assumed that those products do not contain nitrate or nitrite. In that case, the Compendium for sampling and analysis pursuant to the Materials Decree and the Soil Decree, and more specifically CMA/2/IV/4 Total Nitrogen (Matrix Fertiliser – Soil Improvement Device) (https://emis.vito.be/nl/referentielabo-ovam), must be followed for the determination of total nitrogen.

This BAM procedure describes the determination of Kjeldahl-N.

2 EQUIPMENT AND MATERIALS

a. rendering tubes, 250 ml
b. rendering block, for a temperature of 370-380 °C
c. distillation apparatus suitable for connecting 250 ml rendering tubes
d. cooking stones

3 REAGENTS

a. H_2SO_4, 18M;
b. hydrochloric acid, 0.2 mol/l: dilute 16 to 17 ml of concentrated hydrochloric acid to 1 litre. This solution should be adopted;
c. methyl red solution, 2 mmol/l: dissolve 0.5 g methyl red in 1 l ethanol solution; 60 % (v/v) ethanol in water;
d. methylene blue solution, 4 mmol/l: dissolve 1.5 g methylene blue in 800 ml water, make up to 1 l and mix.
e. boric acid indicator solution, 0.3 mol/l: dissolve 20 g boric acid in warm water. Cool and add 10 ml methyl red solution and 2 ml methylene blue solution. Adjust the pH to 4.6 (tipping point of methyl red). Ling to 1 l and mix;
f. sodium hydroxide solution, 9 mol/l: dissolve 360 g sodium hydroxide in water and dilute to 800 ml with water. Cooling, stirring to 1 l and mixing;
g. catalytic converter: 100 g potassium sulphate (K_2SO_4) and 10 g copper sulphate pentahydrate ($CuSO_4.5H_2O$): times and mixes (NEN 7437);
h. anti-foaming agent.

4 PRACTICE

4.1 SAMPLING

Weigh a certain quantity of freshly homogenised sample to the nearest 1 mg (mass m) in a rendering tube. After homogenisation, liquid manure is sampled using a pipette or a graduated disc. The quantity to be processed contains no more than 1 g of dry matter and an estimated nitrogen content of not less than 2 mg and not more than 100 mg.

4.2 DESTRUCTION

Add 20 ml H_2SO_4 and mix. Add 5 g catalyst mixture. Add anti-foaming agent.

Heat slowly until the liquid boils gently. Pay attention to excessive foaming. In the case of liquid manure, water is first evaporated. The temperature then rises further. Heat in such a way that the sulphuric acid condenses about half way through the rendering tube. Boil for 15 minutes after the liquid becomes clear.

The optimum destruction temperature is between 370 °C and 380 °C. The destruction is incomplete at a lower temperature and loses at higher steps.

After rendering, allow the liquid to cool and dilute with 50 ml of water.

4.3 DETERMINATION

Place 50 ml of boric acid indicator solution in a flask. Place the flask under the condenser so that the outlet is below the liquid level.

Add 50 ml of sodium hydroxide solution to the destruate in the rendering tube and immediately connect the tube to the distillation apparatus. Distil at a rate of 10 ml/minute until all ammonia is distilled.

Titrate the contents of the flask with standard hydrochloric acid until the colour changes from green to purple violet. Record the volume used (R_1).

Perform the whole procedure for a blank. Note the volume for that blank destruate (R_0).

Alternatively, the determination of ammonium in the distillate may be carried out by one of the following methods, provided that a suitable absorbent is used:

a. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method;
b. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection;
c. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li^+, Na^+, NH_4^+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998);
d. 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).

For the practical implementation of these provisions, reference is made to BAM/deel3/05, *3.2 Measurement of ammoniacal nitrogen in leaching*.

5 REMARKS

There are several variants of the Kjeldahl-N method. They are useful as long as they do not fundamentally alter the given procedure.

Other catalysts may also be used. Typically, these catalysts are commercially available as tablets.

6 CALCULATIONS

The result is expressed as nitrogen concentration C_N (kg N/1000 kg) in fresh material using the following formula:

$$C_N \quad M_N \quad (V_1 \quad V_0)\, C_{HCl} \quad F \quad m$$

with:
C_N: concentration of nitrogen in the original sample in kg N/1000 kg VM; M_N:
molar mass of nitrogen (14.007 g/mol);
V_1: amount of hydrochloric acid used at titration of the sample in
ml; V_0: amount of hydrochloric acid used when titrating the blank in
ml; m: weight of the test portion in g.
C_{HCl}: concentration of hydrochloric acid in
mol/l, F: dilution factor.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

7 REPORTING LIMIT

The reporting limit is ≤ 0.12 kg N/1000 kg VM.

8 REFERENCES

a. NEN 7437: 1998 Animal manure and manure products – Determination of total nitrogen content
b. NBN EN 13654-1: 2001 Soil improvers and growing media – Determination of nitrogen – Part 1: Modified Kjeldahl method
c. NBN EN 16169: 2012 Sludge, treated biowaste and soil – Determination of Kjeldahl nitrogen
d. NBN EN 13342: 2000 Characterisation of sludges – Determination of Kjeldahl nitrogen
e. ISO 11261: 1995 Soil quality – Determination of total nitrogen – Modified Kjeldahl method
f. NBN EN 25663: 1994 Water Quality – Determination of Kjeldahl nitrogen – Method after mineralisation with selenium (ISO 5663: 1984)
g. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method
h. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection
i. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li+, Na+, NH_4^+, K+, Mn 2 +, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998)
j. 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 (ISO15923-1: 2013)

Liquid manure and liquid treated manure — analysis of samples with a dry matter content < 2 %

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2 Sample preparation 3

3 Methods of analysis 3

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3.2 Determination of total N 3

3.3 Determination of NH_4– N 4

3.4 Determination of total P 4

4 Reporting limits and units 4

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1 PRINCIPLE

This method is applicable for the determination of dry residue, total N, NH_4– N and total P in flows with a dry matter content of less than 2 %.
Relevant matrices are effluents, effluent, biopurification effluent, washing water and grease water.

2 SAMPLE PREPARATION

The liquid samples are homogenised by shaking or mixing.

3 METHODS OF ANALYSIS

3.1 DETERMINATION OF THE DRY RESIDUE

The determination of the dry residue shall be carried out in accordance with WAC/III/A/001.

3.2 DETERMINATION OF TOTAL N

For the determination of total N, the following methods apply:

- NBN EN ISO 11905-1: 1998 Water quality – Determination of nitrogen – Part 1: Method using oxidative digestion with peroxodisulfate (ISO 11905-1: 1997) (WAC/III/D/032) [1]
- ISO 29441: 2010 Water quality – Determination of total nitrogen after UV digestion – Method using flow analysis (CFA and FIA) and spectrometric detectio n[1]
- NBN EN 12260: 2003 Water quality – Determination of nitrogen – Determination of bound nitrogen (TN_b), following Oxidation to nitrogen dioxide (WAC/III/D/033) 1
- Sum of Kjeldahl-N and nitrite and nitrate

If the methodology 'sum of Kjeldahl-N and nitrite and nitrate' is applied, the analysis report must: the following shall be clearly indicated:

(1) which parameter was calculated (e.g. Total N),
(2) the analytical results of the parameters used for the calculation (e.g. Kjeldahl-N, TON),

When calculating sum or difference, the following guidelines shall be followed:

a. If 1 of the parameters < reporting limit, the lower bound approach shall be applied (i.e. measurement value = 0). (for example: Kj-N = 5 mg N/l, TON < 0.1 mg N/l, result: total N = 5.0 mg N/l)
b. If both parameters < reporting limit, the highest reporting limit shall always be used (example 1: Total N < 1 mg N/l, TON < 0.1 mg N/l, result: Kj-N < 1 mg N/l; example 2: Kj-N < 1 mg N/l, TON < 0.1 mg N/l, result: Total N < 1 mg N/l)

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[1] high concentrations of organic substances may result in an underestimation of the nitrogen concentration due to insufficient oxidation capacity.

3.3 B ELECTION OF NH_4– N

For the determination of NH_4– N, the following methods apply:

- ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method (WAC/III/E/020)
- NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732: 2005) (WAC/III/E/021)
- ISO 5664: 1984 water quality – Determination of ammonium – Distillation and titration method (WAC/III/E/022)
- NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li^+, Na^+, NH_4^+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998) (WAC/III/E/023)
- 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) (WAC/III/C/002)

3.4 DETERMINATION OF TOTAL P

For the determination of total P, the following methods shall apply:

- Destruction shall be carried out in accordance with:
  - WAC/III/B/001 Cleaning for the determination of selected elements in water — nitric acid digestion or
  - WAC/III/B/002 Contamination for the determination of selected elements in water – aqua regia solubilisation

  Note: The digestion procedure is based on a test portion of 25.0 ml ± 0.1 ml. Any test portion greater than 5 ml is permitted, provided that the volume of acids used is adjusted proportionally.

- The analytical measurement shall be performed with ICP-AES according to WAC/III/B/010 *Determination of the selected elements with inductively coupled plasma – atomic emission spectrometry*

4 REPORTING LIMITS AND UNITS

For the calculation of the content expressed in fresh material (VM), a bulk density of 1 kg fresh material/l may be used.

The dry rest parameter is expressed in kg/1000 kg VM.
The reporting limit for the parameter total N is ≤ 2 mg/l or 0,002 kg N/1000 kg VM.
The reporting limit for the parameter NH_4– N is ≤ 0.25 mg/l or 0.00025 kg N/1000 kg VM.
The reporting limit for the parameter total P is ≤ 0.3 mg P/l or 0.0007 kg P_2O_5/1000 kg VM

5 REFERENCES

- Water Sampling, Measurement and Analysis Compendium (WAC), Water Sampling, Measurement and Analysis Compendium (WAC) | EMIS (vito.be)

Liquid livestock manure – Reporting

1 GENERAL

The reporting shall be carried out in accordance with BAM/part 8/20. The sampling report drawn up on the basis of the field records (sampling form) shall be added to the analysis report or incorporated into the analysis report.

Without prejudice to the provisions of BAM/part 8/20, the analytical report shall include:

a. laboratory letterhead paper with at least name, address, telephone, e-mail;
b. unique report number;
c. unique sample number and, if applicable, sample number assigned by the manure bank via SMIL [1;]
d. date of sampling;
e. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number) If the sample was not taken by a sampler attached to the laboratory, this should be explicitly mentioned in the analytical report;
f. client present at sampling (Y/N);
g. type of manure (e.g. sow slurry, pig meat slurry, calf slurry...) or type of treated manure (e.g. effluent of [type of digestate/type of manure], thin fraction after separation of [type of digestate/type of manure]...). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. The same description should be used as the one used on the MAD if applicable. If the sample is derived from treated manure, this must be stated unequivocally and explicitly;
h. description of the sampling location (e.g. manure cellar, loading of the transport, manure silo...);
i. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the sampled house or storage;
j. date on which the sample was received by the laboratory;
k. the date on which the sample was taken for analysis;
l. date on which the report was sent;
m. name and signature of the person in charge of the laboratory (possibly digitally);
n. name and address of the person to whom the report is delivered.

2 PARAMETER AND UNITS

Dry matter	kg/1000 kg VM
Ammonium	kg N/1000 kg VM
Total nitrogen	kg N/1000 kg VM
Total phosphorus	kg P_2O_5/1000 kg VM

[1] sampling Noding Internet Loket (https://www.vlm.be/nl/doelgroepen/laboratoria-en-staalnemers/SMIL)

Reported values shall be rounded to 2 decimal places for values ≤ 1, 1 decimal place for values > 1 and to one integer for values > 100.

3 NAME OF WEBSITE NOTIFICATION (SMIL)

Data on sampling, analysis results and GPS data catalogues are reported to the Flemish Land Agency via the SMIL application in accordance with the provisions in BAM/part 8/03.

Solid livestock manure – Scope

The methods relate to the sampling and analysis of solid livestock manure as provided for in the Decree of 22 December 2006 concerning the protection of waters against pollution caused by nitrates from agricultural sources (hereinafter referred to as the Fertiliser Decree) and its implementing decrees:

'Livestock manure' means both livestock excrements (whether or not mixed with litter) and all intermediate or final products resulting from a physical, chemical or microbiological (production) process in which livestock excrements (whether or not mixed with litter) are involved, regardless of their proportion.

The raw, untreated excrements of livestock (whether or not mixed with litter) are hereinafter referred to as 'manure'. All final and intermediate products resulting from a physical, chemical or microbiological (production) process in which manure was a raw material are hereinafter referred to as 'treated manure'.

"Solid" livestock manure means:

1. solid manure with a dry matter content exceeding 30 %;
2. solid treated manure with a dry matter content of more than 15 %.

For the sampling of solid manure, the methods of analysis described in BAM apply. For the sampling of solid treated manure, the methods described in CMA [1] apply, taking into account BAM/part 4/01-B.

For the sample preparation of solid manure, the methods described in BAM apply. Samples with a dry matter content between 15 and 30 % can be classified with both liquid and solid manure. The subdivision of the laboratory sample delivered into the relevant matrix type and the related sample pre-treatment can be carried out based on the estimated dry matter content combined with a visual assessment. However, the physical state based on the visual observation is determinative for carrying out sample pre-treatment.

For the sample preparation of solid treated manure, the methods described in CMA apply, taking into account BAM/part 4/02.

For the analysis of solid manure, the methods described in BAM apply. For the analysis of solid treated manure, both the BAM and CMA methods may be used. If the CMA methods of analysis are applied, the results shall be converted to the units as prescribed in the corresponding BAM methods.

The following table gives an overview of the methods to be applied depending on the matrix.

Parameter	Solid manure methods	Methods for solid treated manure
Sampling	BAM/part 4/01-A	CMA/1/A.15 EXPERIENCE, CMA/1/A.17, CMA/1/A.18 and

[1] compendium for sampling and analysis in implementation of the Materials Decree and the Soil Decree (https://emis.vito.be/nl/referentielabo-ovam)

Parameter	Solid manure methods	Methods for solid treated manure
Sample preparation	BAM/part 4/02	CMA/5/B.1 and BAM/part 4/02
Dry matter content	BAM/part 4/03	BAM/part 4/03 (or CMA/2/IV/1)
Total phosphorus	BAM/part 4/04	BAM/part 4/04 (or CMA/2/IV/19)
Ammoniacal nitrogen	BAM/part 4/05	BAM/part 4/05 (or CMA/2/IV/6 § 5.7 + CMA/2/IV/7)
Total nitrogen	BAM/part 4/06	BAM/part 4/06 and CMA/2/IV/4

The implementing laboratory must ensure that sampling or analysis is always carried out in accordance with the methodology described and is responsible for this.

Solid manure – Sampling

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1 PRINCIPLE AND SCOPE

This procedure describes the sampling of solid manure by sampling storage (mostly meshes but also containers, sheds, etc.), sampling at the removal of manure from the house to storage via manure tapes or sampling directly in the house for certain types of poultry houses. Its purpose is to obtain a representative laboratory sample.

2 HYGIENE MEASURES

In the case of sampling, the sanitary rules in force at the farm must be complied with at the request of the client (e.g. boots by disinfecting bath, use of overalls on site, showering, etc.).

Both the protective clothing (overalls, footwear, etc.) and all sampling material must be clean when entering the farm in order to avoid cross-contamination from previously visited farms.

3 SAMPLING TERMS AND DEFINITIONS

a. *grip*: a quantity of material taken from the lot at the time of sampling in one operation but pooled to form a composite sample for analysis with other handles;
b. *sampling point*: place in the batch where a grip is taken;
c. *sample*: a portion of material selected from a larger quantity of material;
d. *composite sample*: the amount of material resulting from the aggregation of several grips. The identity of the original grips is lost as a result of such mixing;
e. *laboratory sample*: a sample intended for laboratory inspection or test.
   Note: the laboratory sample is the final sample from the point of view of sampling, but is the initial sample from the point of view of the laboratory;
f. *blending*: combining components, particles or layers in a more homogeneous state;
g. *party*: a defined quantity of material.

4 SUPPLIES

Equipment and containers must be clean.

a. GPS logger or other device with built-in GPS function to record coordinates in WGS84 format in decimal degrees to 5 decimal places;
b. shovel or handshaft (Figure 1) with (straight) raised edges of different volumes (if necessary – see also paragraph 5.3.2);
c. Edelman boron, for example, of the combination type (see Figure 2), with a minimum diameter of 70 mm and a leaf width of 35 mm (see also paragraph 5.3.2);
d. gutsboron: with diameter of 30 mm and effective length of 60 cm, possibly with corresponding spatula to remove the contents from the boron.
e. fork for fattening (reeds);
f. plastic bags;
g. personal protective equipment, depending on the sampling conditions: strong gloves (or 2 pairs of disposable gloves over each other) and boots;
h. collection receptacle in which the grips can be collected: tray, bucket or trolley;
i. scale, sailing or tray of homogenisation;
j. hand scoop or truweel for homogenising, dividing and filling sample containers or scraping the manure tape;
k. sampling bag (plastic) or sample container with cover of at least 5 litres capacity
l. thick pin or labels (pre-printed) identifying the sampling bags or containers;
m. sampling forms for reporting the data of the samples;
n. disinfectant;
o. if necessary/possible: a wheel loader/shovel with shovel loader and driver.

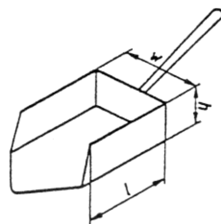

Figure 1 Handshaft

Figure 2 Edelmanboor

5 SAMPLING OF MANURE STORAGE

5.1 LOT AND LOT DEMARCATION

Sampling of solid livestock manure in storage will often be carried out from stockpiles (meshes). Stockpiles are referred to as ‘static lots’. It also includes stored material in bunkers, containers, sheds, loading units, etc.

In the context of analyses for processed manure, sampling will often be carried out from stockpiles containing stored manure products. Stockpiles are referred to as ‘static lots’. It also includes stored material in bunkers, containers, sheds, loading units, etc.
The batch shall be unequivocally described by, inter alia, the dimensions of the batch and the identification of the nature of the material. The dimensions are defined by:

ground area and height. The batch can be further described using typical characteristics (e.g. farmyard manure from different houses...).

If several lots are found at one location, a distinction should be made between the different lots: delineate the parties. As a rule of thumb, each demarcated lot is sampled separately. Each storage unit is thus considered as a separate lot. This means that each pile, container, lorry, silo, laadeenhei d, etc. is in principle sampled separately, unless they contain a similar load. If, within a single storage unit, it is still possible to distinguish between different types of manure, visually or on the basis of origin, origin or type of manure, the lots shall be sampled separately.

Taking into account the practical feasibility of sampling, the maximum lot size shall be 1 000 m^3. Lots larger than 1 000 m^3 are divided into two or more (more or less equal) sub-lots. Each sublot (maximum 1 000 m^3) is then sampled and analysed separately.

5.2 Monster

The purpose of sampling, as described in this procedure, is to take a sample with an average composition that is representative of the whole pile of manure. Therefore, one composite sample is taken per sampling, composed of several grabs (see also point 5.3) taken at different locations in the pile of manure (sampling points).

The minimum sample quantity of a laboratory sample shall be 5 litres.

5.3 Number, location and quantities of ditches

5.3.1 Number of grips

A grip is the amount of manure that can be taken in a single operation at a given location (sampling point) (e.g. one shank, drilling pitch, drilling, handhold).
The larger the lot, the more steps must be taken as a minimum to obtain a representative sample.
In the case of manual sampling, these are the minimum numbers:

- lots ≤ 20 m^3: a minimum of 9 grips;
- lots > 20 m^3 but ≤ 500 m^3: a minimum of 18 grips;
- lots > 500 m^3 but ≤ 1000 m^3: a minimum of 30 grips;
- lots > 1 000 m^3 must be divided into sublots and the sublots must be sampled and analysed separately.

The proposed quantities and numbers are always a minimum requirement. More grabs may be taken and improve the representativeness of the sample.

5.3.2 Grey size

The grey size and equipment used to sample the manure shall be chosen in such a way as to minimise discrimination between the different materials present in the manure.

Farmyard manure with straw or litter
Farmyard manure with straw or litter shall be sampled as a pasty material. The presence of straw can disciripen the sampling by separating the material to be sampled when taking a grip. Due attention should be paid to this.

Preferably, the Edelmanboor should be used to sample a fixed meshole. The typical shape of the Edelman boron ensures a minimum level of friction during the turnaround and hauling of the boron. An Edelmanboor combination type is best suited for the sampling of solid manure: 70 mm diameter and 35 mm leaf width. The Edelmanboor combined blades are slightly wider and more convex than the clay-type blades. As a result, cohesive matter such as manure can still be easily unloaded and removed from the boron. The drilling point is longer than for the clay type, which makes it easier for the boron to turn into the slurry. Other types of drilling may be used provided that they meet the following sampling requirements: the drilling body must be filled and the ratio of manure to litter in the grips must be representative of that in the batch (visual evaluation).

Granular/rulle manure (model example chicken manure)

Granular/rulle manure is sampled using a shovel or shovel. Ensure that the opening of the shells is large enough for the size of the manure particles of the manure to be sampled: the entrance to the shovel shall be at least three times greater than the D95 [2] of the material. The shank shall have upright edges so that the material cannot fall back during moulding and shall have a minimum volume of 250 ml. Conversely, the excess material above the edges of the shank is removed (e.g. with a spatula), as it does not belong to the grip.

5.3.3 SAMPLING LOCATIONS

The different sampling points shall be evenly spaced over the perimeter of the lot.

Sampling enclosed or semi-enclosed storage units such as trucks, containers, bunkers and storage sheds creates an additional difficulty in terms of accessibility/accessibility and homogeneous distribution of the grips. Stockpiles are (usually) accessible along the entire perimeter; trucks, containers are accessible only on one side (often the top). The grips can therefore only be taken on the accessible side, which naturally influences the representativeness of the sample. Where the stockpile is sampled horizontally, a container or truck will have to be sampled vertically, which further increases the difficulty of sampling.

Make sure that sampling is fully described and documented at all times, especially if sampling involves a restriction of accessibility (for example, when only 1 or 2 sides of the hope could be sampled).

[2] D95 (maximum grain size): grain size corresponding to the (hypothetical) sieve size to which a maximum of 5 % (w/w) of the material remains after sieving

5.4 SAMPLING

5.4.1 GENERAL

For the sampling of solid livestock manure, one composite sample consisting of several grabs shall be taken. The steps shall be taken using the following techniques, in order of preference and in order of representativeness of the sample taken:

1. Steel with a wheel charger (highest preference, § 5.4.2)
2. Drilling for steel (Paragraph 5.4.3)
3. Steel sampling by means of landing steel (lowest preference, § 5.4.4)

Where a steel sampling technique is not applicable, a lower preference may be justified. The reasons for this must be included in the sampling form.

5.4.2 METHOD OF SAMPLING BY WHEEL CHARGER/SHOVEL/BULLDOZER

With the wheel charger, take a cargo or shovel at a minimum of 4 (or an equal number greater than 4) different places in the batch. The same number of loads (shovel) shall be distributed in such a way that the same number of shovel heads are removed from the outside (surface) as from the centre (bulk) of the consignment. In order to reach the centre of a large batch, the wheel charger first removes some cargo material from the batch to arrive at the bulk of the material. The removed cargoes are not included in the sampling; only the following shovel from the bulk of the material is charged for sampling.

As with the other techniques, no sample should be taken from the surface of the hope. Therefore, before a shovel is taken from the outside to sample, remove about 30 centimetres from the surface.

Place each sublot (= 1 full shovel) on a clean substrate, separating the different sublots.

-Take a number of grips from each sublot with:

- or a shovel with a capacity of 1 litres or more: Make sure the shovel is fully filled and remove any surplus material that is on top of the shovel/shovel (it is not in the grip). In other words, ensure that all sections have the same volume;
- or a 70 mm Edelmanboor: ensure that each handle is fully filled;
- or a 30 mm gut boron, 60 cm long: make sure each handle is fully filled over its entire length

The number of grips to be taken per sublot depends on the sampling device chosen and is shown in Table 1.

Sampling device	Number of grips per sublot	Minimum number of grabs total
Shovel/shovel minimum 1 l	4	16
Edelmanboor	5	20
gutsboron	5	20

Collect the grips in a bucket, dish, collector or crusher. The grips taken are mixed on site for the preparation of the laboratory sample (see point 7).

5.4.3 MANUAL PROCEDURE FOR EDELMANBOOR SAMPLING

Calculate the volume of the lot to be sampled by estimating the area of land and the mean height and determine the necessary number of gradients according to §5.3.1.

By appointment, the grips are taken at a height of between 30 and 150 cm in relation to the ground. The spatial distribution of the grips shall be homogeneous both horizontally and vertically. Avoid unnecessary risks by walking up or hoping to walk up for unreachable or poorly reachable sampling points. Record these unreachable or poorly reachable parts of the batch.

If X greps (X = 9, 18 or 30) are sampled with a boron, the pile is sampled in X/3 places uniformly distributed over the surface of the pile. Drilling shall be carried out at a downward angle of approximately 45°. The first drilling (on the surface) is removed and the boron is then filled three times from the same borehole so that sub-samples are obtained from different depths in the hope.

Sampling with the Edelmanboor:

Stick the drill to the handle and place it on the blade. Turn the boron right and put some pressure on hope. After about 21 ⁄ 4 complete rounds (of 360°), the boron excavated itself in the hope.

As a result, the boron will be filled up to the bracket with sufficient material. Depending on the composition of the manure heap (pure manure or treated with other waste), more or less frequent rotation is necessary to achieve the desired result.

Raise the boron slightly and rotate upwards. Before unloading the material, shake the drill with the tip on a collector or sails. Rock with the drill on the base of the tray or sails. The material is loose and can be removed from the boron by hand or further ticks on the base.

Remarks:

- Avoid an overcrowded boron. This makes it very difficult to unload the material. An overcrowded boron can have a lot of suction power when hovered, which makes collection very difficult and causes sample loss.
- Depending on the consistency of the manure, it may help to apply more or less force to the boron during drilling so that the material is slightly compacted.
- It is important that all drilling rigs have the same volume. Stoppers with significantly less volume should be removed.

The grips taken are mixed on site for the preparation of the laboratory sample (see point 7).

5.4.4 MANUAL PROCEDURE FOR SHOVEL SAMPLING

Calculate the volume of the lot to be sampled by estimating the area of land and the mean height and determine the necessary number of gradients according to § 5.3.1.

By appointment, the grips are taken at a height of between 30 and 150 cm in relation to the ground. The spatial distribution of the grips shall be homogeneous both horizontally and vertically. Avoid unnecessary risks by walking up or hoping to walk up for unreachable or poorly reachable sampling points. Record these inaccessible on poorly accessible parts of the batch.

If X slides (X = 9, 18 or 30 see § 5.3.1) are taken with a shovel or shovel, the X slides are uniformly distributed over the surface of the pile.

Each time:

- X/3 grips taken at a depth of at least 30 cm below the surface after removal of the surface layer;
- X/3 grips taken at a depth of at least 60 cm below the surface after removal of the surface layer;
- X/3 grips taken at a depth of at least 90 cm below the surface after removal of the surface layer.

Shovel/shovel sampling with adapted opening:

Push the shovel/shovel upwards as far as possible in the material. Ensure that the shovel/shovel is fully filled, and that all grips are of approximately the same size. Remove excess material that is on top of the shovel/shovel (it is not in the grip). Each time, remove the material from the shovel/shovel in the collector or on a tarpaulin.

The grips taken are mixed on site for the preparation of the laboratory sample (see point 7).

6 SAMPLING OF POULTRY MANURE IN THE HOUSE

6.1 DEMARCATION

Three specific methods can be distinguished for the sampling of poultry manure in the house, depending on the type of house:

a. sampling at turning off the manure belts (battery shed);
b. sampling in a housing housing area with gratings;
c. sampling in a stall with ground housing without gratings.

Depending on the type of house, a different technique will have to be used. It is therefore very important to first check thoroughly which method of sampling should be followed.

6.2 MONSTER

The purpose of sampling, as described in this procedure, is to take a sample with an average composition that is representative of the entire house. As a result,

sampling one composite sample taken from multiple grips taken from different places in the house or from the manure belts (sampling points).

6.3 Sampling at tipping of manure belts (battery stall)

6.3.1 Number, location and quantity of grips

Preferably, sampling should be carried out on the cross-band (collection band of the different manure bands, floors) just before the manure enters the shed, i.e. immediately outside the house (Figure 3). If this is not possible, the grips must be taken from the manure belts themselves. Please note that there is a proportional sampling of the different floors.

The minimum number of handles shall be 18.

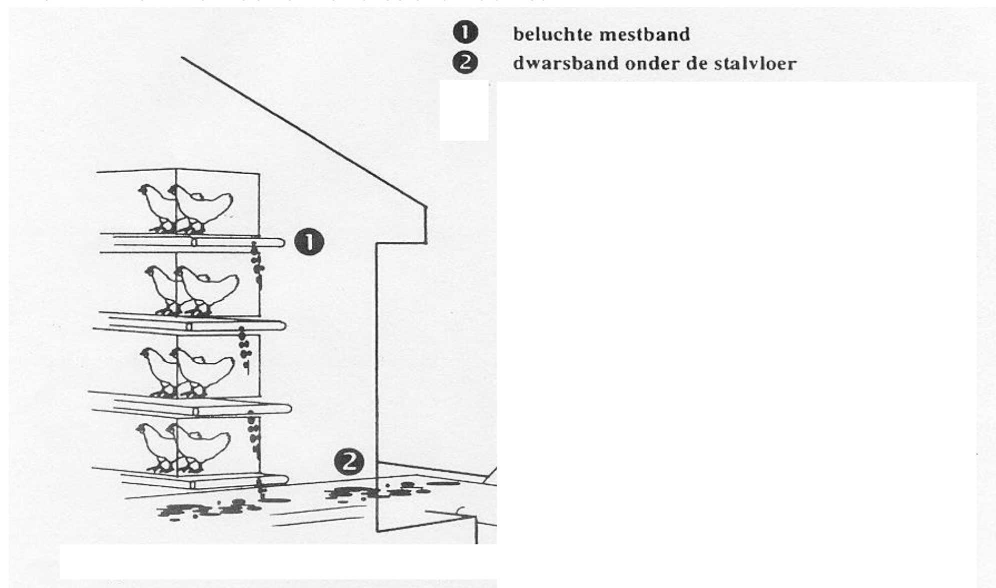

Figure 3 Situation of manure belts in a battery stall

6.3.2 Sampling

Keep the dish, tray, bucket or trolley underneath the band, skip the manure with a hand scoop or trunk over the entire band inside. Wait one to two minutes and repeat the same operation. The total turn-off time varies from company to company, but is usually around 30 minutes.

The grips taken are mixed on site for the preparation of the laboratory sample (see point 7).

6.4 Sampling in a stable with ground housing without grating

6.4.1 Number, location and quantity of grips

Note: work should be done on a surface basis.

In general, the sample of solid manure must be representative. It is therefore necessary to take grips in various places in the house. There are differences in manure composition within the house, as the manure may or may not be close to a feeding or drinking place.

An example of sampling is developed below:

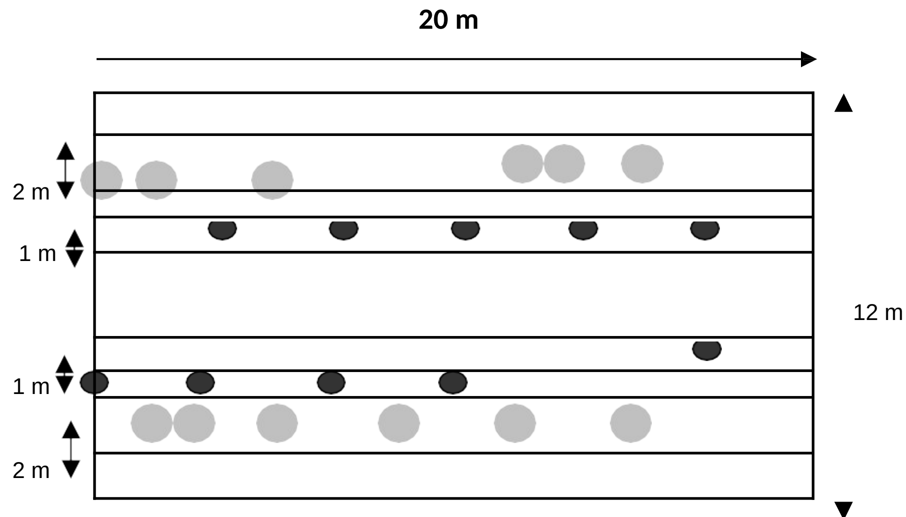

Figure 4 Situation of feeding points (grey) and drinking points (black)

Step 1
Determine the length and width of the house; the farmer is usually aware of this.
In this example: 20 in 12 m.

Step 2
Count the number of feeding and drinking lines (Figure 4) and make the best possible estimate of the manure area affected by each of them.
In this example:
2 lines – positioning: 4 of the 12 m length affected by the line (= 2/6).
2 waterlines – location: 2 of the 12 m length affected by the waterline (= 1/6). 6 out of 12 m length not affected (= 3/6).

Step 3
Calculation of the number of grips: the number of steps to be taken must always be at least 18. Always take a multiple of the denominator of the location distribution.
In this example: a multiple of six steps must be taken, i.e. 18 steps (6 x 3).

Step 4
Determine the number of crossings to be distributed to the house (Figure 5). The easiest thing to take as a number here is the multiple taken in step 3 to arrive at the number of grabs.
In this example: in step 3, 3 is the multiple used, i.e. 3 crosses.

Figure 5 Situation of crossings

Step 5

Distribution of the grips: proportional to the area distribution above. Sampling points shall be selected in such a way that all parts of the house are covered approximately equally.

In this example:

Bird line samples: 2/6 – X 3 6 out of 18 grabs.

Waterline samples: 1/6----x 3 3 out of 18.

Other samples: 3/6-----x 3 9 out of 18 bars.

In order to keep the case clear, it is recommended that the zigzag be carried out three times, each time for a different type of grip (Figure 6). Cross the pen and grab the line, come back and graze the waterline and the other *grabs* for the third time.

Figure 6 Situation of grips to be taken

6.4.2 SAMPLING

Beacon with the hand scoop or truweel to cover a certain area, for example 15 cm by 15 cm. Place the entire layer of the defined surface in the trolley or bucket. It is important to define and create an equal area for each rope (18 grids).

The grips taken are mixed on site for the preparation of the laboratory sample (see point 7).

6.5 SAMPLING IN A HOUSING HOUSING UNIT WITH GRATE

6.5.1 NUMBER, LOCATION AND QUANTITY OF GRIPS

Note: work should be carried out on a **volume basis**.

In general, the sample of solid manure must be representative. It is therefore necessary to take grips in various places in the house. There are differences in manure composition within the house, as the manure may or may not be close to a feeding or drinking place. An example of an establishment of such a house is shown below (Figure 7: side view, Figure 8: top view)

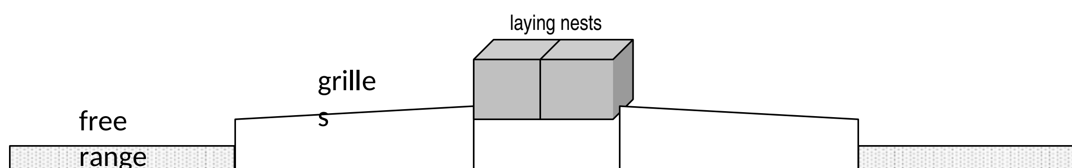

Figure 7 Cross-sectional area of a house with ground housing with beech

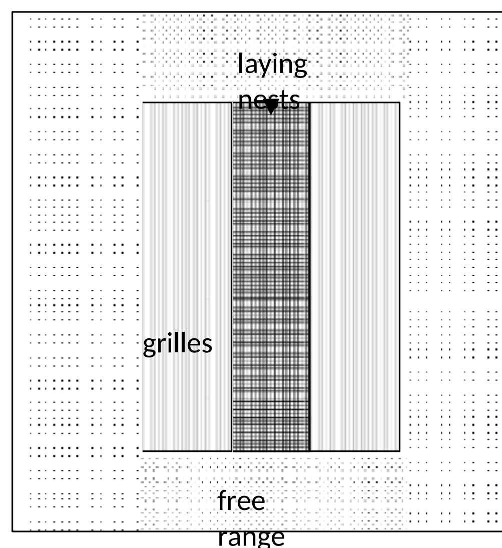

Figure 8 Upper view of a house with ground housing with beech

6.5.2 SAMPLING

Provide two separate trays, dishes or buckets: one to collect the grips taken under the grilles and one to collect the grilles from the free range.

Step 1 *Determination of the ratio*
The quantity of material to be collected is distributed under the grid and in the free range on a volume basis. To this end, checks are carried out (to be asked to the farmer or to

estimate in situ) the volumes of manure in the free range on the one hand and under the grilles on the other to determine the ratio between the two. The amount of material to be collected under the grid and in the free range shall be in the same proportion.

In the above example at the end of the cycle, the amount (volume) of manure under the grilles (40 cm high) was equal to the amount (volume) of manure in the free range (7 to 8 cm high). There was therefore a ratio of 1 to 1. That is, the amount of material taken under the grids must be equal to the amount of material collected from the free range.

<u>Step 2 Girls</u> *out of the free range*
Beacon with the hand scoop or truweel to cover a certain area, for example 15 cm by 15 cm. Place the entire layer of the defined surface in the first container, dish or bucket. It is important to define and create an equal area for each rod. Take <u>at least 9 grips</u>.

Please note: again, the distribution of feeding lines and any water lines should be taken into account as described in point 6.4.1.

<u>Step 3</u> *steps below the grid*
Use a shank or Edelmanboor (possibly gutsboor) for sampling. Sample to the bottom and ensure proper distribution of grips across multiple locations below the grid. Collect the material in the second container, dish or bucket. Take as many steps as possible until the amount of material collected in the second container/shell/bucket has the <u>same ratio</u> compared to the first container/shell/bucket as determined in Step 1.

In the example: This would mean that both buckets would have to contain the same amount of collected material, as the ratio was 1 to 1.

The grips taken are mixed on site for the preparation of the laboratory sample (see point 7).

7 HOMOGENISE AND PREPARE THE LABORATORY SAMPLE

The grips taken are mixed in situ to form a homogeneous composite sample. The composite sample shall, if possible, be placed whole in the sample container. If the sample size of the composite sample is too large to transfer the whole sample into the sample container (which will usually be the case), the amount of the composite sample is pre-reduced (by quartering) to the amount of material needed to prepare the laboratory sample.

a. <u>Homogenise:</u>
   All grabs from the batch shall be spread on an inert substrate. To do so, use a plastic dish, sails (a bucket is less suitable for further dividing/quartering). Use a shovel or larger shovel for mixing.
   A good homogenisation technique is to pick up the material by creating the outer sides of the material towards the centre each time. The hope formed is then flattened and spread back. This procedure is repeated several times.

Another way of working is to create the material several times from one hope to another. For this purpose, use 2 dishes or sails (or a combination of both) if the amount of material is too large to achieve it within one surface.

b. Reducing by quartering:
Spread the homogenised manure sample in a circular, reduced layer thickness into the collector box or tray. Divide the circle through two diagonals in 4 quarters.
Remove two opposite quarters (not included in the laboratory sample). Combine the remaining quarters and homogenise again. If necessary, repeat the operation until a sample of the correct size (see paragraph 5.2) is obtained.

The homogenisation, reduction and filling of the sample containers may also be carried out by hand if desired. In this case, for hygienic reasons, it is recommended that 2 pairs of gloves be worn together.

The guidelines on sample size set out in points 5.2 and 6.2 also apply to the packaging of the material. The laboratory sample is packed in a solid plastic sample bag or a well-sealed container such as a bucket with a lid.

If the conditions or facilities do not allow the compounding and homogenisation to be carried out in a responsible manner, the handles shall be individually wrapped and handed over to the laboratory with the necessary instructions for compounding the composite sample.

Alternatively, manure composition can be sampled during transport. The sampling of cargo must be carried out in accordance with CMA/1/A.15 § 3.2.5. (Sampling of containerised cargo).

8 IDENTIFICATION OF SAMPLES

The tag (number, barcode...) of the sample must be unambiguous so that afterwards no misunderstandings may arise as to the origin of the sample.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be indicated on the (digital) sampling form accompanying the sample:

a. client, address, farmer number and operator number;
b. client and/or third parties present at the sampling;
c. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number);
d. date and time of sampling;
e. own sample number or sample coding;
f. type of manure (e.g. pig manure, broiler manure, cattle manure). The manure codes used by the Flemish Land Agency and included in SMIL must be used for this purpose;
g. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the sampled house or storage. Those coordinates shall be determined locally by a GPS device and stored electronically.

h. description of the sampling location (e.g. blade, shed, container, battery housing, housing housing with/without bedding...);
i. description of sampling (carried out in the house, hope, sampling pattern, house description...) including the sampling equipment used (shovel, Edelmanboor...);
j. a photograph of the lot taken after sampling;
k. the estimated volume of the lot
l. the number of fractional bars needed to obtain the minimum sample volume required; volume of the field and laboratory sample and whether or not to carry out sample reduction in situ;
m. significant remarks and/or deviations that may affect the interpretation of the analytical result.

The laboratory's sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

9 MONSTERCONSERVATING AND TRANSPORT

- The sample is stored chilled (5 ± 3) °C immediately after sampling. All transports must be refrigerated (with refrigeration box or refrigeration inside the wagon).
- Refrigeration must be traceable during storage.
- The sample must be prepared for analysis no later than the seventh day after sampling.

10 REFERENCES

a. From working instructions: taking of poultry samples May 2004.Drawing up in the framework of the project "Evaluation of manure excretion figures and manure composition figures for poultry". Cooperation between the Belgian Soil Service and the livestock pilot company in Geel, on behalf of the Mestbank
b. VLM working document, problems/bottlenecks, suggestions on sampling of different manure types. Annual consultation of laboratories: Discussion of the sampling method according to the compendium. (7227/06/2003)
c. VLM working document, Evaluation of manure excretion and composition figures for poultry, Annex 1: Sampling protocols for the 'Mest and Mineral Poultry Farming Practice Figures' project, Mestbank project proposal June 2003
d. Coffey R.D., Parker G.R., Laurent K.M. (2003). Sampling animal manure. University of Kentucky, College of Agriculture, ID 148.
e. Hochmuth G.J., Jones J.T. (2003). Collection a Poultry Litter Sample for Analysis. University of Florida, IFAS Extension.
f. Goan C., Walker F. (2004). Poultry litter Sampling and testing. University of Tennessee, Agricultural Extension Service, SP563.

Solid treated manure – Sampling

CONTENT

1 Scope 3

2 Hygiene measures 3

3 Identification of samples 3

4 Sample preservation and transport 4

1 SCOPE

For the sampling of solid treated manure such as solid (and pasty) digestates and compost from the (co) processing or (co) processing of livestock manure, reference is made to CMA/1/A.15 *Waste – raw materials: Solid materials sampling techniques*, CMA/1/A.17 *Waste – raw materials*: *Sampling techniques (liquid) pasty materials* and CMA/1/A.18 *Waste – raw materials: sample preparation on site*.

2 HYGIENE MEASURES

In the case of sampling, the sanitary rules in force at the farm must be complied with at the request of the client (e.g. boots by disinfecting bath, use of overalls on site, showering, etc.).

Both the protective clothing (overalls, footwear, etc.) and all sampling material must be sufficiently clean when entering the farm to avoid cross-contamination from previously visited farms.

3 IDENTIFICATION OF SAMPLES

The tag (number, barcode...) of the sample must be unambiguous so that no afterwards misunderstandings may arise as to the origin of the sample.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be indicated on the (digital) sampling form accompanying the sample:

a. client, address, farmer number and operator number;
b. client and/or third parties present at the sampling;
c. reference of MAD/Neighbouring scheme BR manure disposal document in case of cargo sampling;
d. type of manure (e.g. (solid fraction) digestate, compost...). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. The same description as the one used on the MAD, if applicable, shall be used;
e. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the sampled storage. Those coordinates shall be determined locally by a GPS device;
f. description of the place of sampling (for example, hope, shed, container, etc.);
g. sampling description (sampling pattern);
h. the number of fractional bars needed to obtain the minimum sample volume required;
i. sampling equipment used;
j. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number);
k. date and time of sampling;

l. own sample number or sample coding;
m. significant remarks and/or deviations that may affect the interpretation of the analytical result.

The laboratory's sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

4 MONSTERCONSERVATING AND TRANSPORT

- The sample is stored chilled (5 ± 3) °C immediately after sampling. All transports must be refrigerated (with refrigeration box or refrigeration inside the wagon).
- Refrigeration must be traceable during storage.
- The sample must be prepared for analysis no later than the seventh day after sampling.

Solid manure and solid treated manure – Sample pre-treatment

CONTENT

1 Principle 3

2 Material 3

3 Reagents 4

4 Procedure for homogenisation and reduction 4

5 Method for sample dried with tartaric acid 4

6 Calculation of the drying factor 5

7 Quality check 5

8 Reference 5

The following method describes the procedure for the pre-treatment of solid manure samples prior to analysis. This is based on a laboratory sample with a volume of 5 litres.

Samples of solid manure with a dry matter content between 15 and 30 % can be classified with both liquid manure and solid manure. The subdivision of the laboratory sample delivered into the relevant matrix type and the related sample pre-treatment can be carried out based on the estimated dry matter content combined with a visual assessment. However, the physical state based on the visual observation is determinative for carrying out sample pre-treatment.

The sample preparation of solid treated manure is described in the Compendium for sampling and analysis in implementation of the Materials Decree and the Soil Decree, and more specifically in CMA/5/B.1 *Sample preparation of Fertiliser Soil Improvement* Agents (https://emis.vito.be/nl/referentielabo-ovam).

For both solid manure and solid treated manure, samples shall be:

a. be kept cool at a temperature of (5 ± 3) °C at all times to avoid conversions;
b. processed for analysis no later than the seventh day after sampling.

1 PRINCIPLE

In this procedure, the raw sample as a whole is first thoroughly mixed. A portion of at least 500 g is taken as a laboratory sample after mixing and quartering.

After mixing/grinding, provision shall be made for representative sub-samples for the determination of:

a. dry matter at 105 °C: fresh sample;
b. total phosphorus: sample dried at 105 °C and milled to < 0.5 mm (see BAM/Part 4/03);
c. total nitrogen, ammoniacal nitrogen: sample dried with tartaric acid and ground to < 0,5 or 1 mm (see point 5).

Note: The test sample for dry matter determination may continue to be used for the determination of total phosphorus.

2 MATERIAL

The usual laboratory glassware and also:

a. reducer;
b. dry tinplate;
c. composite plastic cards made of inert material;
d. drying dishes;
e. balance, accurate to at least 1 mg;
f. dosing device;
g. oven set at a temperature of 70 °C ± 5 °C;
h. cross-impact mill, fitted with a sieve with openings having a diameter of 1 mm.

A cutting or knife mill is recommended as a grinder for solid manure (see Figure 1).

Figure 1: Knife mill

For straw samples, the laboratory sample shall be homogenised by successively placing several sub-samples in the cutting mill until a sufficiently representative quantity for analysis is obtained.

3 REAGENTS

Use only reagents of analytical grade:

a. ultra pure water;
b. tartaric acid solution c ($C_4H_6O_6$) = 0,445 mol/l: dissolve 667 g tartaric acid in about 8 l of water and make up to 10 l with water.

4 PROCEDURE FOR HOMOGENISATION AND REDUCTION

The raw sample is ‘thoroughly mixed’ or ‘homogenised by manual conversion’. Remove non-manure objects. Then prepare a sub-sample of at least 500 g.

Divide the sample into parts A and B if one or more determinations in the fresh product are also to be carried out in the sample.

To do so, proceed as follows: Collect the ground sample on a drying can, mix and divide the sample with a mixing card into two equal portions by repeated application of the quartering method.

If not carried out on the same day, store the part A intended for analysis in the fresh product at (5 ± 3) °C. Respect the required shelf life for the parameters to be determined.

Continue with Part B according to point 5 to obtain a sample dried with tartaric acid.

5 METHOD FOR SAMPLE DRIED WITH TARTARIC ACID

Weigh an empty container to the nearest 0.1 g (mass m_0).

Using a spoon, take a sub-sample from at least 10 different places of the ground and mixed sample. Place approximately 250 g of the sample in a container and weigh to the nearest 0.1 g (mass m_1).

Using a dosing apparatus, add 300 ml of tartaric acid solution. Mix the added amount of tartaric acid solution with the spoon through the sample to obtain a homogeneous suspension. The spoon must be crushed with any lumps that may be present.

Note 1: Tartaric acid is added to prevent ammonia from evaporating during sample preparation by drying.

Note 2: a blank procedure is carried out for each batch (25 g white sand + 30 ml tartaric acid ($c_{(C_4H_6O_6)}$ = 0.445 mol/l)). The parameter ammonium is determined on this procedure blank, for extraction and analysis see BAM/part 4/05. As a guideline, the content of the procedure is blank for ammonium < 0.1 kg N/1000 kg

Dry in oven at 70 °C ± 5 °C until air-dry. During drying, shake the sample with the spoon and cycle. Weigh the container and its contents to the nearest 0.1 g (mass m_2). Place the air-dry sample in the crucible and reduce to < 0,5 or 1 mm.

6 CALCULATION OF THE DROOGFACTOR

In the case of further determinations carried out on the sample dried with tartaric acid, the drying factor must be included in the final calculations.

Calculate the drying factor (D) from the equation:

$$D \quad \frac{m_2 \quad m_0}{m_1 \quad m_0}$$

with:
D: drying factor;
m_0: mass, in grams, of the empty container;
m_1: mass of the drying dish and fresh sample, in grams;
m_2: mass of the container and its contents after drying, in

grams, rounded off to 3 decimal places.

7 QUALITY CHECK

As a quality control, at least 1 sample per day shall be analysed in duplicate for at least 1 relevant parameter. For this purpose, 2 sub-samples are taken after sample pre-treatment and go through the entire analytical route.

8 REFERENCE

NEN 7431: 1998 Animal manure and manure products – Sample pre-treatment by mixing, drying and grinding – Stackable manure

Solid manure and solid treated manure – dry matter content

CONTENT

1 Principle 3

2 Equipment and materials 3

3 Practice 3

4 Remarks 3

5 Calculations 4

6 Reference 4

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 4/02.

The dry matter content (in relation to fresh material) must be determined to allow conversion to fresh material in the determination of total phosphorus.

The method consists of drying a predetermined quantity of homogenised sample at a temperature of 105 °C ± 5 °C for a specified time.

2 EQUIPMENT AND MATERIALS

a. drying dishes or suitable containers for a large quantity of sample. It must be possible to divide 250 g of the sample into them so that the maximum thickness is between 2 and 2.5 cm;
b. drying oven, mechanically ventilated, set at a temperature of 105 °C ± 5 °C;
c. desiccator;
d. balance, capable of weighing to the nearest 0,001 g;
e. mill, for reducing the size of the dried sample.

3 PRACTICE

Departure from fresh material not pre-treated with tartaric acid. A sufficient subsample size (± 250 g) shall be taken to ensure the representativeness of the sample.

The containers are prepared by drying them in the oven at 105 °C ± 5 °C and cooling. The empty containers are weighed (m_0).

Place about 250 g of material in the drying dish. Re-weigh (m_1).

Place dishes in the pre-heated oven. Dry for 24 hours at 105 °C ± 5 °C. Remove flasks from the oven and allow to cool to ambient laboratory temperature. Given the size of the dishes, it is not practically feasible to allow the dishes to cool in a desiccator. The error introduced by cooling in the lab is negligible due to the large quantity of sample. Reroads (m_2)

Note: If the dried specimen is further used for the total P determination, the sample is ground to < 0.5 mm.

4 REMARKS

a. Weighings shall be made to the nearest 10 mg.
b. The total P content can be determined on a test portion of the dried sample.

5 CALCULATIONS

$$DS \quad m_2 \quad m_0 \, 1000 \, m_1 \quad m_0$$

with:
DS: dry matter content in kg/1000 kg
VM; m_0: mass of empty container in grams;
m_1: container mass + fresh sample in g; m_2: container mass + dry sample in g.

Round the result to 2 decimal places for values ≤ 1, 1 decimal place for values > 1 and to one integer for values > 100.

6 REFERENCE

NEN 7432: 1998 Animal manure and manure products – Determination of dry matter and organic matter contents – Gravimetric method

Solid manure and solid treated manure – Total phosphorus

CONTENT

1 Principle 3

2 Equipment and materials 3

3 Reagents 3

4 Design procedures 3

4.1 Digestion with rendering and HNO_3 destruction 3

4.2 Digestion with HNO_3/HCl (aqua regia) 4

5 Analytical determination of phosphorus in the rendering solution 4

6 Calculations 5

7 References 5

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 4/02.

For the determination of total P in solid manure or solid treated manure, the following methods of rendering and analysis may be applied:

- The dried sample is ashed at 550 °C, then the ash is dissolved in HNO_3. The determination of phosphorus in the solution is carried out spectrophotometrically or with ICP-AES.
- The dried sample is solubilised with an aqua regia (HNO_3: HCl) rendering. The determination of phosphorus in the solution is performed with ICP-AES.

For the determination of total P in solid manure, the sample may be solubilised with sulphuric acid, hydrogen peroxide and copper sulphate according to NEN 7433. The determination of phosphorus in the solution is carried out spectrophotometrically or with ICP-AES. On the same solubilising solution, it is possible to determine total N (= Kjeldahl-N), provided that the sample was dried with tartaric acid during sample preparation.

2 EQUIPMENT AND MATERIALS

a. ashing dishes;
b. oven set at 550 °C ± 25 °C;
c. desiccator;
d. heater plate;
e. ash-free filter paper;
f. acid-resistant rendering block, programmable to at least 105 °C;
g. disposable rendering tubes of 50 ml, acid-resistant;
h. compact condenser.

3 REAGENTS

a. HNO_3, 14 mol/l;
b. HNO_3, 1 mol/l;
c. HCl, 12 mol/l.

4 DESTRUCTION PROCEDURES

4.1 O NOTIFICATION WITH INCINERATION AND HNO_3 DESTRUCTION

Weigh, to the nearest 1 mg, 2,5 to 0.5 g of dry sample, ground to < 1 mm (m).

Ash that sample at 550 °C for 4 hours. The axle shall be grey white. If the ash is not white: add a few drops of $_{14M\ HNO}$ 3 and ash again for 1 hours.

Quantitatively transfer the ash into a 100 ml beaker containing 20 ml 1M HNO_3 1M. Leave to distil for one hour on a heater or in a hot water bath.

Filter and collect filtrate in a 100 ml volumetric flask and rinse well with 1M HNO_3. Make up to 100 ml with 1M HNO_3.

4.2 O NOTICE WITH HNO_3/HC L (AQUA REGIA)

The rendering may also be carried out in a heatable rendering block with rendering tubes fitted with a compact condenser.

Note: Alternatively, a watch glass or a shut-off cap (tightening and half turn back) may be used for the condenser.

Weigh 1 g of dried sample, ground to < 0.5 mm, to the nearest 1 mg (m) in a rendering tube. Gradually add 4 ml of 14M HNO_3 and 12 ml of 12M HCl.

Place the condenser on the rendering tubes. Leave rendering tubes at room temperature to allow a slow reaction of the organic matter. Carry out the rendering programme with incremental warming, for example:

a. warm up in 20 minutes to 45 °C, 5 minutes at 45 °C;
b. warm up in 10 minutes to 65 °C, 10 minutes at 65 °C;
c. warm up to 105 °C, 120 °C for 105 minutes.

Filter the sample after destruction. Stirring with ultra pure water to make up to 50 ml.

5 ANALYTICAL DETERMINATION OF PHOSPHORUS IN DESTRUCTION OPLOSSING

The analytical determination of phosphorus in the solubilising solution may be carried out according to:

a. NBN EN ISO 11885: 2009 Water quality – Determination of selected elements by Inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885: 2007).

The analytical determination of phosphorus in the solubilising solution can be performed spectrophotometrically according to:

a. NBN AND ISO 6878: 2004 water quality – Determination of phosphorus – Ammonium molybdate spectrometric method;
b. NBN AND ISO 15681-1: 2005 water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 1: Method by flow injection analysis (FIA) (ISO 15681-1: (2003);
c. NBN AND ISO 15681-2: 2005 water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2: (2003);
d. ISO 15923-1: 2013 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection.

Note: For the spectrophotometric methods, the phosphorus content is determined in a five-fold dilution of the solubilising solution.

6 CALCULATIONS

The measured phosphorus concentration shall be converted to a concentration C_P (kg P_2O_5/1000 kg) in fresh material according to the following formula:

$$C_P = \frac{C_1}{M} \times F \times V \times DS \times 2.29 \times \frac{1}{1000}$$

with:
C_P: phosphorus concentration in the original sample in kg P_2O_5/1000 kg VM;
C_1: measured phosphorus concentration obtained in mg P/l;
f: dilution factors, if any;
V: volume of solubilising solution in litres;
DS: dry matter content determined in kg/1000 kg VM;
m: mass of dry sample taken under processing in grams.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

7 REFERENCES

a. NEN 7435: 1998 2nd draft Animal manure and manure products – Determination of phosphorus content in destruates
b. NBN EN 13650: 2001 Soil improvers and growing media – Extraction of aqua regia soluble elements
c. NBN EN ISO 6878: 2004 Water quality – Determination of phosphorus – Ammonium molybdate spectrometric method
d. NBN EN ISO 15681-1: 2005 Water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 1: Method by flow injection analysis (FIA) (ISO 15681-1: 2003)
e. NBN EN ISO 15681-2: 2005 Water quality – Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA) – Part 2: Method by continuous flow analysis (CFA) (ISO 15681-2: 2003)
f. NBN EN ISO 11885: 2009 Water quality – Determination of selected elements by Inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885: 2007)
g. ISO 15923-1: 2013 Water quality – Determination of selected parameters by discrete analysis systems – Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection.
h. C. Vanhoof, A. Cluyts, K. Duyssens, E. Poelmans, Wendy Wouters and K. Tirez, *Sustainability of N parameters and destruction of P in manure samples*, VITO report 2011/MANT/070, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/rapport_mest_N_en_P_2011.pdf
i. C. Vanhoof and K. Tirez, *Evaluation of methods of analysis for the determination of inorganic parameters in digestates*, VITO report 2012/MANT/R/005, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/Rapport_2011_digestaten_finaal.pdf
j. C. Vanhoof and K. Tirez, *Harmonisation of inorganic analytical methods for the analysis of compost, manure and various digestate* streams, VITO report 2012/MANT/R/086, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/2012_Bodemimproved_resources_finaal.pdf

Solid manure and solid treated manure – Ammonium nitrogen

CONTENT

1 Principle **3**

2 Extraction procedure **3**

2.1 Equipment and materials *3*

2.2 Reagents *4*

2.3 Practice *4*

3 Determination of ammonium in the extract after steam distillation **4**

3.1 Principle *4*

3.2 Procedure *4*

3.3 Calculations *4*

4 Spectrophotometric determination of ammonium in the extract **5**

4.1 Measurement of ammoniacal nitrogen in leaching *5*

4.1.1 Ammoniacal nitrogen by manual spectrophotometric method 5

4.1.2 Ammoniacal nitrogen with continuous flow analysis (CFA) using spectrophotometric detection 6

4.1.3 Ammoniacal nitrogen with ion chromatography 6

4.1.4 Ammoniacal nitrogen with a discrete analytical system (spectrophotometric detection) 6

4.2 Calculations *7*

5 References **7**

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 4/02.

For the determination of the ammonium content in solid manure or solid treated manure, a KCl extraction is performed from the sample, dried after adding tartaric acid and milled to < 0,5 or 1 mm. The ammonium content of the extract is then determined according to:

a. ISO 5664: 1984 water quality – Determination of ammonium – Distillation and titration method;
b. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method;
c. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection;
d. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li^+, Na^+, NH_4^+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998);
e. 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).

Note: for heavy load matrices, due consideration shall be given to the analysis to measure interference free.

The following controls are included in the determination of ammonium in solid or solid treated manure:

— Procedure blank: during the drying of the sample with tartaric acid, a blank procedure is carried out on each batch (25 g white sand + 30 ml tartaric acid ($c(C_4H_6O_6)$ = 0.445 mol/l)). The concentration of ammonium-N in fresh material, expressed as kg N/1000 kg, is calculated as follows:

$$C_N = \frac{C_I \times V_{EXT}}{M} \times D$$

with:

C_N concentration of ammonium-N in fresh material, expressed as kg N/1000 kg
C_i concentration of ammonia N in the extract after blank correction, in mg N/l
m mass of extracted sample, in g (i.e. 5 g, see point 2.3)
V_{ext} volume of extraction solvent, in ml (i.e. 50 ml, see point 2.3)
D drying factor determined according to BAM/part 4/02

As a guideline, the content of the procedure is blank for ammonium < 0.1 kg N/1000 kg. (see BAM/part 4/02 § 5)

- Blank KCl solution: this is the extraction solution
- Optional: QAQC 1 Lines control sample for accreditation package A.2.1 Fertiliser/soil improvers – inorganic parameters (see CMA/6/D)

2 EXTRACTION PROCEDURE

2.1 Equipment and materials

a. shaking device;

b. ploughing filter or similar.

2.2 Reagents

a. 1 mol/l potassium chloride solution: dissolve 74.6 g KCl in 1 l of water.

2.3 Practice

Weigh to the nearest 5 mg (m), 1 g of the sample dried after the addition of tartaric acid.

Add 50 ml 1M KCl (v_{ext}). Shake for 30 minutes at constant temperature.

The extract is centrifuged or filtered. Rinse the filter with sample solution. The first part of the filtrate is rejected. Collect the remaining filtrate in a dry container.

3 DETERMINATION OF AMMONIUM IN THE EXTRACT AFTER STEAM DISTILLATION

3.1 Principle

Ammonium in a solution containing alkali-labile nitrogen components is exempted by the addition of MgO. The resulting ammonia is released by steam distillation and collected in excess acid. The amount of ammonium is determined by return titration.

Sodium hydroxide is not used during distillation and the duration of distillation is kept as short as possible to avoid the determination of alkali-labile organic nitrogen compounds.

3.2 Procedure

The procedure described in ISO 5664: 1984 shall apply with the following additions:

a. § 2.3 sensitivity: not applicable;
b. Paragraph 4 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
c. Paragraph 6 Sampling: not applicable;
d. § 7.1 selection of test portion volume ($V_{test\ portion}$): other volumes may be used if they are appropriate for this application;
e. Paragraph 7.2.3: other endpoint detections are also possible.

3.3 Calculations

The pre-treatment of samples shall be taken into account.

The result is expressed as nitrogen concentration C_N (kg N/1000 kg) in fresh material using the following formula:

$$C_N = M_N \times \frac{(R_1 - V_0) \times C_{HCL}}{M} \times D \times \frac{V_{EXT}}{V_{TEST\ PORTION}}$$

with:
C_N: concentration of ammonium in the original sample in kg N/1000 kg VM; M_N:
molar mass of nitrogen (14.007 g/mol);
V_1: volume at titration of the sample in ml; V_0: volume at titration of the blank in ml;
m: mass, in grams, of the sample taken under processing; C_{HCl}:
concentration of hydrochloric acid in mol/l,
D: drying factor;
V_{ext}: volume of extraction solvent in ml;
$V_{test\ portion}$: volume of test portion in ml.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

4 SPECTROPHOTOMETRIC DETERMINATION OF AMMONIUM IN THE EXTRACT

The determination of ammonium can be carried out in the extract by one of the following methods:

a. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method;
b. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection;
c. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li^+, Na^+, NH_4^+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998);
d. 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).

4.1 MEASUREMENT OF AMMONIACAL NITROGEN IN LEACHING

4.1.1 AMMONIACAL NITROGEN BY MANUAL SPECTROPHOTOMETRIC METHOD

The procedure described in ISO 7150-1: 1984 shall apply with the following additions:

a. § 1.5 sensitivity: the minimum absorption shall be checked, but the concentration used and the procedure used may differ from the described ISO procedure;
b. Paragraph 4 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
c. Paragraph 6 Sampling: not applicable;
d. Paragraph 7.3: other relevant concentration levels may be used if they are appropriate for this application. The same procedure shall be applied for samples and standards;
e. § 7.5 calibration: the methodology may deviate from the described procedure if the calibration line is established with at least 5 calibration solutions and complies with this application.

4.1.2 Ammoniacal nitrogen with continuous flow analysis (CFA) using spectrophotometric detection

The procedure described in NBN EN ISO 11732: 2005 shall apply with the following additions:

a. § 3 determination of ammoniacal nitrogen with flow injection analysis (FIA) and spectrophotometric determination: not applicable;
b. Paragraph 4.3 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
c. Paragraph 4.4.3 Sampling: not applicable;
d. Paragraph 4.5.2 Control performance tool: the minimum absorption shall be checked, but the concentration used and the procedure used may differ from the described ISO procedure;
e. § 4.5.3 blank control reagents: blank control of reagents is optional.

4.1.3 Ammoniacal nitrogen with ion chromatography

The procedure described in NBN EN ISO 14911: 1999 shall apply with the following additions:

a. Paragraph 6 Reagents: other reagents or concentrations may be used if they are appropriate for this use;
b. § 8 quality requirements for separation column: other concentrations may be used to evaluate separation conditions;
c. Paragraph 9 Sampling: not applicable.

4.1.4 Ammoniacal nitrogen with a discrete analytical system (spectrophotometric detection)

The procedure described in NBN EN ISO 15923-1: 2024 shall apply with the following additions:

a. § 5 other reagents and concentrations may be used if they are appropriate for this use;
b. Paragraph 7 shall not apply;
c. Annex B to H: deviations from the implementation of the described methods 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;
d. § 8.1 and § 8.2: additional quality control for the determination of parameters ammonium, nitrate and nitrite in leaching. The analysis of these samples must include at least 1 of the following quality checks:
    1) analysis of the sample by at least 1 degrees, with a bias of not more than 10 % relative to the theoretical value;
    2) at least 2 measurements of the same sample with a dilution factor differing by at least a factor of 2, resulting in 2 measurement results within the measuring range differing by not more than 10 %.

Note: False negative results may occur in the determination of ammonium at high concentrations. The quality checks referred to above are intended to remedy this situation.

4.2 CALCULATIONS

Determine the concentration of ammonium in leaching, taking into account any dilutions.

The result is expressed as nitrogen concentration C_N (kg N/1000 kg) in fresh material using the following formula:

$$C_N = \frac{C_1 \times V_{EXT}}{M} \times D$$

with:
C_N: concentration of ammonium in the original sample in kg N/1000 kg VM; C_1:
concentration of ammonium in the extract after blank correction in mg N/l;
m: mass of sample extracted in grams; V_{ext}: total
volume of extract in l;
D: drying factor.

Round the result to 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

5 REFERENCES

a. ISO 5664: 1984 water quality – Determination of ammonium – Distillation and titration method
b. ISO 7150-1: 1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method
c. NBN EN ISO 11732: 2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection
d. NBN EN ISO 14911: 1999 Water quality – Determination of dissolved Li+, Na+, NH_4^+, K+, Mn 2 +, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (ISO 14911: 1998)
e. 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)
f. NEN 6604: 2007 Water – Determination of ammonium, nitrate, nitrite, chloride, orthophosphate, sulphate and silicate content using a discrete analytical system and spectrophotometric detection
g. C. Vanhoof, A. Cluyts, E. Poelmans, W. Wouters and K. Tirez, *Evaluation of discrete analyser for the determination of nitrate and ammonium in soil and manure*, VITO report 2012/MANT/R/04, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/2011_rapport_discrete_analyser_VLM.pdf

Solid manure and solid treated manure – Total nitrogen

CONTENT

1 Principle ____ 3

2 Reporting ____ 3

3 References ____ 4

1 PRINCIPLE

Sample preparation shall be carried out as described in BAM/Part 4/02.

The determination of the total N content in solid manure and in the thick fraction obtained after separation of liquid manure is carried out on the sample, dried after the addition of tartaric acid and ground to < 0,5 or 1 mm, using the following methods:

a. NBN AND 13654-2: 2001 soil improvers and growing media – Determination of nitrogen – Part 2: Dumas method;
b. NBN EN 16168: 2012 Sludge, treated biowaste and soil – Determination of total nitrogen using dry combustion method;
c. NBN EN 13654-1: 2001 Soil improvers and growing media – Determination of nitrogen – Part 1: Modified Kjeldahl method;
d. sum of Kjeldahl-N, nitrate and nitrite nitrogen.

The Kjeldahl-N method is described in:

a. NBN EN 16169: 2012 Sludge, treated biowaste and soil – Determination of Kjeldahl nitrogen;
b. NEN 7437: 1998 Animal manure and manure products – Determination of total nitrogen content.

It is assumed that solid manure does not contain nitrate or nitrite. This also applies to the thick fraction obtained after separation of liquid manure. The determination of total nitrogen in solid livestock manure or the thick fraction obtained after separation of liquid manure may be limited to the determination of Kjeldahl nitrogen. The determination of Kjeldahl nitrogen includes a rendering with H2SO4 and a catalyst mixture that converts organic nitrogen compounds into ammonium. After rendering, ammonia is exempted by adding sodium hydroxide and distilling in a suitable absorbent. Ammonium is then determined in that distillate.
Alternatively, for Kjeldahl nitrogen determination, the sample may be solubilised with sulphuric acid, hydrogen peroxide and copper sulphate according to NEN 7433, followed by titrimetric or spectrophotometric determination of ammonium content. On the same solubilising solution, it is possible to determine total P spectrophotometrically or with ICP-AES.

If the analysis is carried out on solid treated manure, except for the thick fraction obtained after separation of liquid manure, it should not be assumed that those products do not contain nitrate or nitrite. In that case, for the determination of total nitrogen, the Compendium for sampling and analysis pursuant to the Materials Decree and the Soil Decree, and more specifically CMA/2/IV/4 *Total Nitrogen from Fertiliser Soil Improvement Substance (https://emis.vito.be/nl/referentielabo-ovam)*, *must* be followed.

2 REPORTING

The result is expressed as nitrogen concentration C_N (kg N/1000 kg) in fresh material. Around the arrive at 2 decimal places for values ≤ 1 and 1 decimal place for values > 1.

The calculation of the total N content in fresh material shall take into account the drying factor determined according to BAM/deel4/02.

3 REFERENCES

a. NEN 7437: 1998 Animal manure and manure products – Determination of total nitrogen content
b. NBN EN 13654-2: 2001 Soil improvers and growing media – Determination of nitrogen – Part 2: Dumas method
c. NBN EN 16168: 2012 Sludge, treated biowaste and soil – Determination of total nitrogen using dry combustion method
d. NBN EN 13654-1: 2001 Soil improvers and growing media – Determination of nitrogen – Part 1: Modified Kjeldahl method
e. NBN EN 16169: 2012 Sludge, treated biowaste and soil – Determination of Kjeldahl nitrogen
f. NBN EN 13342: 2000 Characterisation of sludges – Determination of Kjeldahl nitrogen
g. ISO 13878: 1998 Soil quality – Determination of total nitrogen content by dry combustion (elemental analysis)
h. ISO 11261: 1995 Soil quality – Determination of total nitrogen – Modified Kjeldahl method
i. NBN AND 25663: 1994 water Quality – Determination of Kjeldahl nitrogen – Method after mineralisation with selenium (ISO 5663: 1984)

Solid livestock manure – Reporting

1 GENERAL

The reporting shall be carried out in accordance with BAM/part 8/20. The sampling report drawn up on the basis of the field records (sampling form) shall be added to the analysis report or incorporated into the analysis report.

Without prejudice to the provisions of BAM/Part 8/20, the analytical report shall include the following information:

a. laboratory letterhead paper with at least name, address, telephone, e-mail;
b. unique report number;
c. unique sample number and, if applicable, sample number assigned by the manure bank via SMIL [1;]
d. date and time of sampling;
e. Identification of the sampler (e.g. initials, identification code, SMIL steel nemer number). If the sample has not been taken by a sampler attached to the laboratory, this should be explicitly mentioned in the analytical report;
f. client present at sampling (Y/N);
g. type of manure or treated manure (e.g. pig manure, broiler manure; cattle manure, (solid fraction) digestate, compost...). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. The same description as the one used on the MAD, if applicable, shall be used;
h. description of the sampling location (e.g. blade, shed, container, battery housing, housing housing with/without bedding...);
i. GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the sampled house or storage;
j. date on which the sample was received by the laboratory;
k. the date on which the sample was taken for analysis;
l. date on which the report was sent;
m. name and signature of the person in charge of the laboratory (possibly digitally);
n. name and address of the person to whom the report is delivered.

2 PARAMETER AND UNITS

Dry matter	kg/1000 kg VM
Ammonium	kg N/1000 kg VM
Total nitrogen	kg N/1000 kg VM
Total phosphorus	kg P_2O_5/1000 kg VM

Reported values shall be rounded to 2 decimal places for values ≤ 1, 1 decimal place for values > 1 and to one integer for values > 100.

[1] sampling Noding Internet Loket (https://www.vlm.be/nl/doelgroepen/laboratoria-en-staalnemers/SMIL)

3 NAME OF WEBSITE NOTIFICATION (SMIL)

Data on sampling, analysis results and GPS data logos are reported to the Flemish Land Agency via the SMIL application in accordance with the provisions in BAM/part 8/03

Processed manure – Scope

1 SCOPE

Both the approval of biogas and composting plants and the placing on the market of processed manure and processed manure products require, inter alia, microbiological analyses to demonstrate compliance with the requirements set out in Regulation (EC) No 1069/2009 of the European Parliament and of the Council of 21 October 2009 laying down health rules as regards animal by-products and derived products not intended for human consumption and repealing Regulation (EC) No 1774/2002 (Animal by-products Regulation) and in Regulation (EU) No 142/2011 implementing the Animal by-products Regulation, and subsequent amendments.

The Regulation provides that digestion residues and compost must meet certain standards (Annex V, Chapter III, Section 3, point 1):

> Representative samples of the digestion residues or compost taken during or immediately after processing in the biogas or composting plant to monitor the process must comply with the following standards:
>
> *Escherichia coli*: N = 5, c = 1, m = 1000, M = 5000 in 1 g;
> or
> *Enterococcaceae*: N = 5, c = 1, m = 1000, M = 5000 in
>
> 1 g; and
>
> representative samples of the digestion residues or compost taken during storage or at the time of removal from the facilities concerned must meet the following standards:
>
> *Salmonella*: absence in 25 g: N = 5, c = 0, m = 0, M
>
> = 0 where:
> N: number of samples to be tested;
> m: threshold value for the number of bacteria; the result is considered satisfactory if, in any case, the number of bacteria does not exceed m;
> M: maximum value for the number of bacteria; the result is considered unsatisfactory if the number of bacteria in one or more samples is M or more;
> c: number of samples the bacterial count of which may be between m and M, the sample still being considered acceptable if the bacterial count of the other samples is m or less.

The Regulation provides that processed manure and derived products from processed manure must meet certain marketing standards (Annex XI, Chapter I, Section 2, points (b) and (d)):

1. representative samples of the manure taken during or immediately after processing at the plant in order to monitor the process must comply with the following standards:

Escherichia coli: N = 5, c = 5, m = 0, M = 1000 in 1 g;
or
Enterococcaceae: N = 5, c = 5, m = 0, M = 1000 in 1 g;

and

representative samples of the manure taken during or on withdrawal from storage at the plant of production or the biogas or composting plant must comply with the following standards:

Salmonella: absence in 25 g: N = 5, c = 0, m = 0, M

= 0 where:
N: number of samples to be tested;
m: maximum value for the number of bacteria; the result is considered unsatisfactory if the number of bacteria in one or more samples is M or more;
c: number of samples the bacterial count of which may be between m and M, the sample still being considered acceptable if the bacterial count of the other samples is m or less.

2. the approval of new biogas and composting plants shall demonstrate that the processed manure and derived products from processed manure have undergone a treatment that suppresses spore-forming agents and toxin formation. This was translated into the following standard:

   Clostridium perfringens: maximum 1000 CFU (colony-forming units) in 1 g.

The Executive Laboratory must ensure that sampling and analysis is always carried out in accordance with the methodology described in BAM and is responsible for this.

Processed manure – Sampling

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1 SAMPLING TERMS AND DEFINITIONS

a. *spot sample:* a single sample taken from the lot in one operation and at one location;
b. *sampling point:* place in the lot from which a spot sample is taken;
c. *sample:* a portion of material selected from a larger quantity of material;
d. *laboratory sample:* a sample intended for laboratory inspection or test;
Note: the laboratory sample is the final sample from the point of view of sampling, but is the initial sample from the point of view of the laboratory;
e. *party:* a defined quantity of material produced under uniform conditions.

2 SAMPLING GUIDELINES

2.1 LOCALISATION

As the Regulation distinguishes between sampling 'during or immediately after processing to monitor the process' (for *E. coli* or *Enterococcaceae* and for *Clostridium perfringens* [1]) and 'during storage or at the exit of the products from the holding' (for *Salmonella* spp.), it is important that the identification of the lot determines whether it is a lot 'storage' or a lot 'immediately after processing'. This is best sought from the producer. [...]

2.2 LOT AND LOT DEMARCATION

2.2.1 SOLID PROCESSED MANURE PRODUCTS

In the context of analyses for processed manure, sampling will often be carried out from stockpiles containing stored manure products. Stockpiles are referred to as 'static lots'. It also includes stored material in bunkers, containers, sheds, cargo units and sealants.

The batch shall be unequivocally described by, inter alia, the dimensions of the batch and identification of the nature of the material. Dimensions are defined by ground area and height. The batch can also be described on the basis of typical characteristics, such as grain or piece size, colour, determination 'during storage' or 'immediately after processing'...

If several lots are found at one location, a distinction should be made between the different lots: delineate the parties. Thus, in order to avoid the phenomenon of 'dilution' of certain properties when sampling multiple (small) lots, individual lots are not considered as a single lot for microbiological characterisation. As a rule of thumb, each demarcated lot is sampled separately. Each storage unit is thus considered as a separate lot. This means that each pile, container, lorry, silo, loading unit... is in principle sampled separately, even if they

[1] the *Clostridium perfringens* parameter is determined only when new biogas and composting plants are approved. The parameter is therefore not applicable in the quarterly analyses.

contain a similar load. If, within a single storage unit, it is still possible to distinguish between different types of manure products, either visually or on the basis of origin, origin or type of manure, the lots shall be sampled separately.

Taking into account the practical feasibility of sampling, the maximum batch size shall be 1 000 m^3. Batches larger than 1 000 m^3 are sampled with a higher frequency on an annual basis by participation of the Flemish Land Agency.

It is certainly useful to record the batch or situation photographically, possibly with a recognisable object to represent the location or dimensions (see section 5).

2.2.2 LIQUID PROCESSED MANURE PRODUCTS

Liquid processed manure may also be present in the processing process. Immediately after processing, a liquid manure can be pumped, where a tap is available in the piping circuit for sampling. In the case of storage units in a silo or bunker, a tap should be provided for sampling. If this is not the case, no sampling can be carried out at that location and this will be described in the sampling form and
— report.

2.3 MONSTER

The purpose of sampling, as described in this procedure, is to take samples representative of the entire stock of manure or manure products.

For the determination of *Escherichia coli* or *Enterococcaceae* and for the determination of *Salmonella spp*. the Regulation requires the testing of 5 representative samples each. This means that, for the determination of those parameters, 5 independent single point samples are taken separately from each other (no composite sample (s)) (see also paragraph 2.5.1.). Such a point sample is taken in one operation at one defined location in the lot (sampling point) or time in the material flow.

For the determination of *Clostridium perfringens*, one composite sample is formed per sampling, composed of the 5 point samples taken (to be carried out by the analytical laboratory after delivery of the samples).

2.4 SAMPLE QUANTITY

2.4.1 SOLID PROCESSED MANURE PRODUCTS

The sample size of a laboratory sample depends on the grain size in which the processed manure products are presented, in order to ensure the representativeness of the sample in relation to the original lot:

a. grain size < 10 mm: 0.2 litres per point sample;
b. grain size 10-40 mm: 0.6 litres per point sample;
c. grain size > 40 mm: 1 litres per spot sample.

2.4.2 LIQUID PROCESSED MANURE PRODUCTS

The sample quantity of a laboratory sample is 200 ml of liquid fertiliser product.

2.5 Number, location and quantity of point samples

2.5.1 Number of point samples

Given the standards set out in BAM/Part 7/00 Scope for the five samples for *E. coli* or *Enterococcaceae* indicates a threshold (m) and a maximum value (M) for the number of bacteria, this does not result in the formation of point samples. The composition of point samples may result in a dilution of the number of bacteria present, resulting in a point sample that originally exceeds the standards being determined by dilution below the standards. Thus, by default, only 5 point samples are taken for one lot per sampling. A point sample is the amount of (manure) product that can be taken in a single operation at a given location (sampling point) (e.g. for solid product one shank, drilling pitch, drilling; for liquid product, filling a container to a tap).

2.5.2 Point sample size specific to solid processed manure

In order to give each individual particle of material in the lot the same chance to be sampled, the size of a point sample shall be adjusted to the particle size of the manure product to be sampled. The grover the material, the larger the spot sample is taken.
This also means that the sampling equipment used must be adapted to the grain size of the material to be sampled. By appointment, the opening of the drill or shank is taken, if possible, about 2-3 times larger than the largest material grain.

If the grain size < 10 mm is a suitable tool in addition to a shovel, a jaw/gut/equivalent boron is also a suitable tool. Materials with a larger particle size are sampled using a shovel. Care must be taken to ensure that the opening of the shells is large enough for the grain size of the manure product to be sampled. The shovel should preferably have upright edges so that the material cannot fall back during creation. Conversely, the excess material above the edges of the boron or shank is removed (e.g. with a spatula) as it does not belong to the point sample.

2.5.3 Place point samples

For the determination of *Escherichia coli* or *Enterococcaceae* (immediately after processing) and *Salmonella* spp. (storage), the batch for solid processed manure is pre-divided into 5 segments. One spot sample shall be taken from each segment. For a liquid manure product, locate in the production process the sampling point (tap) with the manure flow immediately after the exit of the heat treatment or from a storage unit. Storage units for liquid processed manure products are usually tanks or bunkers fitted with a crane where sampling can be carried out. The diameter of the supply to the tap and of the tap itself will influence the flow rate. At high flow rates, it may be appropriate to use a bucket to avoid liquid manure spillage.
Sampling solid manure products in enclosed or semi-enclosed storage units such as trucks, containers, bunkers and storage sheds adds to the difficulty of accessibility/accessibility and homogeneous distribution of point samples.
Stockpiles are (usually) accessible along the entire perimeter; trucks, containers are accessible only on one side (often the top). The point samples can therefore only be taken from the accessible side, which naturally influences the representativeness of the sample. Where the stockpile is sampled horizontally, a container or truck must be sampled vertically, which further increases the difficulty of sampling. In such cases, it may be possible to switch to others,

more specialised sampling equipment (e.g. soil boron for non-cohesive material).

The sampling must always be fully described and documented (see paragraph 5), especially if the sampling involves accessibility restrictions (for example, if only one or 2 sides of the pile could be sampled).

3 SUPPLIES

Equipment and sample containers must be clean and (demonstrably) sterile (sterile purchased or sterilised by wet or dry sterilisation, or cleaned and disinfected with a disinfectant just before sampling).

a. clean shovel or shovel, preferably with upright edges (for example, with an opening of at least 12 cm): see Figure 1
b. pitch or gut boron (for sampling material of grain size < 10 mm): with drill body slide 30 mm and effective length 30 to 60 cm (minimum capacity 200 ml). With spatula, if necessary, to remove the contents from the boron: see Figure 2. Other types of equivalent drills are permitted and can only be used provided that they can be properly disinfected.
c. hand handles, with raised edges, known volume (200 ml/600 ml/1000 ml depending on the maximum grain in the batch (D95)) and opening at least 3 times greater than D95: see Figure 3
d. personal protective equipment, depending on the sampling conditions: strong gloves (or 2 pairs of disposable gloves over each other), boots, if required safety shoes and definitely recommended: face mask fitted with green (K) filter (ammonia protection)
e. sealable sampling bags (plastic) (minimum capacity of 200 ml) or sample containers with lid (with capacity of 200 ml for liquid material and between 200 ml and 5 litre, depending on the grain size of solid manure (product) to be sampled)
f. bucket
g. ethanol, moist ethanol cloths or equivalent
h. adsorbent paper
i. thick pin or labels (pre-printed) identifying the sampling bags – or containers
j. sampling forms for reporting the data of the sample
k. ice or cold storage box
l. if available: a wheel charger/shovel with shovel and driver

Figure 1: sampling trowel handshaft

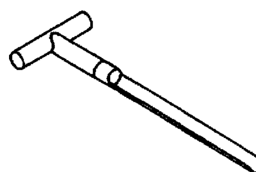

Figure 2: gutsboron

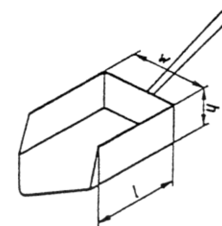

Figure 3: upright

4 SAMPLING PERFORMANCE

4.1 General

For the sampling of processed manure products, 5 samples shall be taken for the determination of *Escherichia coli* or *Enterococcaceae* and 5 samples shall be taken for the determination of *Salmonella* spp. and one composite sample for the determination of *Clostridium perfringens* (only in the case of the approval of new biogas and composting plants), composed of part of each of the 5 samples taken immediately after processing.
All preparations and operations must be carried out using aseptic techniques and sterile material to avoid microbiological contamination from external sources of the samples. Equipment and containers must be clean and sterile. A refrigerated box is needed for transport.

4.2 Solid processed manure products

Sampling may be carried out manually, using a pitch or gut boron or equivalent, or shovel/shovel (see procedure point 4.2.1), or in combination with any available means of transport such as wheel loaders or shovels (see procedure point 4.2.2). The advantage of the combined method is that a wheel charger allows sampling to be carried out to a greater extent in the hope, whereas the manual method limits sampling to the outer layer of the lot. In particular for larger lots (> 50 m^3), the combined method (with wheel charger) provides a higher degree of representativeness of the sample compared to the lot. If such means of transport are available, the combined method (with wheel charger) is preferable to the manual sampling only.

4.2.1 Manual method of sampling by stabling or gut boron or shank or by hand

The manual sampling method is applicable for sampling batches up to 1 000 m^3.

a. Calculate the volume of the lot to be sampled by estimating the area of soil and the mean altitude. Adapted to the size of the total production/storage, the minimum frequency of 4 samples is increased on an annual basis (to be determined by the Flemish Land Agency).
b. Take a point sample with a pitch or gut boron or equivalent boron (grain size < 10 mm) or with a shovel/shovel with adapted opening (grain size > 10 mm). Push the shovel/shovel or boron as inclined as possible in the material. Ensure that the shank or boron is fully filled, and that all point samples are of approximately the same size.
c. Remove the stuffed shovel or boron from the material. Remove the excess material that is on top of the boron or shank (which is not part of the point sample). Collect the point sample in the sample sampler inoculum. If a pitch or gut boron or equivalent is used: take a spatula to scrape the contents from top to bottom of the drill (the edges of such drill are sharp!).
d. Sampling may also be carried out directly with a sterile sampling container (sampling bag). The sterile bag can be moved by hand with the outside as a kind of glove. In this way, a spot sample can be taken directly into the bag.
e. Try to take a spot sample at different depths: take, for example, two point samples at the surface, and the other three point samples at a minimum depth of 30 cm in the hope. For the latter, remove the upper layer of the pile (approximately 30 cm) by shovel at the selected sampling point, so that the deeper material can be reached.

Logically, the intrusion depth of a pitch or guts boron or equivalent boron (depending on the type and length used) is greater than the intrusion depth when using a shovel/shovel.

f. Repeat the operation at the different sampling points so that the pile of manure or (processed) manure products is sampled uniformly.
g. The different sampling points shall be evenly spaced over the perimeter of the lot. By appointment, point samples shall be taken at a height of between 0 and 150 cm in relation to the ground. The spatial distribution of point samples shall be homogeneous both horizontally and vertically. Avoid unnecessary risks by walking up or hoping to walk up for unreachable or poorly reachable sampling points.
h. The 5 point samples are contained in 5 separate containers and are identified.

4.2.2 METHOD OF SAMPLING BY WHEEL CHARGER/SHOVEL/BULLDOZER

This combined sampling method is applicable for batches up to 1 000 m³. For parties
> 50 m³ is the most appropriate method.

a. Calculate the volume of the lot to be sampled by estimating the area of soil and the mean altitude. Adapted to the size of the total production/storage, the minimum frequency of 4 samples is increased on an annual basis (to be determined by the Flemish Land Agency).
b. Take a load or shovel with the wheel charger at 5 different representative places in the batch. In order to arrive at the bulk of a large batch, the wheel charger first removes some cargo material from the batch. The samples do not include the loads removed; only the following shovel from the bulk of the material is charged for the sampling.
c. Where possible, the locations where the wheel charger is inserted shall be spatially distributed over the batch (for example, on both sides of the batch).
d. The shovel loads are each tipped separately next to the batch on clean, inert substrate.
e. Using a shovel/shovel, take one point sample (see paragraph 2.3.1) from this sublot.
f. Make sure the shank is fully filled. Remove any surplus material on top of the shovel/shovel (this is not part of the point sample).
g. Repeat the operation on the other sublots so that the pile of manure products is sampled uniformly.
h. The 5 point samples are contained in 5 separate containers and are identified.

4.3 LIQUID PROCESSED MANURE PRODUCTS

4.3.1 IMMEDIATELY AFTER THE HEAT TREATMENT

a. In the production process, locate the sampling point (tap) with the manure flow immediately after the exit of the heat treatment.
b. Uniquely identify and record the situation to be sampled (pipe circuit, storage unit,...) and the exact time of sampling.
c. In the case of external storage, the tap shall be flammed immediately before sampling, provided that this operation has been authorised by the operator or the person responsible for production. Otherwise, as always in internal storage, the tap is cleaned and disinfected with ethanol (wipes).
d. Open the tap and collect a quantity of processed manure product in a bucket (‘rinse’); this quantity is not part of the sampling.

e. Then collect a point sample of at least 200 ml directly into a sterile sample sampler.
f. Close the tap and close the container.
g. After rinsing, point (d) to (f) shall be repeated for the remaining 4 point samples. For each point sample, use a new sterile sample container
h. Carry out rinsing volumes appropriately.

4.3.2 FROM A STORAGE UNIT

a. If the liquid processed manure to be sampled is from a storage unit, uniquely identify the storage via a feature (e.g. silage number). Note also the contents and, if relevant (e.g. silo), the filling rate of the batch, expressed in volume (m^3).
b. In the case of external storage, the tap shall be flammed immediately before sampling, provided that this operation has been authorised by the operator or the person responsible for production. Otherwise, as always in internal storage, the tap is cleaned and disinfected with ethanol (wipes).
c. Open the tap and collect a quantity of manure in a bucket ('rinse'); this quantity is not part of the sampling.
d. Then collect a point sample of at least 200 ml directly into a sterile sample sampler. Perform this operation above the bucket to prevent spills.
e. Close the tap and close the container.
f. After rinsing, repeat point (c) to (e) for the remaining 4 point samples. For each point sample, use a new sterile sample container.
g. Carry out rinsing volumes appropriately.

5 IDENTIFICATION OF SAMPLES

The tag (number, barcode...) of the sample must be unambiguous so that no misunderstandings can subsequently arise as to the origin of the samples.

Sampling data shall be reported in accordance with BAM/part 8/20. Without prejudice to the provisions of BAM/part 8/20, the following information shall be indicated on the (digital) sampling form accompanying the sample:
a. client, address, operator number;
b. client and/or third parties present during sampling;
c. reference of MAD/Neighbouring scheme BR manure disposal document in case of cargo sampling;
d. type of processed product (e.g. digestate, compost...) and, if possible, the type of manure from which the processed product is derived (e.g. dried thick fraction – pig manure). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. The same description shall be used as the one used on the MAD, if applicable;
e. the GPS coordinates in WGS84 format, in decimal degrees to 5 decimal places, of the sampled lot. Those coordinates shall be determined locally by a GPS device;
f. Identification of the sampler (e.g. initials, identification code, SMIL steel sampler number);
g. date and time of sampling;

h. description (container, composting cell, shed...) and dimensions of the sampled lot, location plans, sketches or photographs are always useful, and even mandatory in the case of mixed lots or lots divided into sublots (> 1 000 m^3 or poorly accessible);
i. always indicate whether the sample was taken from a lot ‘during storage’ or ‘immediately after processing’;
j. the number and volumes of containers filled and the information necessary to identify the samples as indicated on the container;
k. own sample number or sample coding;
l. significant remarks and/or deviations that may affect the interpretation of the analytical result.

The laboratory’s sample management system shall allow for the unambiguous retrospective tracing of any information relating to an individual sample.

6 MONSTERCONSERVATING AND TRANSPORT

The maximum storage period of 24 hours of samples intended for bacteriological analysis shall apply from the time (date/hour) of sampling. The samples should therefore be delivered to the analytical laboratory in time to ensure that shelf life is respected. Samples should therefore preferably be sent immediately and chilled to the analytical laboratory.

Samples must be stored at (3 ± 2) °C on arrival at the analytical laboratory.

Processed manure – Sample pre-treatment

1 PRINCIPLE

After opening the package, the sub-samples are weighed directly in sterile stomacher bags aseptically; it is not homogenised. The test portion to be weighed is poured out of the inner part of the pot/bag. The creation of the outer layer of the material from the pot/bag is avoided.

For microbiological analysis, the following quantity shall be weighed:

a. for *E.coli/Enterococci* 5 times 10 g material;

b. for *Salmonella* 5 times 25 g material;

c. for *Clostridium perfringens* 1 times 10 g material.

The analyses shall be carried out as far as possible without interruption, otherwise the samples shall always be stored in the refrigerator.

From the processed manure to be analysed, an initial suspension in buffered peptone water (see BAM/part 7/05 paragraph 1.2.1, BAM/deel7/06 paragraph 3.1) is made to obtain the most uniform distribution of microorganisms from the sample.

For the *Salmonella* analysis, this step serves as a precursor.

For the determination of *Escherichia coli*, *Enterococcaceae* and *Clostridium perfringens*, 1 ml of the initial suspension, representative of 0.1 g sample, should be tested. This is then accounted for to 1 g of processed manure.

a. Standard procedure:
   A suspension of samples taken for the placing on the market of processed manure and processed manure products does not require decimal dilutions to be carried out. If too high numbers of CFU per plate are reached with the initial suspension, a sample does not meet the requirements described under BAM/deel07/00.
b. Exceptional procedure:
   A suspension of samples of the digestion residues or compost taken during or immediately after processing in the biogas or compost production plant to monitor the process should not be analysed as an initial suspension but only as a decimal dilution. If too high numbers of CFU per plate are reached with the first decimal dilution of the suspension, a sample does not meet the requirements described under BAM/deel07/00.

2 REFERENCES

a. ISO 6887-1: 2017: Microbiology of the food chain – Preparation of test samples, initial suspension and decimal dilutions for microbiological examination – Part 1: General rules for the preparation of the initial suspension and decimal dilutions

b. ISO 7218: 2007 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations

c. ISO 7218: 2007/Amd 1: 2013: Microbiology of food and animal feeding stuffs – General

requirements and guidance for microbiological examinations.

Processed manure – Detection of Escherichia coli

1 PRINCIPLE

The detection of *Escherichia coli* shall be carried out in accordance with ISO 16649-2 Microbiology of food and animal feeding stuffs – Horizontal method for the enumeration of *beta-glucuronidase-positive Escherichia coli* – Part 2: Colony-count technique at 44 °C using 5-bromo-4-chloro-3-indolyl β-D-glucuronide.

The number of *Escherichia coli* is expressed as the number of CFU in 1 g of sample. The report shall also indicate or refer to the method used.

Depending on the origin and design of the sampling and analysis (see BAM/part 7/00 Scope):

a. standard procedure for the Flemish Land Agency '**Marketing of processed manure**': when samples analysed for the placing on the market of processed manure and processed manure products, only the initial suspension shall be used;
b. specific procedure "**biogas/compost recognition**" (in case of exceptional question): analysis of samples for approval of biogas and composting plants: only a dilution of 1/10 of the initial suspension is used.

To 10 g of weighed homogeneous sample in a sterile plastic bag add 90 ml of buffered peptone water (at room temperature) aseptically (i.e. ultimately a 1/10 mass/volume ratio). Homogenisation in the homogeniser for 2 minutes.
For the standard procedure: for analysis, immediately after homogenisation, 10 ml of the initial suspension (representative of 1 g of treated manure) is transferred into 10 empty and sterile petri dishes, followed by pour plate with the chromogenic medium.
For the specific procedure: for analysis, after homogenisation, a 1/10 diluted suspension of the sample extract is prepared and 10 ml of it is transferred into 10 empty and sterile petri dishes, followed by casting plate with the chromogenic medium.

2 REFERENCE

a. ISO 16649-2: 2001 Microbiology of food and animal feeding stuffs – Horizontal method for the enumeration of beta-glucuronidase-positive Escherichia coli – Part 2: Colony-count technique at 44 degrees C using 5-bromo-4-chloro-3-indolyl beta-D-glucuronide
b. ISO 7218: 2007 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations
c. ISO 7218: 2007/AMD 1: 2013 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations – Amendment 1
d. ISO 6887-2: 2017 17 microbiology of the food chain – Preparation of test samples, initial suspension and decimal dilutions for microbiological examination – Part 2: Specific rules for the preparation of meat and meat products.

Processed manure – Enterococcaceae detection

1 PRINCIPLE

For the detection of *Enterococcaceae*, only the *genus* Enterococci is determined in a fixed matrix, referring to the media described in ISO 7899-2 for water analyses: Water quality-Detection and enumeration of intestinal enterococci part 2: Membrane filtration method, or to use *Enterococcus* chromogenic media as an alternative.

The preliminary analytical steps on a fixed matrix follow ISO 6887-2 Microbiology of the food chain – Preparation of test samples, initial suspension and decimal dilutions for microbiological examination Part 2 Specific Rules for the preparation of meat and meat products.

An equivalent chromogenic medium may also be used for the analytical method.

The number of *Enterococcaceae* is expressed as the number of CFU in 1 g of sample. The report shall also indicate or refer to the method used.

Depending on the origin and design of the sampling and analysis (see BAM/part 7/00 Scope):

a. standard procedure for the Flemish Land Agency '**Marketing of processed manure**': when samples analysed for the placing on the market of processed manure and processed manure products, only the initial suspension shall be used.
b. specific procedure "**biogas/compost recognition**" (in case of exceptional question): analysis of samples for approval of biogas and composting plants: only a dilution of 1/10 of the initial suspension is used.

To 10 g of weighed homogeneous sample in a sterile plastic bag add 90 ml of buffered peptone water (at room temperature) aseptically (i.e. ultimately a 1/10 mass/volume ratio). Homogenisation in the homogeniser for 2 minutes.
For the standard procedure: for analysis, immediately after homogenisation, 10 ml of the initial suspension (representative of 1 g of treated manure) is transferred into 10 empty and sterile petri dishes, followed by pour plate with the chromogenic medium.
For the specific procedure: for analysis, after homogenisation, a 1/10 diluted suspension of the sample extract is prepared and 10 ml of it is transferred into 10 empty and sterile petri dishes, followed by casting plate with the chromogenic medium.

2 REFERENCES

a. ISO 7899-2: 2000 Water quality – Detection and enumeration of intestinal *enterococci* – Part 2: Membrane filtration method
b. ISO 7218: 2007 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations
c. ISO 7218: 2007/AMD 1: 2013 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations – Amendment 1
d. ISO 6887-2: 20 17 microbiology of the food chain – Preparation of test samples, initial suspension and decimal dilutions for microbiological examination – Part 2: Specific rules for the preparation of meat and meat products

Processed manure – Detection of Salmonella spp.

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1 Method ISO 6579-1: 2017 & ISO 6579-1: 2017/AMD1: 2020

The detection of *Salmonella* spp. according to ISO 6579-1: 2017 and ISO 6579-1: 2017/AMD1: 2020 shall comprise the following successive stages:

a. pre-ripening in a non-selective liquid medium;
b. collapse of *Salmonella* in two selective media after the pre-collapse;
c. plating and identification;
d. biochemical and serological confirmatory assays.

1.1 Media and material

a. buffered peptone water BPW;
b. Rappaport-Vassiliadis medium with soya RVS/modified Semi-Solid Rappaport-Vassiliadis MSRV;
c. Muller-Kauffmann tetrathionate novobiocin bouillon MKTTn;
d. xylose lysine deoxycholate agar XLD (preferably in additional large trays 140 mm);
e. second agar medium at the discretion of the laboratory (see Annex E of ISO 6579-1: 2017). In order to select as completely as possible all strains of the genus *Salmonella* spp., at least two selective "*Salmonella* media" should be used. XLD is laid down in the standard and a second medium should be chosen depending on the possible growth from the spectrum of *Salmonella*. A combination of the media can be determined from the specifications in the media manuals of the different brands;
f. non-selective agar (e.g. Nutrient agar);
g. triple sugar/iron agar TSI;
h. urea agar Christensen;
i. L-lysine decarboxylation medium;
j. reagent for detection of -galactosidase (optional);
k. reagent for indole reaction (optional);
l. physiological saline (0.85 % NaCl);
m. monovalent or polyvalent anti H, o, (Vi) sera for *Salmonella* or *Salmonella* latex agglutination Test or equivalent test;
n. autoclave 121 ± 3 °C; incubators of 36 ± 2 °C and [...] 41,5 ± 1 °C, water bath at 45 ± 1 °C, suitable glassware, pipettes, pH meter, petri dishes, inoculum;
o. Stomacher or equivalent homogeniser.

1.2 Procedure

1.2.1 Initial suspension of sample and pre-ripening in a non-selective liquid medium (buffered peptoon water)

Note: initial suspension of the sub-samples in buffered peptoonwater for analysis of *Enterococcaceae* or *E.coli* and *Salmonella* spp. may also be carried out together on 25 g.

a. add 25 ml buffered peptone water aseptically to 225 g homogeneously weighed sample in a stomacher bag (1/10 weight/volume ratio);
b. if the samples contain fragments that may damage the stomacher bag, the whole shall be wrapped in an additional (stomacher) bag, prior to the homogenisation process;
c. homogenisation in the homogeniser for 2 minutes;
d. the bag is closed by clips or tapes;

e. the stomacher bag is incubated between 34 °C and 38 °C for 18 ± 2 h.

1.2.2 SELECTION AND VALIDATION OF THE METHOD OF COLLISION IN TWO SELECTIVE MEDIA

ISO 6579-1: 2017 & AMD1: 2020 specifies a specific method for detection of *Salmonella* spp. It shall apply to:

a. products intended for human consumption and animal feed;
b. environmental samples in food production and processing;
c. samples from the primary production phase such as animal faeces, dust and swabs.

With this method, most Salmonella serovars are intended to be detected. The detection of some specific serovars may require additional breeding steps. For *Salmonella Typhi* and *Salmonella Paratyphi*, the procedure is described in ISO 6579-1: 2017 Annex D.

The selective release medium MSRV shall be used for this purpose. This is intended for the detection of movable *Salmonella* spp. (and is therefore not suitable for the detection of non-movable *Salmonella* spp.).

Positive MSRV plates show a grey-white cloudy area extending from the inoculated drop. The cloudy area is characterised by a white halo with a clearly defined border.

In order to detect *Salmonella* from processed manure, the most appropriate application method must be validated on the matrix to be analysed.

1.2.3 COLLAPSE OF SALMONELLA IN TWO SELECTIVE MEDIA RVS AND MKTTN

Bring the RVS and MKTTn broths to room temperature.
From the stomacher bag, the buffered peptone is suspended:

a. Transfer 0.1 ml into 10 ml RVS broth: incubation at 41.5 °C ± 1 °C (care not to exceed 42.5 °C) for 24 ± 3 h;
b. Transfer 1 ml into 10 ml MCTC broth: incubation between 34 °C and 38 °C for 24 ± 3 h.

1.2.4 A RECOMMENDATION OF *S ALMONELLA* IN TWO SELECTIVE MEDIA MSRV AND MKTT N

Bring the MSRV plates and MKTTn broth to room temperature.
From the stomacher bag, the buffered peptone is suspended:

a. 0.1 ml pipette applied to the MSRV plates via 3 drops of suspension. Apply the 3 drops containing a total of 0.1 ml equally spaced on the surface of the medium: incubation at 41.5 °C (care should be taken not to exceed 42.5 °C) for 24 ± 3 h without stirring the plates;
b. Transfer 1 ml with pipette to 10 ml of MKTTN broth: incubation between 34 °C and 38 °C for 24 ± 3 h.

Important Note: it is appropriate to incubate the RVS/MSRV and MKTTn continuously for 24 ± 3 h, specifically to detect slowly growing *Salmonella*. If no typical *Salmonella* colonies are observed at point 1.2.5, re-plate on the selective agar media after further incubation.

1.2.5 Plating and identification

If available, additional large scales shall be grafted. Otherwise, two ordinary dishes shall be inoculated successively using the same inoculum needle. Between the grafting of the first and second scales, the needle shall not be flammed.
After incubation at 24 ± 3h:

a. using a platinum needle, inoculate from the liquid culture RVS or Semi-Solid plates MSRV and MKTTn broth one additional large scale or two ordinary petri dishes each of the selective media XLD and the additional medium;
b. incubation of the XLD dishes (top agar bottom) between 34 °C and 38 °C for 24 ± 3 h. The second medium is incubated according to the supplier's instructions. After incubation, the presence of typical and possible *Salmonella* spp. colonies is checked;
c. on XLD:
   1. typical *Salmonella* colonies have a black centre and a slightly transparent reddish colour by changing the colour of the indicator;
   2. H_2S negative variants of *Salmonella* show a pink colour and a dark rose centre;
   3. lactose *positive Salmonella* gives yellow colonies with or without black;
d. on the second medium: check of presumptive *Salmonella* colonies according to the characteristics of the medium used.

1.2.6 Biochemical and serological confirmatory assays

1.2.6.1 Selection of colonies for confirmatory assays

a. For the confirmatory tests, one typical or five suspect colonies are picked up from both selective media and stretched out on a nutrient agar plate in such a way that well-insulated colonies are obtained. Alternatively, if well-isolated colonies (of pure culture) are available on the selective media, biochemical confirmation can be carried out directly on a suspect, well-isolated colony of a selective plate. The culture on the non-selective (nutrient) agar medium can then be carried out in parallel with the biochemical tests to control the purity of the colony taken from the selective agar medium.
b. Incubation of the shells between 34 °C and 38 °C for 24 ± 3 h.
c. Use pure cultures for confirmatory assays.

One isolate shall be tested. If negative, the other four isolates shall be subjected to the confirmatory tests. For epidemiological studies, at least five isolates shall be tested.

1.2.6.2 Biochemical confirmatory assays

A biochemical identification is carried out on a pure colony to be examined using the media and testing:

a. Triple Sugar Iron agar TSI (glucose + acid + gas positive, hydrogen sulphide formation positive 92-97 % *S*. Paratyphi 10 %, lactose negative 99 % *S*. Paratyphi positive and sucrose negative 99 %);
b. Urea hydrolysis (99 % negative);
c. Lysine decarboxylation (95 % positive; *S*. Paratyphi negative; *S*. Typhi 98 % positive);
d. — Galactosidase reaction (negative 98 %) (optional);
e. Indolin production (99 % negative) (optional).

These tests may also be carried out with a commercial biochemical kit and, if necessary, supplemented by the tests mentioned above.

The interpretation of the results of an identification kit shall be carried out in accordance with the relevant manual.

If *Salmonella* spp. strains are identified, a serological confirmation shall be carried out.

1.2.6.3 SEROLOGICAL CONFIRMATION TEST

Elimination of car agglutinating strains: place a drop of saline solution on a substrate and dissolve a del of a suspect colony by means of an inoculum. By rotating motion for 30-60 s, the autoagglutination is checked. If positive, no further serological test shall be performed.

The agglutination test for the detection of the presence of Salmonella O and H antigens (if *Salmonella* Typhi VI antigens) shall be carried out on each pure colony (not auto-agglutination). The test shall be carried out in accordance with the manufacturer's instructions. Agglutination is compared with a positive and negative control. If agglutination occurs, the reaction is reported positive.

1.2.6.4 SPECIES IDENTIFICATION

If there is a need for species identification, an isolate for this purpose is inoculated into transport agar slant; incubation of the lettuce between 34 °C and 38 °C for 24 ± 3 h. The tubes are sent to an accredited institute, where final typing can take place.

1.2.6.5 M ICROBIOLOGICAL IDENTIFICATION OF S ALMONELLA BY MALDI-TOF MS (MATRIX-ASSISTED LASER DESORPTION-IONISATION TIME-OF-FLIGHT MASS SPECTROMETRY)

The MALDI-TOF MS technology can be used to identify *Salmonella*. This requires a validation in accordance with ISO 16140.

2 Procedure VIA PCR assay SALMONELLA spp. or via Vidas SALMONELLA

Vidas *Salmonella* technology or Real-Time PCR Assay for *Salmonella* spp. technology may be used for the determination of *Salmonella*. Both techniques have Afnor validation in accordance with ISO 16140, initially in food matrices, but also in environmental matrices.

3 EXPRESSION OF RESULT

Depending on the results and interpretation, *Salmonella* spp. is expressed as detectable/not detectable in 25 g of sample.

The report also mentions the BAM method used.

4 QUALITY CHECK

Use of a blank control on the selective media for each measurement set.

Use of a positive control per lot of analytical media.

Validation of the analytical method on different matrices (wet and dried manure): test repeatability. For this purpose, a control sample is inoculated with a reference *Salmonella* strain and treated as any other unknown sample.

Inference the correctness from second line check.

5 REFERENCES

a. ISO 6579-1: 2017 Microbiology of the food chain – Horizontal method for the detection, enumeration and serotyping of Salmonella – Part 1: Detection of Salmonella spp.
b. ISO 7218: 2007 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations
c. ISO 7218: 2007/AMD 1: 2013 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations – Amendment 1
d. ISO 6579-1: 2017/Amd 1: 2020 Microbiology of the food chain – horizontal method for the detection, enumeration and serotyping of Salmonella – part 1: detection of Salmonella spp. – Amendment 1: Broader range of incubation temperatures, amendment to the status of annex D, and correction of the composition of MSRV and SC
e. https://nf-validation.afnor.org/en/food-industry/salmonella-spp/

Processed manure – Detection of Clostridium perfringens

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1 PRACTICE

The detection of *Clostridium perfringens* shall be carried out by:

a. isolation and enumeration of characteristic colonies by analysis of the initial suspension with the selective medium SC agar via the pour plate method;
b. confirmation of characteristic colonies;
c. calculate the number of *Clostridium perfringens* per gram of sample from the number of colonies confirmed.

2 MEDIA AND MATERIAL

a. buffered peptone water BPW;
b. (T) SC agar;
c. for the confirmatory test: Fluid Thioglycolate Medium TGM, lactose sulphite medium LS (with Durham tube) or a commercial biochemical kit;
d. autoclave 121 ± 3 °C; incubator 36 ± 2 °C, water bath at 45 ± 1 °C, water bath at 46 ± 0.5 °C suitable glassware, pipettes, pH meter, petri dishes, inoculum, anaerobic jar and reagents;
e. Stomacher or equivalent homogeniser.

3 PROCEDURE

3.1 INITIAL SUSPENSION OF A SAMPLE IN BUFFERED PEPTONE WATER

a. Add 10 ml buffered peptone water aseptically (1/10 weight/volume ratio) to 90 g homogeneously weighed sample in a stomacher bag.
b. If the sample contains fragments that may damage the stomacher bag, the whole shall be wrapped in an additional (stomacher) bag, prior to the homogenisation process.
c. Homogenisation in the homogeniser for 2 minutes.
d. From the buffered peptone suspension 1 m l[1] of the sample extract to be analysed is transferred from the stomacher bag to an empty and sterile Petri dish (1 ml suspension is representative of 0.1 g sample). The time between the application of the suspension in the petri dish and the pouring of SC should be kept as short as possible and should not exceed 15 min.
e. Petri dish to be filled with 15 ml liquid SC agar medium stored at 44-47 °C in a water bath.
f. Mix inoculum and medium with the shell by rotating movements.
g. Allow agar to solidify and then pour with a second layer of liquid medium (10 ml).
h. Allow agar to solidify and dry in laminar flow and incubate inverted anaerobic at 36 °C for 20 ± 2 h.

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[1] the criterion is max. 1000 CFU *Clostridium perfringens* in 1 g of treated manure, which means max. 100 CFU/plate.

3.2 Colony enumeration and selection for confirmation

a. Count the black presumptive *Clostridium perfringens*.
b. Select five characteristic colonies from the biochemical confirmation scale.

3.3 Biochemical confirmatory assay

A commercial biochemical kit may be used for identification. The interpretation of the results of an identification kit shall be carried out in accordance with the relevant manual. Otherwise, the following method shall be used.

3.4 Inoculation and incubation

a. Incubate each of the selected colonies with an inoculum in liquid TGM.
b. Anaerobic incubation at 36 °C for 18-24 h.
c. After incubation, pipette 5 drops of TGM culture into MS medium.
d. Incubation in a water bath at 46 °C for 18-24 h.

3.5 Interpretation

a. Examine each tube LS medium for gas production and for black discoloration of the medium by iron sulphite precipitation. Tubes containing Durham tubes filled with gas in excess of 1/4 and containing a black precipitate are considered positive.
b. In case of doubt, if a Durham tube with less than 1/4 is filled in a black MS tube, immediately pipette 5 drops of LS culture into a new MS tube.
c. Incubation in a water bath at 46 °C for 18-24 h.
d. Examine these tube (s) as mentioned above.

Bacteria that give black colonies on SC medium and give a positive confirmation in LS medium are considered *Clostridium perfringens*. Otherwise, the result is negative.

3.6 Microbiological identification of Clostridium perfringens by MALDI-TOF MS (MATRIX-ASSISTED LASER DESORPTION-IONISATION TIME-OF-FLIGHT MASS SPECTROMETRY)

The MALDI-TOF MS technology can be used to identify *Clostridium perfringens*. This requires a validation in accordance with ISO 16140.

3.7 Calculation of the result (see ISO 7218: AMD. 1)

Calculate the number of *Clostridium perfringens* **a** in 1 grams of sample using the following formula:

$$a = b/A \times C$$

with:
C: the number of *presumptive Clostridium perfringens* colonies;
A: the number of inoculated colonies for confirmation (= 5);
b: the number of confirmed colonies.

4 EXPRESSION OF RESULT

The number of *Clostridium perfringens* is expressed as the number of CFU in 1 g of sample. If more than 100 colonies per plate have been determined as *Clostridium perfringens*, the number shall be reported as
> 1000 CFU *Clostridium perfringens per* g sample. If absent, < 10 CFU/g sample is reported. The report shall also indicate the BMM method used.

5 QUALITY CHECK

Use of a blank control on the selective media for each measurement set.

Use of a positive control per lot of analytical media.

Validation of the analytical method on different matrices (wet and dried manure): test repeatability. For this purpose, a control sample is inoculated with a reference *Clostridium perfringens* strain and treated as any other unknown sample.
Inferring the correctness from the second line check.

6 REFERENCE

a. ISO 7937: 2004 Microbiology of food and animal feeding stuffs – Horizontal method for the enumeration of Clostridium perfringens – Colony-count technique
b. ISO 7218: 2007 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations
c. ISO 7218: 2007/AMD 1: 2013 Microbiology of food and animal feeding stuffs – General requirements and guidance for microbiological examinations – Amendment 1.

Processed manure – Reporting

1 GENERAL

The reporting shall be carried out in accordance with BAM/part 8/20. The sampling report drawn up on the basis of the field records (sampling form) shall be added to the analysis report or incorporated into the analysis report.
Without prejudice to the provisions of BAM/Part 8/20, the analytical report shall include the following information:

a. laboratory letterhead paper with at least name, address, telephone, e-mail;
b. unique report number;
c. unique sample number and, if applicable, sample number assigned by the manure bank via SMIL [1];
d. date and time of sampling;
e. Identification of the sampler (e.g. initials, identification code, SMIL steel nemer number). If the sample has not been taken by a sampler attached to the laboratory, this should be explicitly mentioned in the analytical report.
f. client present at sampling (Y/N);
g. type of processed product (e.g. digestate, compost...) and, if possible, the type of manure from which the processed product is derived (e.g. dried thick fraction – pig manure). The manure codes used by the Flemish Land Agency and included in SMIL should be used for this purpose. The same description as the one used on the MAD, if applicable, shall be used;
h. description of the place of sampling (e.g. container, composting cell...) and or the sample was taken from a lot 'during storage' or 'immediately after processing';
i. GPS coordinates in WGS84 format in decimal degrees to 5 decimal places of the sampled lot;
j. date on which the sample was received by the laboratory;
k. the date on which the sample was taken for analysis;
l. date on which the report was sent;
m. name and signature of the responsible laboratory (possibly digitally);
n. name and address of the person to whom the report is delivered.

2 UNITS

The number of *Escherichia coli* is expressed in number of CFU/g sample.

The number of *Enterococcaceae* is expressed in number of CFU/g of

sample.

Salmonella spp. shall be reported as detectable/not detectable in 25 g of sample.

For *Escherichia coli*, *Enterococcaceae* and *Salmonella* spp. the report shall indicate the results of each of the 5 samples tested separately.

The number of *Clostridium perfringens* is expressed in number of CFU/g sample.

[1] sampling Noding Internet Loket (https://www.vlm.be/nl/doelgroepen/laboratoria-en-staalnemers/SMIL)

3 NAME OF WEBSITE NOTIFICATION (SMIL)

Data on sampling, analysis results and GPS data logos are reported to the Flemish Land Agency via the SMIL application in accordance with the provisions in BAM/part 8/03

Validation and quality control of liquid manure samplers when pumping

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2 **Equipment and materials** 3

3 **Procedure for the extended procedure** 3

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1 PRINCIPLE AND SCOPE

This procedure describes the method of validation of sampling devices used for sampling from watery to pasty liquid streams, for example as mentioned in BAM section 3.
The purpose of the tests is to verify whether:

a. the reproducibility of the sampling is sufficient to ensure the representativeness of the sample for the sampled lot;
b. the way in which a grip is taken out of the liquid flow does not give rise to discrimination when sampling a heterogeneous liquid. For devices that have been validated, this procedure also describes the quality control to be carried out regularly.

Two procedures are described here: an extensive procedure to be carried out only once by the manufacturer, designed to test the compliance of a newly designed device, and a short procedure to verify the quality of a device at regular intervals.

The following observations should be taken into account:

a. The comprehensive and complete procedure should only be carried out when a new type of appliance that has not been previously validated is put into service. Devices that have been subject to this procedure and validated by, or on behalf of, the manufacturer do not need to be re-validated by the user.
b. The short procedure should be carried out on a regular basis regardless of the type of appliance. The frequency is determined by the user depending on the number of annual samples taken, with a minimum of two checks per year.

2 EQUIPMENT AND MATERIALS

a. The sampling device to be validated;
b. The sampling (reference) device of a type which has already been validated with this procedure and whose proper functioning has been demonstrated in accordance with the short procedure;
c. leak-proof laboratory sample receptacles with a volume of at least 750 ml. The size of the containers must be adapted to the grey size of the appliance tested, so that all the grips can be combined in a single container;
d. refrigerated boxes with sufficient refrigeration elements or equipment to ensure refrigerated transport of samples;
e. personal protective equipment.

3 PROCEDURE FOR THE EXTENDED PROCEDURE

3.1 DEVICE DESIGN REQUIREMENTS

The design of the device to be validated shall meet the following requirements:

a. The apparatus is placed in the piping and can take samples from piping under negative pressure as well as under excess pressure with respect to atmospheric pressure.

b. The apparatus takes out of the liquid stream without interrupting the pumping process. The minimum sample volume obtained by taking at least five grabs is 650 ml.
c. The grips are taken in such a way that at the time of sampling part of the flow is sampled, the moment at which a grip is taken is clearly defined and determined (automatically or not) by the operator.
d. The components in contact with the sample flow consist of materials that are sufficiently resistant to corrosion from the streams to be sampled so that no contamination of the sample can occur.

A detailed description of both the construction and the principle of operation of the device shall be included in the validation report to demonstrate compliance with the above requirements.

3.2 PILOT SCHEME: SAMPLING AND ANALYSIS

The tests to evaluate whether the device to be validated meets the following requirements are described below:
a. The variation of the grab size within one sample of a minimum of five grabs, expressed as the coefficient of variation (1s), is not greater than 0,075 (7.5 %).
b. There is no statistically significant difference in grey size as a function of the dry matter content of the sampled liquid within the fork from 0 to 150 kg DS/tonne.
c. In the samples taken, no significant differences are measured for the parameters total nitrogen, total phosphorus and dry matter compared to the concentrations in samples taken simultaneously with a reference apparatus [1].

A number of samples shall be taken placing the apparatus to be validated in series with the reference apparatus so that the batches tested can be sampled simultaneously. The number of grips to be taken to obtain a sufficient sample volume depends (or may depend) on the apparatus used. As far as chemical analyses are concerned, at least the number of grips common to both appliances should be used. In other words, the number of handles does not have to be identical. As regards the weighing tests, it may be appropriate to use an equal number of grips in order to maintain the uncertainty of the calculated results over the apparatus.

Where possible, it is appropriate to use two reference devices. The results of the second apparatus are then considered as back-up and should not be used a priori.

The measurements and analyses described below shall be carried out both on (the samples taken with) the apparatus to be validated and on (the samples taken with) the reference apparatus. A summary of all the samples and analyses to be carried out is given in **Error! Reference source not found**.

Four different lots (manure cellar, storage, etc.) shall be sampled and five samples shall be taken from each lot. The minimum volume pumped for each sample is 6 m^3. The lots to be sampled shall be selected in such a way that:
a. two lots with a dry matter content of less than 20 kg/tonne;
b. two lots with a dry matter content of more than 80 kg/tonne.
The following parameters shall be determined on the samples taken.

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[1] the reference device is a device that can be shown to meet the requirements set out in this document.

3.2.1 WEAVING TEST

During the first of the five samples taken from each of the four lots, the mass of each handle is determined. For the remaining samples, only the mass of the final samples (consisting of x bars) shall be determined.

3.2.2 ANALYSES

The total nitrogen, total phosphorus and dry matter content shall be determined on all samples. The analyses must be carried out by a laboratory approved for these parameters in accordance with VLAREL.

Table 1: sampling and measurements

Party		Sampling	Mass per handle	Total mass Monster	Dry matter content	Tot-N	Tot-P
1	DS < 20 kg/tonne	1	M11,..., m15	M11	D11	N11	P11
		2	—	M12	D12	N12	P12
		3	—	M13	D13	N13	P13
		4	—	M14	D14	N14	P14
		5	—	M15	D15	N15	P15
2	DS < 20 kg/tonne	1	M21,..., m25	M21	D21	N21	P21
		2	—	M22	D22	N22	P22
		3	—	M23	D23	N23	P23
		4	—	M24	D24	N24	P24
		5	—	M25	D25	N25	P25
3	DS > 80 kg/tonne	1	M31,..., m35	M31	D31	N31	P31
		2	—	M32	D32	N32	P32
		3	—	M33	D33	N33	P33
		4	—	M34	D33	N34	P34
		5	—	M35	D34	N35	P35
4	DS > 80 kg/tonne	1	M41,..., m45	M41	D41	N41	P41
		2	—	M42	D42	N42	P42
		3	—	M43	D43	N43	P43
		4	—	M44	D44	N44	P44
		5	—	M45	D45	N45	P45

3.3 DATA PROCESSING

The results of the experiments described above are processed as follows. Remark
The volume of sampling, per handle or per sample, is determined here by weighing. Therefore, if grey or sample size variance is calculated from the weighted masses, it is tacitly assumed that the density of the sampled liquid does not change substantially during the course of the test. If this requirement may be assumed not to be met, the calculation may be made after compensating for the sample density based on the measured dry matter content as follows:

$$P = 1.003 + 4.32\ 10^{-4} \approx DS$$

$$V = \frac{1000 \quad \text{DIE M}}{P}$$

with:
P: density in tonnes/m^3,
DS: dry matter content in kg/tonne;
V: volume of the handle in ml;
M: mass of the grip in grams.

3.3.1 WEAVING TEST

3.3.1.1 VARIABILITY OF THE GREY SIZE

The variability of the grey size is calculated as the pooled coefficient of variation over the results of the weights obtained from the four samples.

Grip	Party 1	Party 2	Party 3	Party 4
1	M11	M21	M31	M41
2	M12	M22	M32	M42
3	M13	M23	M33	M43
4	M14	M24	M34	M44
5	M15	M25	M35	M45
...	...	...	...	...
# measurements (n)	N_1	N_2	N_3	N_4
Variances (σ^2)	Σ_1^2	Σ_2^2	Σ_3^2	Σ_4^2

Calculate the pooled variance as:

$$\Sigma^2_P = \frac{\sum_{I=1}^{4} (N_I - 1)\, \Sigma^2_I}{\sum_{I=1}^{4} (N_I - 1)}$$

and the pooled relative coefficient of variation as:

$$CV_R = \frac{\sqrt{\Sigma^2_P}}{\mu}$$

with μ the average over all measurements.

Note: For the purpose of this calculation, it is tacitly assumed that the variances are homogeneous. If this is not the case, for example if a clear influence of the dry residue on variance is observed even after the density equalisation, the calculation shall be performed separately for the sampling at low and high dry residue.

3.3.1.2 INFLUENCE OF DRY MATTER CONTENT ON SAMPLE SIZE (PART)

In order to determine the influence of the dry matter content on the size of the (sub-) sample, a 2-sided t-test at 95 % significance level shall be used to determine whether the average sample volume for lots 1 and 2 differs significantly from that for lots 3 and 4.

Monster 80 kg/tonne	DS < 20 kg/tonne	DS >
1	M11	M31
2	M12	M32
3	M13	M33

4	M14	M34
5	M15	M35
1	M21	M41
2	M22	M42
3	M23	M43
4	M24	M44
5	M25	M45
average	$\mu_{DS <}$	$\mu_{DS >}$
variance	$\Sigma^2_{DS <}$	$\Sigma^2_{DS >}$

Remarks:

a. It is assumed that the datasets are normally distributed, which is not tested.

b. As $\sigma^2_{DS <}$ and $\sigma^2_{DS >}$ very different, the homogeneity of the variances should be tested first. If these are found not to be homogeneous, a Welch t test should be used.

3.3.1.3 ANALYTICAL RESULTS

The analytical results for dry matter, Tot-N and Tot-P in the samples taken from both apparatus shall be examined for significant differences with a paired t-test (two-sided, 95 % significance).

Remarks:

a. It is assumed that the datasets are normally distributed, which is not tested.

b. It can be assumed that the variances are homogeneous. However, if they appear to be clearly different and confirmed (F-test), a Welch-t test should be switched to use.

3.4 EVALUATION

The apparatus shall meet the requirements laid down if:

a. the apparatus complies with the design requirements (3.1);

b. the pooled relative coefficient of variation (3.3.1.1) is not greater than 0,075;

c. there is no significant influence of the dry matter content on the sample volume (3.3.1.2);

d. no significant difference is observed for any of the three parameters (dry matter, total nitrogen and total phosphorus) between the samples taken with the reference apparatus and those taken with the apparatus to be validated.

4 PROCEDURE FOR LIMITED PROCEDURE – QUALITY CONTROL

Each appliance is subject to normal sleeves when used. In this type of apparatus, the most common shortcoming is the wear of gaskets between the moving parts. This causes leaks, which can lead to poor reproducibility of the size of the grips. This must be checked at least every six months by carrying out the weighing test described in 3.3.1.1. However, it is sufficient to perform the test on a single sample (weighing at least five grabs).

ANNEX A: SAMPLE CALCULATION

As an example for the above rationale, measurements are processed, which were carried out with the device used by VITO. In order to compare the analytical results, samples were taken using an identical apparatus from the Flemish Land Agency.

It was assumed that there were no significant differences in density between the different parties, the weighted masses were equated to the volumes.

A.1 Variability of the grey size

	Well 1	Well 2	Well 3	Well 4
	Fattening pigs	Sows	Z & b	Piglets
mass (g) of partial steel 1	168.4	171.9	166.9	185.9
mass (g) of partial steel 2	173.7	161.4	142.3	176.9
mass (g) of partial steel 3	168.8	175.5	148	177.6
mass (g) of partial steel 4	168.2	162.2	155	182.2
mass (g) of partial steel 5	174.7	153.1	163.2	170
Σ	853.8	824.1	775.4	892.6
µ	170.8	164.8	155.1	178.5
Σ	2.83	8.00	9.16	5.37
VAR	8.03	64.02	83.82	28.81
overall µ	167.3			
Pooled variance	46.17			
CV_R	**0.041**			

The pooled relative coefficient of variation is less than 0,075 and therefore meets the requirement.

A.2 Influence of dry matter content on sample size

Monster	mass (g)	DS (kg/tonne)		Monster	mass (g)	DS (kg/tonne)
Put 1 M1	853.8	105		Put 2 M1	824.1	11
Put 1 M2	863.4	105		Put 2 M2	869	13
Put 1 M3	877.5	106		Put 2 M3	814.6	9
Put 1 M4	845.1	106		Put 2 M4	824.6	9
Put 1 M5	850.2	106		Put 2 M5	791.5	10
Put 3 M1	775.4	98		Put 4 M1	892.6	19
Put 3 M2	911.0	92		Put 4 M2	898.4	13
Put 3 M3	869.0	95		Put 4 M3	880.9	13
Put 3 M4	916.7	100		Put 4 M4	877.7	13
Put 3 M5	875.4	102		Put 4 M5	868.6	12
μ	863,8			μ	854,2	
^{2}	1382			2	1240	

Variances may be assumed to be the same based on an F-test.
There is no significant difference between the mean grey size at the 95 % interval (t-test)

A.3 Analytical results

Pit	Cargo	Dry matter (DS) VITO	Dry matter (DS) VLM
1	1	105	105
1	2	105	105
1	3	106	105
1	4	106	107
1	5	106	106
2	1	11	15
2	2	13	11
2	3	9	9
2	4	9	9
2	5	10	9
3	1	98	98
3	2	92	94
3	3	95	96
3	4	100	101
3	5	102	101
4	1	19	20
4	2	13	12
4	3	13	12
4	4	13	12
4	5	12	13

Pit (N)	Cargo	Total nitrogen (N) VITO	Total nitrogen VLM
1	1	6.55	6.39
1	2	6.65	6.45
1	3	6.31	6.38
1	4	6.31	6.31
1	5	6.47	6.43
2	1	2.75	2.87
2	2	2.72	2.65
2	3	2.50	2.50
2	4	2.54	2.57
2	5	2.59	2.55
3	1	6.52	6.56
3	2	6.66	6.80
3	3	6.89	6.77
3	4	6.94	6.98
3	5	7.04	7.00
4	1	2.99	3.06
4	2	2.66	2.68
4	3	2.67	2.61
4	4	2.69	2.78
4	5	2.70	2.76

Pit	Cargo	Phosphorus (P2O5) VITO	Phosphorus (P2O5) VLM
1	1	5.68	5.61
1	2	5.90	5.79
1	3	5.76	5.81
1	4	5.96	6.02
1	5	5.74	5.91
2	1	0.84	1.08
2	2	0.74	0.60
2	3	0.34	0.37
2	4	0.34	0.35
2	5	0.52	0.31
3	1	4.96	5.02
3	2	4.82	4.78
3	3	4.92	4.88
3	4	5.09	4.90
3	5	5.18	5.03
4	1	1.11	1.15
4	2	0.56	0.61
4	3	0.56	0.43
4	4	0.66	0.67
4	5	0.62	0.66

Parameter	Average difference	95 % interval	Significant?
Dry matter	—0,150	—0,781 + 0,481	No
Tot-N	0,003	—0,039 + 0,045	No
Tot-P	0,012	—0,038 + 0,070	No

It follows from the above that no significant difference is found between samples taken with both devices for any of the three parameters.

Permitted deviation in multiple measurements of nitrate residue and P-Al from agricultural parcels

CONTENT

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2 Practice 3

2.1 Reproducibility of sampling and measurement 3

2.1.1 Nitraat-N 3

2.1.2 Ammonium lactate extractable phosphorus (P-Al) 4

2.2 Evaluation of differences via z-zcore 4

2.2.1 Calculation of z-scores 4

2.2.2 Evaluation and interpretation of results 4

2.3 Remark 5

1 PRINCIPLE AND SCOPE

This procedure describes the testing and interpretation of re-measurements of nitrate residue and plant-available phosphorus (P-Al) from agricultural parcels as listed in BAM section 1.
The purpose of the tests is to verify that the difference between measurements is in line with the measurement uncertainty that can be expected for the method described in BAM deel1/01 (sampling) and BAM parts 1/02,03,04 and 11 (determination).

This procedure is used to evaluate measurements as part of second-line control, evaluation of contra samples, etc.

For the nitrate nitrogen parameter, the evaluation described here is only applicable for evaluating determinations over the full depth sampled and not for the sub-samples per layer.

2 PRACTICE

2.1 REPRODUCIBILITY OF SAMPLING AND MEASUREMENT

2.1.1 Nitraat-N

The reproducibility of the measurement in accordance with BAM was determined by sampling and analysing over a thousand parcels spread over the entire agricultural area in duplicate during the nitrate resistance campaigns between 2011 and 2013. The dataset therefore included a large number of different situations in terms of cultivation (s), soil conditions, nitrate residue size, weather conditions, time lag between sampling, etc. The two samples and analyses were carried out by various laboratories, although approved, within the deadline for the nitrate resistance campaign. The calculated reproducibilities should therefore be considered as maximum values as they contain contributions from both sampling, analysis and an interlaboratory and a temporal component.

Analysis of variance shows that the factor with the greatest impact on differences between two consecutive measurements is the last crop. The table below shows the pooled coefficients of variation for these crop groups between which a statistically significant difference could be demonstrated.

Crop group (s)	CVR
Potatoes	0,22
Cereals	0,29
Maize	0,26
Grass	0,32
Other [1]	0,30

Table 1: coefficient of variation by crop group (s)

2.1.2 Ammonium lactate extractable phosphorus (P-Al)

For P-Al, the coefficient of variation was pooled from ring test results with sampling since 2014 and a field test was carried out on a dozen plots following a sample optimisation study. The last crop does not affect this, a fixed coefficient of variation of 0,12 is used.

2.2 EVALUATION OF DIFFERENCES VIA Z-ZCORE

2.2.1 Calculation of z-scores

Differences between two or more measurements can be evaluated by calculating z- scores if:

$$Z_I = \left|\frac{(X_I - M)}{CV\ M}\right|$$

With
:

z_i: the z-score for the i-th measurement
X_i: the i-th measurement
μ: arithmetic mean of all measurements
CV: is the coefficient of variation according to Table 1 in case of NO 3-N, or equal to 0,12 for P-Al

2.2.2 Evaluation and interpretation of results

After calculation, z-scores are evaluated if:

$$Z = \begin{cases} \leq 2 \text{ EXPECTED} \\ 2 < z \leq 3 \text{ doubtful} \\ > 3 \text{ UNLIKELY} \end{cases}$$

- When the z-score is less than or equal to 2, the difference can be allocated to the expected variance caused by sampling and analysis. The measurement is perfectly acceptable.
- When the z-score is greater than 2 but less than or equal to 3, there is still only 5 % probability that the difference is explained by the expected variance caused by sampling and analysis. The measurement is *questionable*. *Further research* may be carried out and/or

[1] ornamental plants, fruit, flowers, sugar beet, fodder beet, vegetables, strawberries, etc.

interpretation of cross-compliance is carried out but measurement cannot be accepted automatically.
- When the z-score is greater than 3, there is less than 1 % probability that the difference was caused by the expected variance caused by sampling and analysis. The measurement is *unlikely* and cannot be accepted.

Where evaluation of two analytical results reveals an inexplicably large difference (z-score > 2) it is certainly recommended to carry out a third sampling and measurement.

Values that mutually result in z-score (s) less than or equal to two may be averaged.

2.3 REMARK

Local conditions such as the presence or absence of crop residues, prevailing temperature and soil moisture, rainfall, time lag between measurements, etc. may influence the differences between two consecutive measurements. As a result, the actual variance for the plot in question may differ from the values shown in Table 1.

Reporting of samples, results and GPS data logos via SMIL

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1 Principle 3

2 Soil sampling and analysis 3

2.1 Nitrate residue determination 3

2.1.1 Pre-notification of sampling 3

2.1.2 Loading of analytical results and GPS data logos 4

2.2 Other soil samples 4

2.2.1 Notification of sampling 4

2.2.2 Reporting the results 4

3 Manure sampling and analysis 5

1 PRINCIPLE

This procedure determines which samples must be notified via the Staalname Melding Internet Loket (SMIL), an internet application made available by the Mestbank, and how both the sampling, the results and the GPS datalog data must be notified.

2 SOIL SAMPLING AND ANALYSIS

All applications for sampling and analysis of agricultural parcels are submitted by the farmer via the sampling application on the Mestbankloket (SNAPP). The accredited laboratory receives these applications via SMIL and may accept or refuse an application. By accepting the application, the laboratory indicates that it will carry out the sampling (s) and analysis (s). If an accepted sampling has not been carried out, it must be reported and the reason given.

2.1 NITRATE RESIDUE DETERMINATION

2.1.1 PRE-NOTIFICATION OF SAMPLING

Each sampling for the determination of the nitrate residue should be **pre-notified via SMIL at the latest the day before sampling**. The pre-notification shall include the following information:

a. the planned date of sampling
b. the time block in which the sampling of the lot or sub-lot, as the case may be, will be carried out.
c. the identity of the sampler who will carry out the sampling (if the pre-notification is made in xml, the sample sampler number of the registered sampler is used for this purpose).

The time block shall be selected from the list of time blocks available in SMIL. Sampling for sunrise and after sunset is not permitted. A pre-reported sampling should be initiated within the chosen time block; multiple samples can be pre-reported within one time block.

If the sampling was not carried out on the notified date, it must be postponed to a later date or notified at the latest the day after the notified sampling date if the sampling is not planned again. When sampling is planned again, the new planned sampling and time block must again be notified no later than the day before sampling takes place.

Pre-notification, and if necessary postponement, shall be made either by the approved laboratory or by the registered sampler itself.

If the sampling was not carried out by the sampling officer indicated in the pre-notification, the accredited laboratory must report (intermediate report) the name of the sampling officer who actually carried out the sampling no later than the day after the sampling took place.

2.1.2 LOADING OF ANALYTICAL RESULTS AND GPS DATA LOGOS

Analytical results shall be reported via SMIL no later than 14 day after sampling. The following information shall be included:

a. the steel number
b. nitrate nitrogen [1], expressed in kg N/ha and rounded to the integer, per layer sampled and the final depth of the layer sampled
c. the date of analysis

No later than 3 working day after sampling, GPS data logged data, linked to the registered sampler's steel sampler number, are loaded in SMIL in gps exchange format (gpx).

2.2 OTHER SOIL SAMPLES

2.2.1 NOTIFICATION OF SAMPLING

Each sampling for determining the phosphate class, for derogatory nitrogen samples, for derogatory phosphate samples or for a mandatory nitrogen sample with fertilisation advice must be notified via SMIL at **the latest on the day the sampling is carried out**. This notification is triggered by the acceptance of the application submitted by the farmer for this purpose via SNAPP. The farmer's application must therefore be accepted by the laboratory in SMIL no later than the day on which the sampling is carried out.

2.2.2 REPORTING THE RESULTS

2.2.2.1 GENERAL PROVISIONS FOR REPORTING THE RESULTS

No later than 30 days after sampling, the result of the sampling shall be reported via SMIL. The following information shall always be included:

a. the steel number
b. the date of sampling
c. the identity of the sampler who carried out the sampling (if the notification is made in xml, the sample sampler number of the registered sampler is used for this purpose)

2.2.2.2 SAMPLING FOR DETERMINING THE PHOSPHATE CLASS

For sampling in the context of applications for determining the phosphate class, in addition to the data listed under 2.2.2.1, the following data shall also be reported:

a. plant-available phosphate (P-AL) [1] expressed in mg P/100 g air dry soil rounded to the integer
b. the sampling depth

2.2.2.3 SAMPLING FOR MANDATORY NITROGEN SAMPLES WITH FERTILISATION ADVICE

For samples taken in the context of applications for mandatory nitrogen samples with fertilisation advice, the laboratory shall confirm by means of a notification that the nitrate nitrogen content and

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[1] for values below the reporting limit, the reporting limit shall be reported. When this happens occurs, it can be reported in the comment field e.g. with the symbol "<"

ammoniacal nitrogen was determined. This notification shall be accompanied by the following information:
2.2.2.1 the following data shall also be reported:
a. whether a fertilisation opinion was given
b. the internal sample number by which the laboratory uniquely identifies the sample

2.2.2.4 SAMPLES FOR DEROGATORY NITROGEN SAMPLES

For sampling in the context of applications for derogatory nitrogen samples, the laboratory shall confirm by means of a notification:
a. whether the nitrate nitrogen and ammoniacal nitrogen content was determined
b. whether nitrogen fertilisation advice was given
The notification shall always include the information referred to in point 2.2.2.1.

2.2.2.5 SAMPLES FOR DEROGATION PHOSPHATE SAMPLES

For sampling in the context of applications for derogatory nitrogen samples, the laboratory confirms by means of a notification whether the plant-available phosphorus (P-AL) has been determined. The notification shall always include the information referred to in point 2.2.2.1.

3 MANURE SAMPLING AND ANALYSIS

Each sampling for the determination of nitrogen and phosphorus content of manure should be notified via SMIL preferably on the day of receipt of the sample at the laboratory and at the latest at the start of the analysis [2].

The notification shall include the following information:
a. the date of sampling
b. the identity of the sampling officer who carried out the sampling (if the notification is made via xml, the sampling officer's number is used for this)
c. number of farmer or operator
d. number of operation or operation
e. the manure code (to be selected from the list of manure codes available in SMIL)
f. location of sampling (Loading, Storage, Unloading)
g. group if applicable to the holding

The results [shall] be reported via SMIL no later than 14 the day after sampling. The following information shall be included:
a. the steel number
b. location of sampling (Loading, Storage, Unloading)
c. total nitrogen expressed in kg N/1000 kg VM [3]
d. total phosphorus, expressed in kg P_2O_5/1000 kg VM 3

[2] since manure samples must be processed for analysis at the latest on the 7th day after sampling, the sampling must therefore be notified in SMIL at the latest on the 7th day after sampling.
[3] reported values shall be rounded to two decimal places for values ≤ 1 and to one decimal place for values > 1. For values below the reporting limit, the reporting limit shall be reported. When this occurs, it can be reported in the comment field e.g. with the symbol "<"

GPS Data Logging

CONTENT

1 Principle 3

2 Requirements for the GPS data logger 3

3 Use of the logger 3

3.1 Use of the logger in soil sampling 3

3.2 Use of the logger for manure sampling 3

4 Storing the GPS data logos 4

1 PRINCIPLE

A GPS data logger should be used for each sampling. This procedure describes how the GPS data logger should be used for the different samples taken.

2 REQUIREMENTS FOR THE GPS DATA LOGGER

The GPS data logger shall record at least the date, time and associated coordinates in the form of world coordinates according to the WGS84 reference system, in decimal degrees to 5 degrees.

The GPS data logger shall be set such that the time interval between two records:

a. maximum 10 seconds when soil is sampled for nitrate residue determination
b. shall not exceed 5 seconds for all other samples.

3 USE OF THE LOGGER

The accredited laboratory shall make a GPS data logger available to each sampler.

The proper functioning of the GPS data logger shall be checked on a daily basis. A GPS data logger that (possibly) does not function properly shall not be put back into service until it has been established that it is functioning properly again.

Sampling should only start when the GPS data logger is active and thus effectively receives and stores GPS signals.

3.1 USE OF THE LOGGER IN SOIL SAMPLING

For soil samples, the entire sampling path is logged for each (sub) plot. The sampler therefore keeps the GPS data logger up throughout the sampling process and starts logging at least when entering the plot to be sampled and stops logging at the earliest when leaving the plot.

3.2 USE OF THE LOGGER FOR MANURE SAMPLING

For manure samples, at the start of each sampling, the sampler logs the GPS coordinate of the sampling site (house, storage, place of loading or unloading).

4 STORING GPS DATAL DATA DATA

The log-records shall be stored as a gpx file and contain for each data point at least the date, time and corresponding coordinates in the form of world coordinates according to the WGS84 reference system, in decimal degrees to 5 degrees.

Each logged sampling path (soil) or logged sampling site (manure) shall be uniquely linked to the sampling sampler number that carried out the sampling.

Quality requirements for analytical methods

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1 Purpose and scope 3

2 Quality monitoring of sample preparation 3

3 Quality requirements for inorganic parameters 3

1 PURPOSE AND SCOPE

Analysis of a sample involves carrying out as faithfully as possible a full series of steps in the laboratory from the usual sample preparation (sampling by sampling, etc.) up to and including measurement and calculation. The quality system shall include each of these steps.

The measurement quality requirements included in this procedure are generally applicable for the determination of various chemical parameters in soil and manure. As regards the quality requirements for the determination of inorganic parameters, general guidelines were laid down in this procedure. In addition, specific/additional requirements for a specific inorganic parameter may be prescribed in the relevant procedure.

2 QUALITY MONITORING OF SAMPLE PREPARATION

For example, the quality follow-up of sample preparation may include duplicate analyses on a number of parameters (excluding dry matter content). For this purpose, 2 sub-samples are taken after the sample pre-treatment and go through the entire analytical route.

3 QUALITY REQUIREMENTS FOR INORGANIC PARAMETERS

(1) Linearity

- The calibration model (linear or quadratic) shall be recorded during validation and shall be periodically checked according to one of the following procedures:
    - At least a six-monthly check of the calibration model and at each major instrumental intervention; or
    - Residual control standard deviations in linear/quadratic regression analysis, applicable only if the whole measurement range is calibrated.

(2) Sensitivity

- At least at the beginning of each measurement series, the sensitivity of the device shall be checked. The device must be and remain sufficiently sensitive to meet the reporting limit. The procedure used may be completed by the laboratory.
  E.g. ion chromatography, spectrophotometry: area of a particular parameter The independent control standard and/or drift control solution may be used for this purpose.

(3) Calibration

- If calibration is performed by means of a calibration line, at least 5 calibration solutions (including zero if applicable) shall be analysed for each measurement run and spread over the linear range. Then, dmv linear regression, the calibration line equation shall be calculated with a coefficient of correlation of at least 0.995. A maximum of 1 points may be removed, but not the lowest point, and a minimum of 4 points must be retained.

Removal of the highest calibration point results in a reduction of the calibration area. The deviation from each point to the straight line shall not exceed 10 % (except point at level ≤ 2 x reporting limit). The deviation from the point at level ≤ 2 x reporting limit shall not deviate from the straight line by more than 25 %.

- If the reporting limit is lower than the 1 ⁄ 2 of the lowest concentration of the calibration solution (different from zero), an additional check should be performed at the reporting limit level. The deviation from the point at reporting limit level shall not differ from the theoretical value by more than 25 %.
- If one of the criteria is not met, a new calibration will be established with or without new calibration solutions.

Note: Standard solutions are prepared in a similar medium (e.g. acid, H_2O,...) as the samples.

Note: The permitted deviation should always be recorded 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, then a quadratic curve can be used for calibration. A maximum of 1 point may be removed, but not the lowest point, and at least 5 points (different from origin) must be retained. Removal of the highest calibration point results in a reduction of the calibration area. The deviation from each point to the straight line shall not exceed 10 % (except point at level ≤ 2 x reporting limit). The deviation from the point at level ≤ 2 x reporting limit shall not deviate from the straight line by more than 25 %.
- If one of the criteria is not met, a new calibration will be established with or without new calibration solutions.

Note: The permitted deviation should always be recorded 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 shall be 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 measurement value, whichever is higher of the two;
  - Independent control standard and/or drift control: the measured concentration shall not deviate from the actual value by more than 10 %.

- The calibration curve shall be verified at the end of each measurement series by analysis of the drift check. The measured concentration shall not deviate from the actual value by more than 10 %.

- For each set of samples, a procedure blank is included. The procedure blank runs through the full cycle of analysis. The value of the blank must be monitored. Unless stated in the procedure, the blank correction is not applied.

Note: The procedure blank shall always be checked using varying containers.

- Where no daily calibration is performed, at least the following checks shall be performed:
  - Measurement of (highest) calibration standard: the measured concentration shall not deviate from the actual value by more than 10 % (optional).

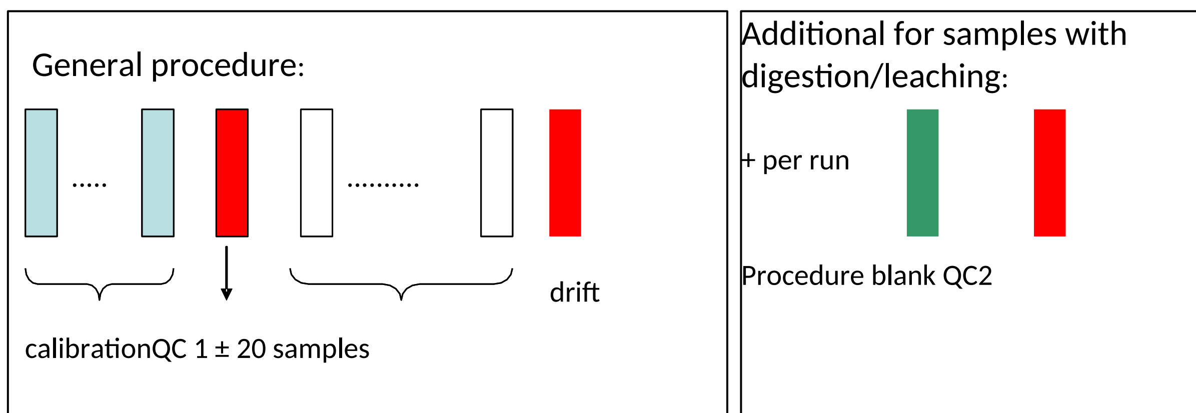
- o Procedure and/or calibration blank solution: the blank measurement value is less than half of the reporting limit or max. 10 % of the measurement value, whichever is higher of the two;
- o Independent control standard and/or drift control: the measured concentration shall not deviate from the actual value by more than 10 %. A linear regression measures 1 control standard, while a quadratic regression measures 2 control standards (e.g. concentration levels 1/3 and 2/3 of the measurement area).

(4) Generally applicable procedure (including sample pre-treatment) for methods using a calibration line

The following procedural clarification is set out in the diagram below:

- QC 1: independently generated audit;
- Analysis sequence of ± 20 samples: this is an indicative number, laboratories should be able to demonstrate that the frequency of implementation of QA/QC has been chosen to provide sufficient guarantees of quality;
- Drift: calibration standard or independent standard (QC1);
- For samples with rendering/leaching, a procedure blank and a QC sample (QC2), both of which have gone through the whole procedure, should be analysed for each run (run of a digestion/leaching device).

Additional provision should be made for dealing with possible memory effects.



The quality control criteria should be defined within the laboratory in such a way as to meet the performance characteristics laid down by law. The measurement value of QC1 shall be within ± 10 % of the actual value.

(5) Control steel

For each batch of samples at least 1 parameter (excluding dry matter) per day or per batch of 50 samples started, 1 control sample shall be included. This control language runs through the full cycle of analysis.

The following may be used as control steels:

- a certified reference material
- a real steel
- defined synthetic matrix
- a husked sample, using a matrix representative of the samples analysed in the laboratory. All representative matrices should be covered over time.

(6) Duplicate analyses (as an alternative to control sample, if not available)

For each set of samples, at least 1 parameter (excluding dry matter) per day or per started set of 50 samples shall be analysed in duplicate. For this purpose, 2 sub-samples are taken after sample pre-treatment and go through the entire analytical route.

Conditions for reporting of sampling data and analytical results by an accredited laboratory

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1 PURPOSE AND SCOPE

This procedure prescribes how laboratories, which are accredited in the Flemish Region according to VLAREL or still have accreditation according to previous regulations, must report sampling data and analysis results, of tasks carried out as accredited laboratories.

The purpose of this procedure is to lay down uniform reporting requirements. The conditions in this procedure are to be regarded as minimum requirements, which take precedence over any bilateral arrangements between the approved laboratory and its client, unless the competent authority provides otherwise.

The scope of this procedure covers all laboratories approved in the Flemish Region for one or more of the following disciplines: water; air; manure; animal feed; soil; wastes and other materials. In terms of content, this procedure covers both sampling and analysis, in so far as the approval of laboratories is provided for.

The ISO/IEC 17025 standard, which lays down general requirements for the competence of laboratories and must be applied by accredited laboratories, already contains reporting requirements, but these are formulated in rather general terms and do not sufficiently address the typical problems of environmental analysis (e.g. limited shelf life of samples...). Reporting requirements in the ISO/IEC 17025 standard, which are not explicitly included in this procedure, shall continue to apply to the accredited laboratories. This also applies to reporting instructions applicable to a specific discipline (possibly sub-domain), for example:

- methods BAM/part n/20 on reporting (see http://www.emis.vito.be/referentielabo-vlm);

This procedure applies to both paper and electronic reporting. If an electronic file is used, it must comply with all the requirements of this procedure; otherwise, a classical paper analysis report that meets all the requirements of this procedure must always be sent.

Provisions in square brackets are not strictly part of this procedure but are added for clarification purposes.

2 DEFINITIONS

For the purpose of this procedure:

1° VLAREL: Decree of the Flemish Government of 19 November 2010 establishing the Flemish Regulation on accreditations relating to the environment;

2° ‘fit for purpose’: suitable for use in the context envisaged by the sponsor (e.g. specific needs) or considered to be known by an accredited laboratory (e.g. applicable regulations);

3° approved laboratory: laboratory, accredited by the Flemish Region, which carries out sampling, measurements, analyses or tests in accordance with the provisions of VLAREL;

4° contractor: accredited laboratory with which the contracting authority has a contractual relationship;
5° final report: the analysis report received by the contracting authority from the contractor;
6° reporting limit: the value below which a component is reported as not quantifiable (‘<’); it shall be at least the limit of quantification, unless otherwise specified in the applicable legislation.

3 PRINCIPLES

The following basic principles were used to define the conditions for reporting:

- the sampling data and analysis results reported by an accredited laboratory shall be ‘fit for purpose’, i.e. fit for use within a prescribed context;
- the approved laboratory must provide these ‘fit for purpose’ sampling data and to obtain analytical results in the most efficient way.

In order to achieve this, it is essential that throughout the analysis process in its broadest sense – from the definition of the need for analyses by the client to the reporting of the results by the contractor, which is the accredited laboratory entrusted with the task – sufficient consultation takes place and clear agreements are made.

Both sponsors and approved laboratories have a share of the responsibility to efficiently obtain ‘fit for purpose’ analytical results.

An approved laboratory may expect the sponsor and shall, if necessary, ask the sponsor to:

- communicates 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, at the latest at the time of sampling (if carried out by the laboratory) or at the time of delivery of the sample to the laboratory;
- align specific requirements as much as possible with the requirements for the use of the accreditation to which the laboratories are subject through VLAREL (e.g. mandatory use of compendium method if available...).

What is crucial is that the developer correctly indicates the nature of the sample and the objectives in the context of which the analysis is carried out.

The successful tenderer and, where appropriate, the other approved laboratories involved in the performance of the contract will be expected to:

- where necessary, assist the developer in defining a sampling 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 analytical work until a fit for purpose result is available, even if that requires additional measurement/analysis;
- optimise its own organisation/methods to minimise the use of comments on sampling and analysis reports.

The ultimate responsibility for correctly formulating the study to be carried out and indicating the applicable regulations lies with the developer.

The final responsibility for the complete and correct reporting in accordance with this procedure is assigned to the accredited laboratory acting as contractor. This can be either the laboratory performing the sampling or the laboratory performing the measurements/analyses (or at least part of them), depending on who has a contractual relationship with the client. Where, for a given contract, the contracting authority enters into a contractual relationship with two or more different contractors, each of those accredited laboratories shall be responsible for its own part of the contract.

The final responsibility for reporting implies responsibility for the necessary coordination between all the approved laboratories involved: ensuring the necessary agreements on critical deadlines in the analysis process, availability of a sampling report...

If the sampling is carried out by another competent body (e.g. inspection service; drilling under the supervision of an approved soil remediation expert; operator if provided for in the regulations...) or carried out by the contracting authority itself, the final responsibility of the approved laboratory is limited to sensitising to compliance with the conditions in this procedure. In such a case, the approved laboratory will refer to the approval status in the analytical results, but not in the sampling data.

4 REPORTING OF CONTRACT DATA

4.1 SAMPLE DESCRIPTION

The description of the sample on the analysis report must be consistent with the context of the analysis order and with the characteristics of the sample (at least the visually observable characteristics). The approved laboratory may expect the sponsor to describe correctly the nature of the sample and the purposes for which the analysis is carried out. On receipt of the sample, however, 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 as such as a normal sample of the type in the description (e.g. sample contains more phases), the accredited laboratory should contact the sponsor to clarify the inconsistencies. The agreements made in this connection must be recorded.

4.2 REVIEW FRAMEWORK

As a service to the sponsor or upon explicit request, an accredited laboratory may include test values in the analysis report. In that case, it must also be clearly indicated from which regulation or other document those assessment values originate.

4.3 SAMPLE SEALING

Where samples are delivered sealed, the approved laboratory shall indicate on the analysis report whether the seal was intact when the samples were received. Where

a non-intact seal shall be photographed by the approved laboratory; it should then be kept with the details of the order and transmitted as evidence at the request of a competent authority.

5 REPORTING OF THE DATA OF MONSTERN

Note: the following conditions apply only if the sampling has been carried out by an approved laboratory; this may be the approved laboratory which also carries out the analyses, or another approved laboratory.

5.1 MINIMUM SAMPLING DATA

The analysis report, or a sampling report accompanying the analysis report, must contain at least the following information on sampling:

- date of collection;
- hour of sampling in the case of waste water, surface water, groundwater, air or waste and other materials;
- ~~name~~ of the sampler (e.g. initials, identification code,...);
- identification (preferably via GPS coordinates) or detailed description of the location where the sample was taken, accompanied where necessary by sketches or photographs;
- recording the characteristics of the measurement site and, if applicable, checking them against the compendium or the other required method according to Article 45 of VLAREL;
- in the case of sampling of a lot: description of the sampled lot, including lot demarcation or mixed lot if applicable; reasons must also be given for the choices made;
- reference to the sampling method, comprising at least the code of the compendium or standard method applicable and the specification of the sampling technique used;
- additional information if required by the sampling method or regulations; for example, for waste and other materials: number of grips taken, grip size, number and size (in litres) of laboratory samples, cf. CMA/1/A.14;
- if more than one subdomain is defined for the approval as a laboratory in the discipline concerned: the sub-domain covered by the sampling

If a sampling report is used to report the above data, the data of which are not reproduced in the analysis report, a clear reference to that sampling report must be made in the analysis report; the analysis and sampling report are then both sent to the client and archived. In practice, the sampling report and the analysis report may also form part of an integrated study report, including, for example, a discussion of the results or technological advice.

If the sampling has been carried out at the request of another accredited laboratory acting as contractor, the laboratory carrying out the sampling shall ensure that a sampling report containing at least the above information is made available to the other laboratory at the latest when the samples are submitted.

A successful tenderer who carries out the sampling himself, but who uses another approved laboratory for the analyses, or part of them, does not have to provide that laboratory with a full sampling report, since the successful tenderer is responsible for the final report. In such a case, it is sufficient to indicate the date on which the sample was taken and, where appropriate, the sub-area to which the sample was taken.

When sampling is carried out in the presence of a competent body which itself records some of the minimum data, the sampler must have the conformity of the recorded data verified by the representative of that competent body.

The name and address of the approved laboratory which carried out the sampling must be unequivocally identifiable from the analysis report, or a sampling report attached to the analysis report, and correspond to the one on which the approval was issued.

5.2 VLAREL accreditation logo and reference to the accreditation status for sampling

In accordance with Article 49 of VLAREL, an accredited laboratory must affix the VLAREL accreditation logo to the analysis report or sampling report:

In addition, the laboratory must clearly indicate the samples for which it is approved and those for which it is not. The following rules shall apply:
- each sampling method in an analysis or sampling report must clearly indicate, by means of a code/symbol of its choice, whether or not it falls within the scope of the laboratory's approval;
- the code or symbol used shall be explained, for example in a footnote to the analysis or sampling report.

If more than one subdomain is defined for the approval as a laboratory in the discipline concerned, this should be taken into account when referring to the approval status.

For the reference to the approval status for measurements, tests and analyses, reference is made to 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 client's request, the sampling was not carried out in full accordance with the compendium or the other method required under Article 45 of VLAREL.*

   In this case, it must be clearly indicated that it is a derogation at the request of the developer; it is also necessary to specify the deviations from the compendium or the other method required under Article 45 of VLAREL. If the developer has indicated why he considers the modification necessary, this may also be indicated.

b) *if, due to local conditions, sampling could not be carried out in full compliance with the compendium or the other required method according to Article 45 of VLAREL.*

   In this case, it must be specified what departed from the compendium or other method required under Article 45 of VLAREL and what local circumstances made it necessary.
   An accredited laboratory is expected to clearly indicate any defects in a client's installation 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 identified.*

   In this case, it must be specified which abnormalities related to the operation are involved and whether they have been reported by the client or established/confirmed by the approved laboratory itself.
   An accredited laboratory should not pronounce on whether reported abnormalities are justified or not or on the impact that the abnormalities may have had on the representativeness of the sampling.

A comment on the analysis report is necessary in the following case:

d) *if, on receipt at the analytical laboratory, a sample is found not to be in conformity with the compendium or the other method required in accordance with Article 45 of VLAREL in terms of preservation, container or quantity.*

   In this case, it must be specified where the requirements of the compendium or the other method required under Article 45 of VLAREL were not met.

The inclusion of comments on sampling should not lead to the omission of the reference to approval and therefore does not exempt the approved laboratory from the other requirements of this procedure.

6 REPORTING ON CRITICAL DEADLINES IN THE ANALYSIS PROCESS

6.1 MINIMUM DATA

The analysis report should include at least the date of receipt of the sample by the laboratory and the date of analysis.

6.2 HANDLING OF PRESCRIBED SHELF-LIFE OF SAMPLES

For the interpretation of shelf-life, the date of sampling is considered to be day 0. If a composite sample has to be prepared by the approved laboratory from several samples provided, day 0 is the date of sampling of the oldest sample.

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 a digestion, extraction or other preparatory action, this is normally the date on which the aforementioned action was carried out. In other cases, it is the date on which the level was measured.

The successful tenderer is responsible for respecting the prescribed shelf-life of the samples for

each parameter to be determined. Where, for a given contract, the contracting authority enters into a contractual relationship with two or more different contractors, each of these accredited laboratories shall be responsible for its own part of the contract.

[An accredited laboratory ultimately responsible for reporting is expected to monitor for each parameter (group) how often the shelf-life is exceeded and to make use of the results of this monitoring in the continuous improvement of its processes or organisation under 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 was exceeded for a particular parameter (group)

In this case, the parameter (group) should be clearly indicated.

The laboratory must, on request, inform the client whether the exceedance is due to the laboratory itself, the preliminary procedure (= late delivery of the sample or the analysis order) or a combination of the two.

b) if the date of sampling is not known by the laboratory drawing up the analytical report.

In this case, it must be clearly indicated that the date of sampling was not communicated to the laboratory and that, as a result, the carrying out of the analyses within the prescribed shelf-life could not be guaranteed.

7 REPORTING OF ANALYTICAL RESULTS AND INFORMATION ON THE METHOD APPLIED

7.1 MINIMUM DATA

The analysis report shall include at least the following information on the analytical results and methods:
- the analytical result and the unit in which it is expressed;
- a reference to the applied isolation, leaching and analysis method;
- additional information if required by the analytical or regulatory method;
- relevant deviations or explanation of the analysis.

The unit in which the final analytical result is expressed must be in accordance with the assessment framework. The naming of the parameters should also be aligned as much as possible with the regulations.

The reference to the isolation, leaching and analysis method, as included in the analysis report, must include at least the code of the applicable compendium method or the other required method according to Article 45 of VLAREL.

Relevant deviations or analytical explanations shall mean any additional information necessary for a correct interpretation of the analytical results by the sponsor and any end user. See also further section 7.4 Use of comments on analysis reports.

The name and address of the approved laboratory which carried out the analysis must be unequivocally identifiable from the analysis report and correspond to the laboratory to which the approval was issued.

7.2 ACCREDITATION LOGO VLAREL AND REFERENCE TO THE ACCREDITATION STATUS PER PARAMETER

In accordance with Article 49 of VLAREL, an accredited laboratory must include the accreditation logo VLAREL on the analysis report:

The laboratory must also clearly state for which measurements, tests and analyses it is approved and for which it is not. The following rules shall apply:
- each analytical result reported must clearly indicate, by a code/symbol of its choice, whether or not it falls within the scope of the laboratory's approval;
- the code or symbol used shall be explained, for example in a footnote to the analysis report.

If more than one subdomain is defined for the approval as a laboratory in the discipline concerned, this should be taken into account when referring to the approval status.

For the reference to the approval status for sampling, reference is made to point 5.2.

7.3 Calculation of additivity or difference levels

If a method for calculating additivity or difference levels is laid down in the legislation in question, the compendium in question or the other method required under Article 45 of VLAREL, that method must be used.

7.4 Use of comments on 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 which, according to the compendium or the other method required under Article 45 of VLAREL, could be determined only indicatively or semi-quantitatively.

In this case, a symbol of one's choice shall be used to replace or clarify the result. That symbol shall be explained by a footnote or note to the analysis report.

A reporting limit in the result field is considered as a result; a comment is only necessary if an overly high (i.e. higher than required by regulation) reporting limit is transmitted (see point (c)). '>' Values are also considered as a result, but their use is only allowed if explicitly stated in the compendium or the other required method according to Article 45 VLAREL (e.g. when determining BOD in water).

The laboratory should make additional information available to the sponsor on request, for example, why no result could be obtained.

b) If a discrepancy in calibration or quality control may compromise the fitness for purpose of the result and that discrepancy could not be remedied by re-measurement/re-analysis with additional precautions, cf. compendium or other required method according to Article 45 of VLAREL.

In this case, the derogation must be clearly defined: to which parameter (s) it refers, what differs and in what direction.

The laboratory should provide additional information upon request, such as the likely impact on the reported analytical result (suspected underestimation or overestimation...)

or make available to the developer what has been done to eliminate the derogation.

[As regards the elimination of such derogation, it is understood that a “reasonable” extra effort on the part of the accredited laboratory (= 1 re-analysis) is sufficient, provided that sufficient additional technical precautions have been taken (e.g. use of an additional purification technique, use of an adapted sample quantity, etc.).

If, at the time the derogation is established, the shelf-life of the sample has already expired, no further re-analysis with additional precautions needs to be initiated.]

c) If a higher reporting limit than required by regulation or by the customer is passed as a result.

In that case, it should be specified that the reporting limit was increased.

The laboratory shall make available to the sponsor, upon request, additional information such as the reason (dilution due to matrix effects that cannot be eliminated, excess blank value...) or the action taken to eliminate that deviation (see point (b)).

[The diluted use of samples to reduce matrix effects is only acceptable if it does not compromise the fitness for purpose of the analytical result. This should therefore not lead to an excessive reporting limit as a result, unless other technical possibilities to limit matrix effects (e.g. compendium or other method required) have proven insufficient for the sample in question.

If one or more of the set of components to be determined by a specific method of analysis are present in the sample at extremely high levels, the reporting limits for the other components should, as a general rule, not exceed the guide or limit value for the content of those components according to the applicable regulatory provisions.]

d) If the compendium or other method required by Article 45 of VLAREL had to be deviated from when carrying out sample preparation, analysis or calculating the content.

In that case, the derogation should be clearly defined, as well as the reason for the derogation.

e) If additivity or difference contents are reported where at least one of the components has a measurement value below the reporting limit (< RG) and the procedure to be applied is not laid down in the legislation in question, the compendium in question or the other method required under Article 45 of VLAREL.

In this case, the way in which the < RG measurement values have been handled shall be clearly explained.

f) Where more than one result is reported for a parameter to be determined and those results differ significantly, taking into account the context of the analysis.

In this case, the difference between the results should be explained and the result which the laboratory considers to be the most reliable should be indicated.

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 approval and therefore does not exempt the approved laboratory from the other requirements of this procedure.

8 REPORTING IN CASE OF (PARTIAL) SUBCONTRACTING

If samples or analyses, which according to VLAREL must be carried out by an accredited laboratory, are subcontracted to another accredited laboratory, the following reporting guidelines apply (for the definition of the term final report, see point 2):

- the sampling or analysis report of the executive laboratory shall be fully annexed to the final report; the final report and its annexes therefore form a single whole;
- the approval status of the subcontracted sampling or analysis shall indicate by a code/symbol of its choice that the subcontracted operations are concerned; the code or symbol used must be explained, for example in a footnote to the final report;
- the method reference for the outsourced samples or analyses in the final report shall be replaced by the term ‘outsourced’ or by the method reference of the executive laboratory.

A contractor who subcontracts part of the contract bears ultimate responsibility for the whole, in accordance with the provisions of the ISO/IEC 17025 standard, and is therefore liable both for the correct transfer of the samples to/from the other implementing laboratory and for the results/data provided by that laboratory.

Processing of personal data following sampling and analysis of manure, soil or fodder in the context of the Fertiliser Decree

CONTENT

1 **Principle** 3

2 **Essential elements of data processing** 3

3 **GPS data** 3

1 PRINCIPLE

The processing of personal data following sampling and analysis of manure, soil or feed under the Fertiliser Decree is carried out in accordance with the provisions of Regulation (EU) 2016/679 of the European Parliament and of the Council of 27 April 2016 on the protection of natural persons with regard to the processing of personal data and on the free movement of such data, and repealing Directive 95/46/EC (hereinafter the GDPR Regulation).

2 ESSENTIAL ELEMENTS OF DATA PROCESSING

In general, the processing of personal data following sampling and analysis of manure, soil or fodder is carried out within the framework of the Fertiliser Decree, with a view to achieving the objectives of the Fertiliser Decree, as referred to in Article 2 of the Fertiliser Decree. In order to achieve these objectives, correct sampling and analysis are necessary.

The personal data processed in the context of sampling and analysis are essential to ensure correct sampling and analysis. The purpose of the processing of personal data in this context is twofold. On the one hand, make it possible to identify the sampling and analysis carried out in an unequivocal manner. On the other hand, these personal data are intended to verify the correctness of the sampling and analysis process.

Personal data processed in the context of sampling and analysis shall be retained for a maximum of 5 calendar years from 1 January following the date of sampling.

The accredited laboratory shall ensure that access to the personal data is limited to the persons who need the data for the performance of their duties.

The accredited laboratory shall ensure that the data subjects concerned by the processing of personal data are informed thereof, in accordance with Articles 12 to 14 of the GDPR Regulation. The accredited laboratory shall ensure at least that each analysis report or sampling report contains either a privacy statement containing the necessary data or a clear and precise reference to a web address where the necessary data can be found.

3 GPS DATA

The GPS data logger to be used for determining the GPS data at the time of sampling shall be capable of being installed and disabled by the sampler himself, so that the sampler is not continuously monitored and the sampler himself must actively and consciously switch on the GPS data logger.

The GPS data logos should only be used to verify the time of sampling and to verify the place of sampling and the sampling pattern.

The accredited laboratory designates a limited number of staff who can transfer the GPS data logbooks of the samples carried out to the Mestbank via SMIL. The sampling officer transmits the GPS data logbooks of the samples he has carried out to the persons designated by the accredited laboratory. Only those responsible within the accredited laboratory have access to GPS data logos.

To be annexed to the Ministerial Decree of.... 2026 approving the compendium of sampling and analysis methods under the Fertiliser Decree (BAM).

Brussels, (date).

The Flemish Minister for Environment and Agriculture,

JO Brouns