ACCREDITATION PROGRAMME
BUILDING MATERIALS DECREE

SECTION: SAMPLE PRE-TREATMENT

AP04 - V

**This document, AP04-A, version 3,
was adopted on March 3, 2005 by the Soil Management
Accreditation Board, incorporated in
Foundation Infrastructure for Quality Assurance of Soil Management
(SIKB) in Gouda**

sikb/documents/05.9069

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Contents of Sample Pre-Treatment (V)

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Right of ownership
This document was commissioned by the Foundation Infrastructure for Quality Assurance of Soil Management (SIKB), PO Box 420, NL-2800 AK Gouda, The Netherlands. The contents of this document are managed by the Soil Management Accreditation Board, incorporated in the SIKB in Gouda. The current version of the Accreditation Programme is available on the SIKB website and has been electronically protected against unapproved alterations. It is not permissible to introduce alterations to the original texts that have been approved and adopted by the Soil Management Accreditation Board in order to derive rights thereof.

Indemnification
With the exception of intentional acts or gross negligence, the SIKB cannot be held liable for losses incurred by the accreditation institution or third parties originating from the application of this Accreditation Programme and the use of this accreditation regulation.

How to order
An electronic version of this document is freely available from the SIKB website: www.sikb.nl. Bound copies of this document can be purchased from the SIKB, PO Box 420, NL-2800 AK Gouda, The Netherlands, E-mail: info@sikb.nl, fax: 0182-540676.

Update service
Alterations to this document approved by the Soil Management Accreditation Board can be obtained from the SIKB by registering via www.sikb.nl. Requests to receive the regular newsletter per post can also be submitted to the SIKB at info@sikb.nl.

Helpdesk / Instructions for use
Please contact your accreditation institution or the SIKB if you have any questions relating to the contents and/or application. See the grievances and dispute regulations in this document for information about disputes.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V1 Introduction

The Accreditation Programme "Building Materials Decree, section sample pre-treatment" (hereafter called AP04-V), describes the pre-treatment tasks that are applied in investigations within the scope of the Building Materials Decree. The quality requirements and criteria these tasks must meet are also defined.

Reference point
The nationally and internationally standardised tasks that are applied in investigations within the scope of the Building Materials Decree are reference points for the Accreditation Programme AP04-V. In conformance with the standardised regulations, a number of tasks are strictly regulated. If a task is not executed in conformance with the defined national and international regulations, the equivalence of the measuring method has to be proven.

Method of recording sample pre-treatment / terminology used
In order to achieve the highest possible degree of clarity regarding the quality, the sample pre-treatment steps have been laid down in this Accreditation Programme. This is in contrast with – for example - analyses, in which various analysis methods can be applied, based on quantified quality requirements (performance characteristics). In order to clarify this difference in definition used in the Accreditation Programme, the decision has been made to adopt a slightly different terminology. The requirements for the sample pre-treatment sections are recorded on so-called "instruction sheets", and the requirements for the sections that can be quantified have been laid down in performance sheets". Moreover, the sample pre-treatment activities are not referred to as "performances", but as "tasks". Alternative terms are also used for quality monitoring (see section V3.5).

Transparent quality
The quality of the sample pre-treatment activities can only be laid down as a description, because the methods of sample pre-treatment have not been validated. The sample pre-treatment methods as described here are a more detailed elaboration compared to the sample pre-treatment methods defined in the standards and the supplementary regulations to these in (the Execution regulations of) the Building Materials Decree. These direct references not only indicate, which standard must be applied under a number of specific circumstances, they also determine in which way the quality requirements set within the scope of the Building Materials Decree must be met. In order to render this quality as transparent as possible, in addition to the standards and enforcement protocols of the Building Materials Decree, a number of quality monitoring points have been included in the procedures.

Accreditation of sample pre-treatment
The sample pre-treatment consists of activities (tasks) that are in itself difficult to test quantitatively. For this reason, the Accreditation Board has adopted the position that generally sample pre-treatment must be accredited in connection with other, more verifiable activities, such as composition- and/or leaching analysis.
Within the scope of the Accreditation Programme AP04, sample pre-treatment can be accredited separately if need be, provided that with the aid of additional quality monitoring measures it can be proven to be a controlled process. This includes the possibility to establish the identity of all samples and sub-samples and the taking of measures to prevent cross contamination between the various samples.

Training
Due to the (currently) very limited possibility to define the quality of the sample pre-treatment on the basis of numerical quality criteria, the actual performance of the sample pre-treatment has a significant influence on the achieved quality.
A number of quality monitoring points have been incorporated in order to achieve the desired quality level. As a rule, one of the most important aspects of quality monitoring is

the education level of the executor. However, (currently) there is no specific training course available; consequently, in this Accreditation Programme, no requirements can be set regarding the training level.

Different situations
On account of the fact that in this section of the Building Materials Decree Accreditation Programme a descriptive definition of the sample pre-treatment had to be adopted, the definition of the method of sample pre-treatment, given a number of specific conditions, has been restricted to minimum requirements. This implies that it in a number of cases it will be possible to make a qualitative improvement on the performance of the sample pre-treatment, resulting in more representative[1] sub- and analysis samples.

References
For the performance of the sample pre-treatment in accordance with the Accreditation Programme it is crucial to know the content of the NEN-standards and protocols referred to. At the same time, sample pre-treatment must be applied in direct connection with the other sections of the Accreditation Programme Building Materials Decree.

The tasks that fall under the Accreditation Programme are given in chapter V2 of this document. In chapter V3, the concepts and parameters used are defined and in chapter V4 the validation of an activity is represented by generally adopted procedures. Chapter V5 described the minimum quality monitoring to be applied during the performance of activities falling under this Accreditation Programme. The technical data of the activities and the related performance characteristics are given in chapter V6.

INSTRUMENTEN VOOR EENVOUDIGER EN BETER BODEMBEHEER
SIKB

[1] *more representative means that the statistical distribution of measurement results from potential analysis samples of equal size (size/volume) is reduced when the quality of the sample pre-treatment is increased.*

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V2 Overview of tasks

This section of the Accreditation Programme includes all "operations" necessary for the pre-treatment of samples for the purpose of composition and leaching analysis within the scope of the "Building Materials Decree". The sample pre-treatment as such is not a separate task, but it is part of the process. In this respect it is more accurate to speak of an operation. The operations included in this section of the Accreditation Programme must always be performed in combination with other sections (composition, leaching). The operations are therefore, (if relevant), included in the package classifications for the specifications of Soil composition, Building materials composition, and Leaching analysis (see AP04-A).

The following operations are described in AP04-V:

- Sub-sampling through quartering
- Sub-sampling through static split distribution
- Sub-sampling through rotatory distribution
- Sub-sampling through core sampling
- Manual sub-sampling
- Reduction < 4 mm.
- Reduction < 1 mm and < 0.5 mm
- Reduction < 0.125 mm
- Drying of materials
- Siphoning off free water

Two additional supportive activities are also defined:

- Packaging of materials
- Storage and preservation of materials

Remark: *The operations mentioned above have been drawn up for soil and building materials.* ***Bituminous materials*** *are an exception to this. Suitable sample pre-treatment regulations for the determination of organic components in materials that contain bitumen are included in draft--NEN 7331.*

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V3 Concepts/parameters

A number of concepts and terms are regularly used in the Accreditation Programme.
The concepts and terms are defined below in order to prevent confusion.
Definitions and testing procedures are as much as possible in keeping with the standards generally applied in The Netherlands, NEN 7777 and the Implementing regulations of the Building Materials Decree. In part, these refer to the definitions included in NEN 7360 and draft--NEN 5709.

V3.1 Material properties

Soil (Building Materials Decree)
Non-moulded building material with a solid structure, of natural origin, not manmade, which may constitute part of the Dutch soil.

Building material (Building Materials Decree)
Material in the quality in which it is intended for use in a construction project, in which the total contents of silicon, calcium or aluminium are more than 10% (m/m) of that material.

Moulded building material (Building Materials Decree)
Building material of which the smallest unit has a volume of at least 50 cm^3 and which under normal conditions retains its shape permanently.

Non-moulded building materials (Building Materials Decree)
Building materials, not being moulded building materials, nor soil.

Bitumen (draft--NEN 7331)
A very viscous liquid or solid substance, primarily consisting of hydrocarbons or derivatives thereof, which is almost completely dissoluble in carbon sulphide.

Asphalt (draft--NEN 7331)
A natural or artificial mixture of bitumen and mineral substances.

V3.2 Samples

Batch (NEN 7360)
The quantity of material which for the purpose of sample pre-treatment is regarded as a unit.

Sample (NEN 7360, draft- NEN 5709)
The quantity of material which for the purpose of sample pre-treatment is regarded as a unit.

Grip (draft--NEN 5709)
The quantity of material that is removed from a batch in one go, but which is combined with other grips into a mixture sample for the purpose of analysis.

Mixture sample (draft--NEN 5709)
The quantity of material created by combining various grips, in which the identity of the original grips has been lost by blending.

Sub-sample (NEN 7360, draft-NEN 5709)
Part of a sample, which is regarded as representative for the whole sample.

Core sample (NEN 7360)
The quantity of material that has been taken from a sample in one go.

Minimum grip- and sample size (NEN 7360, draft-NEN 5709)
The minimum grip size or the minimum sample size of a granular material respectively which during sampling must be taken in order for the sample to remain representative.

Analysis sample (draft- NEN -5709)
A sub-sample obtained through the prescribed sample pre-treatment method that is used in its entirety for an analysis.

V3.3 Sample pre-treatment

Sample pre-treatment (draft-NEN 5709)
The total of activities - such as breaking, grinding, mixing and splitting - required to obtain a sub-sample from a sample.

Pre-reduction (NEN 7360)
The first action when reducing, if the sample contains parts that are larger than the input size of the mechanical break- or reduction equipment.

Reduction (NEN 7360)
The process in which the grain size or piece size of the sample or sub-sample is reduced.

Distribution (NEN 7360)
The process in which the sample is divided into representative sub-samples or analysis samples.

Heterogeneously divided contaminated substance (draft-NEN 5709)
A contaminated substance, the concentration of which is characterised by little to a lot of variation on the sampling scale.

Homogenously divided contaminated substance (draft-NEN 5709)
A contaminated substance, the concentration of which is characterised by no to little variation on the sampling scale.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V3.4 Substance properties

Volatile compounds (draft-NEN 5709)
The group of substances with a boiling point < 300°C (at a pressure of 101 kPa)

Remarks:

1. The volatile aromatic hydrocarbons benzene, toluene, ethyl benzene and xylenes (BTEX), and the volatile halogenated hydrocarbons fall under the group of organic substances. Furthermore, from the perspective of sample pre-treatment, chlorophenols (because of the mono– to tetrachlorophenols inclusively), the volatile chlorobenzenes (mono- and dichlorobenzenes) and cyanides (because of a number of volatile cyanides such as hydrogen cyanide) also fall under this group of substances.
2. In principle it is best to connect the distinction between volatile and lightly-volatile compounds to the vapour tension. Since the vapour tension is known of only a limited number of substances, and due to the relationship between vapour tension and boiling point, the boiling point has been chosen as the distinctive criterion.
3. Naphthalene also falls under this group of substances, but if naphthalene is determined as PAH, sample pre-treatment is performed analogous to the sample pre-treatment if lightly-volatile compounds.

Lightly-volatile compounds (draft-NEN 5709)
The group of substances with a boiling point between 300°C and approx. 500 °C (at a pressure of 101 kPa).

Remark:
The following groups fall under this category:

- Mineral oil;
- Polycyclic aromatic hydrocarbons (PAH);
- Polychlorobiphenyls (PCB);
- Organochloride pesticides (OCP);
- Extractable organohalogen compounds (EOX);
- Chlorobenzene (tri-, tetra-, penta- and hexachlorobenzene).

Non-volatile compounds (draft-NEN 5709)
The group of substances with a boiling point higher than approx. 500 °C (at a pressure of 101 kPa).

Remark:
The following groups fall under this category:

- Elements, including: arsenic, barium, cadmium, cobalt, chrome, copper, molybdenum, nickel, lead, antimony, selenium, tin, vanadium and zinc; and mercury compounds.
- Oxi-anions, including: sulphate;
- Halogenides, including: bromide, chloride, and fluoride.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V3.5 Quality monitoring

As mentioned in chapter V1, it is difficult to test the sample pre-treatment activities quantitatively. This means that it is difficult to establish numerical performance characteristics for the quality of the performance. The quality must be "monitored", but it cannot be "assured". Therefore, in respect of quality control, in this section not the usual but a different terminology is used. All the same, two levels of quality monitoring are referred to. These are:

Process control
Checks executed by the very persons that perform the analysis. This means on the one hand, that the activities performed must be recorded unambiguously and on the other that the checks must focus is on the requirements and equipment applied.

Independent internal quality assessment
Checks of the (quality of the) performance of the activities by an independent expert from within the institute, and (if provided) of the requirements for the training level of the person(s) performing the activity.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V4 Validation of a task

An intra-laboratory validation investigation must be performed if a new task is introduced or an existing task is altered. The validation investigation must prove that the various activities can be executed in conformance with the set requirements.

The sample pre-treatment does not result in numbers that must meet quality requirements. Due to this, a simple validation which involves only the sample pre-treatment method is not possible.

The quality of the sample pre-treatment can only be laid down in a description due to the fact that the sample pre-treatment methods cannot be validated. As a rule, the sample pre-treatment must be executed in conformance with NEN 7310 to and including NVN 7313. Regarding the inorganic substances, the tasks of sample pre-treatment has been laid down in NVN 7312, regarding the organic substances in NVN 7313. Furthermore, supplementary criteria have been laid down for sample pre-treatment in the enforcement protocol for non-contaminated soil and the enforcement protocol for building materials of the implementation regulation of the Building Materials Decree.

V4.1 Testing procedure

During the "validation" of the sample-pre-treatment, an institute must be able to prove that the activities can be performed. The validation must take place in conformance with the specifications in this section of the Accreditation Programme. To this end, an institute must execute the sample pre-treatment in 10—fold, and record all focus points for process control and independent internal quality evaluation. The performance of the actual analyses (composition, leaching analysis) does not fall under this validation.

Depending on the package to be accredited, the "validation investigation" must be executed on the matrixes of table 1. The analysis samples mentioned must be prepared for this. The sample quantity for the validation investigation must be equal to the sample quantity mentioned in the performance sheets. The division of samples at the sampling location does not fall under the validation activities.

Remark: *As batches larger than 20 kg are (can be) divided at the sampling site, the maximum batch size in the validation investigation is 20 kg.*

Table 1 Matrix of validation samples

Material	Material/sample properties	Pre-treatment steps for
Granulate	D_{95} > 4 mm	sub-sample counter appraisal lightly-volatile compounds column test sample storage
Non-contaminated soil	sample size ☐ 9 kg	sub-sample counter appraisal volatile compounds lightly-volatile compounds dry matter, clay, and organic substance inorganic substances sample storage
Contaminated soil	10% > clay > 3% D_{95} > 1 mm	volatile compounds column test sample storage
Cement-bound test piece	Test piece volume > 500 ml cement content > 10%	Lightly-volatile compounds dry matter availability test column test

Remark: *The building materials for the "validation investigation" may originate from the same batch, but they must be sampled separately.*

V4.1.1 Material loss

In the validation investigation, the institution must prove that the requirements set in respect of material losses are not exceeded. The most important causes of loss of material are:
- residual material in the equipment;
- dispersion of the finer fraction of the material particularly.

Criterion:
* The material loss must meet the requirements set in the instruction sheets.

Explanation: *The material used up during "pre-rinsing" the equipment is not regarded as loss of material.*

V4.1.2 Degree of sample size equality

During the validation-investigation, the institution must prove the correct performance of sub-sampling and sample distribution (core sampling, quartering, static split distribution, and rotatory distribution). The method of each activity must be recorded.

Criterion:
* The distribution must comply with the requirements set out in the instruction sheets.

V4.1.3 Contamination during reduction

By pre-treating and preparing washed gravel in the same way as "blank" matrix material, the possible emissions of the elements from the grinding equipment can be determined. To determine these emissions, the same washed gravel is also reduced by means of a mortar or ball mill with agate interior; the contents of the sample obtained by latter method of reduction are considered as original contents of the washed gravel, on which basis the emissions from the grinding equipment can be calculated.
The emission of all elements to be determined in samples must be established in the contamination investigation. The analyses must be executed by a laboratory which is AP04-accredited for these elements.

[1)] Instead of washed gravel, a material may be used which in respect of the emission of elements from grinding equipment is representative for the most extreme samples a

laboratory pre-treats within the scope of AP04 – V. The element concentrations in this material must be sufficiently low for significant testing on the criterion below.

The investigation into the contamination during reduction must be executed in quadruplicate at the very least.

Criterion:

* The increase in the element concentration through contamination may not exceed on average of 25% of the target value for standardised silt (25%) and organic matter content (10%). The maximum increase is numerically represented in table 2. The required detection levels for the determination of the elements in soil have been taken into account (see AP04-SG).

Table 2 Maximum permitted emission from grinding equipment

	Maximum increase (mg/kg)		Maximum increase (mg/kg)		Maximum increase (mg/kg)
Sb	4, 4 (= DL_{req})	Co	5	Ni	9
As	7	Cu	9	Se	10 (=DL_{req})
Ba	50	Hg	0.08	Sn	6 (=DL_{req})
Cd	0.5 (=DL_{req})	Pb	21	V	10
Cr	25	Mo	2.5	Zn	35

Remark: *Contamination primarily occurs through intensive contact between sample and reduction equipment. The contamination during crushing by using manganese steel is negligible.*

V4.2 Execution of the validation activities

V4.2.1 Validation plan

A validation plan must be drawn up for the validation activities. The validation plan must be provided with identification and it must be applied in all parts of the investigation. The validation must be carried out according to the validation plan, which is drawn up before the start of the activities.
The validation plan must contain the following elements at the very least:

- investigation leader;
- the nature and the objective of the validation;
- the date on which the validation plan was established;
- the method and the activities;
- the statistical methods that will be applied for the analysis of the results;
- the methods and techniques that will be used.

Explanation: *If required, the investigation leader may adjust the desired validation or reformulate the set objective/method.*

V4.2.2 Validation report

The validation investigation is rounded off with a validation report. The conclusion of the investigation must be relevant in relation to the validation plan.

V4.2.3 Archiving

During the accreditation investigation, complete documentation must be available for each validation investigation performed.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V5 Quality monitoring of a task

In quality monitoring, two levels can be distinguished: Process Control and Independent Internal Quality Monitoring. These two levels are defined in V3.5.

For the two levels mentioned above, the quality monitoring of the institution must be laid down in separate documents. These must contain the following aspects:
- dealing with out-of-control situations;
- use or preparation of (control) samples;
- responsibilities of the staff.

The two defined levels of quality monitoring do not present additional forms of quality monitoring, but define the minimum form of quality monitoring that must be applied.

V5.1 Process control

Quality monitoring within the scope of process control consists of regular focus points, periodic focus points, facilities at the location/institution and suitability of the equipment. When the focus points are carried out, the compliance of the activity with the data given in the instruction sheets must be investigated.

V5.1.1 Regular focus points

A focus point during the execution of each task is the recording of the separate sample pre-treatment stages for all samples that are treated.

Explanation: *Due to the lack of statistical key figures for sample pre-treatment, high standards are set for recording the activities performed, in order to render the quality of sample pre-treatment traceable. Therefore, each separate stage of sample pre-treatment must be recorded for all samples prepaired in accordance with this Accreditation Programme.*

SG5.1.1.1 Core size

The core size is subject to the grain size of the building material. Sub-sampling using coring methods is executed for sub-sampling for the column test of contaminated soil and on all building materials < 4 mm (D95) for the determination of volatile substances, lightly-volatile substances and physical parameters.

Criteria:
* Building materials larger than 3 mm: the width, height, and length of the sample scoop must be at least equal to three times the maximum grain size (D95) of the building material to be sampled.
* Building materials smaller than 3 mm: the width, height, and length of the sample scoop must be at least equal to ten times the maximum grain size (D95) of the building material to be sampled.
* For a representative sub-sampling from a batch by means of coring, at least 10 cores must be taken for each sub-sample.

SG5.1.1.2 Distribution

Requirements have been set regarding the spread in the distribution for quartering, static split distribution, and rotatory distribution. This distribution must be checked at each stage for each sample.

Criterion:
* The distribution must meet the requirements set out in the instruction sheets. In the event of a breach of this requirement, the sub-samples must be combined and the distribution procedure must be repeated.

Static split distribution is carried out on "field moist" samples. The performance of the distribution is right, if all grains have an equal chance of ending up in the sub-sample. The cohesive behaviour of the sample should not have a negative impact on the distribution process. If during a static distribution of the sample the set requirements are not being met twice, no representative sample can be obtained by means of static distribution. Then, the sample must be dried and divided by rotation in order to obtain a representative sub-sample.

Explanation: *From practical considerations, no quantitative requirements have been set for distribution a sample greater than 20 kg. The execution must be qualitatively defined.*

V5.1.2 Periodical focus points

SG5.1.2.1 Grain size distribution

The focus points for process control of the grain size distribution include that a sieve is used to determine the m/m percentage that meets the requirements of the reduction stage. After reduction of the grain size to < 4 mm, < 1 mm, < 0.5 and < 0.125 mm, the check is performed on 1 in 20 samples, with a minimum of 1 a month and a maximum of 1 a week.

The criteria set for the sample pre-treatment include a reasonably reliable estimation of the maximum grain size; a precise determination is, however, *not* necessary.

Regarding this quality level, it may also be laid down, that in certain situations the determination of the D_{95} is not necessary. This is the case if:

- the material to be sampled can unambiguously be defined (the type of material is known);
- the relevant type of material has at least ten times been analysed previously;
- these previous investigations showed that the material had an (almost) equal maximum grain size (± 5 %);
- the calculation of the minimum grip- and sample size is based on a safe D_{95}, namely a D_{95} that is one mesh-width larger than the widest D_{95} determined during the previous investigations.

Materials that are supplied according to the grain size specification do not have to meet the above mentioned specifications, and the determination of D_{95} can be left out. This applies to a number of building materials, for which on the basis of its technical specifications, the maximum grain size of the relevant material is known in advance. For such materials it is sufficient to maintain this value in order to determine the minimum grip- and sample size, unless there are indications during sampling that the maximum grain size of the batch does nevertheless exceed this value (more than 5 m/m%). In this case, the D_{95} must be determined.

The grain size distribution is checked by means of dry sieving in conformance with draft-NEN 5753 (excluding dissolution) with a sieve with a mesh width of the prescribed size.

Criterion:

* The mass percentage (m/m %) of residual material on the sieve with the agreed mesh-width must be less than 5 %.

SG5.1.2.2 Loss of material

The material loss of 1 in 20 samples must be determined at least once a month and once a week the most. The performance and the criteria are described in V4.1.1.

SG5.1.2.3 Contamination of reduction equipment

Based on existing grinding tools, the emission of the grinding equipment must be determined for the relevant parameters either biannually or when replacing parts of the grinding tools. Relevant parameters are parameters for which the validation investigation shows an increase in concentration. The execution and the criteria are described in V4.1.3.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V5.2 Independent internal quality investigation

V5.2.1 Internal audit

The laboratory must assess the tasks at least once every six-months. These audits should be (preferably) unannounced.

The independent internal quality investigation, during which the institute periodically includes an "in-house" control material, appears to be of no additional value, due to its lack of performance characteristics and its interrelation with the analysis. Independent checks of the tasks may from time to time be performed by another person from the firm in question. This person must have both a technical and contextual understanding of matters relating to sample pre-treatment. Focus points for an independent internal quality assessment are:

- focus points for the checking criteria for process control over the previous period;
- correct performance of the prescribed procedure;
- recording the activities performed;
- cleaning the equipment c.q. checking potential contamination;
- sample storage and storage conditions;
- training of personnel and accommodation.

If the findings of the independent internal quality investigation are not in line with the requirements set for process control, the institution must undertake corrective measures.

V5.2.2 Grain distribution

At least once every quarter, the laboratory must investigate this activity unannounced. The procedural aspects must be included in a separate procedure. For the independent internal quality investigation of the grain distribution, a sieve is used to determine the m/m percentage that meets the requirements of the reduction stage. After reduction to < 4 mm, < 1 mm, and < 0.125 mm, a check is performed.
The execution and the criteria are laid down in V5.1.2.1.

If the findings of the independent internal quality investigation are not in line with the requirements of the instruction sheets that have been set for the process control, the institution must undertake corrective measures.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6 Technical description and quality monitoring of activities

V6.1 Introduction

The type of sample preparation to be executed depends on the test or determination to be performed on the building material/soil and on the question, according to which investigation protocol (or method) the soil/building material must be investigated. The organic composition must be determined for all building materials that are analysed in accordance with the building materials enforcement protocol. For soil analysed in conformance with the enforcement protocol for non-contaminated soil, the inorganic composition must additionally be determined. The inorganic composition must also be determined for soil that does not fall under the non-contaminated soil category. If the inorganic composition lies within a set course, the leaching behaviour of these substances must be determined with the column test. For the remaining building materials, the inorganic composition is not the criterion for determining the leaching properties.

The set requirements for sample pre-treatment of the analyses for inorganic substances, the analysis of organic substances and leaching analysis are different. Therefore, the stages of sample pre-treatment can only be partially applied for multiple purposes. Usually, the sample pre-treatment for the various determinations must be executed separately.

Sample pre-treatment starts immediately after sampling. For sample pre-treatment, the activities include packaging, sample storage and sample preservation and sample transfer. Because of this, part of the activities that fall under sample pre-treatment will be executed by the institution that takes care of the sampling.

There are two main objectives of sample pre-treatment:
- obtaining material with the correct grain size for the test or analysis;
- obtaining a representative (sub-)sample from the sample obtained during sampling.

Splitting the sample into sub-samples must be executed with the methods included in the diagrams or a qualitatively better method, if this is possible with the given material properties (moisture content in particular).

Often, the sample grain size will not meet the requirements set for the composition- and leaching analysis. The minimum sample size to be worked with during the sample pre-treatment is determined on the basis of table 3, unless deviation from it is expressly stated. If the sample does not meet the maximum grain size laid down for the specific test or determination, the sample must be reduced to the required maximum grain size.

Remark: *The grain size for the determination of volatile and lightly-volatile substances may deviate from table 3. In case the grain size is smaller, the sample size laid down in the instruction sheets c.q. the performance sheets is applied. If the grain size is larger, the sample size in table 3 must be used.*

Table 3 Relationship between sample size and grain size (NVN 7312)

Grain size (mm)	Minimum sample quantity (g)
< 0.125	5*
0.25	50
0.50	100
0.71	250
1	500
2	1,000
4	2,000
8	3,000
16	5,000
32	10,000
64	20,000

* deviating from NVN 7312

The division of samples into sub-samples can be executed in different ways. The usual techniques are quartering, static split distribution and rotatory distribution. On account of qualitative considerations, rotatory distribution is preferred to static split distribution, and static split distribution to quartering. Dividing the sample into sub-samples must be carried out with methods included in the diagrams or, if possible, a qualitatively better method with the given material properties (moisture content in particular).

In this Accreditation Programme, the sample pre-treatment is divided into different paragraphs. The sub-division is made on the basis of the physical characteristics of the building materials in connection with practical feasibility and the influence on the analysis methods. **Bituminous materials** are an exception to this. Suitable sample pre-treatment techniques for the determination of organic components in materials that contain bitumen are included in draft-NEN 7331.

The following classification has been made:
- sample pre-treatment of soil and sludge (diagram § V6.2.3)
- sample pre-treatment of non-moulded building materials (diagram § V6.3.3)
- sample pre-treatment monoliths and moulded building materials (diagram § V6.4.3)

References to diagrams
In these paragraphs, for the relevant group of materials a decision diagram has been drawn up for the tasks to be executed. In the diagram, for each task reference is made to the instruction sheets
(§ 6.5). It is recommended that the diagrams are passed through for each separate determination (composition analyses and/or leaching), starting from sampling. The diagrams differ in certain respects from the diagrams in the enforcement protocols.

Within the scope of enforcement, it may be necessary to immediately split-off a sub-sample for counter appraisal. This option has been included in the diagrams.

The substances that are methodologically treated in the same way are combined in the diagrams. The sample pre-treatment depends (primarily) on the physical properties of the parameters to be determined. The following representatives have been included in the diagrams:

Volatile substances:
- determination of BTEX and VOH
- determination of parameters for the research protocol
- determination of chlorophenols (soil)
- determination of cyanide (soil)
- determination of chlorobenzenes (mono- and dichlorobenzenes)

Non-polar moderately volatile substances:
- determination of PAHs
- determination of PCBs and OCBs
- determination of mineral oil
- determination of organonitrigen compounds
- determination of EOX
- determination of chlorobenzene (tri-, tetra-, penta- and hexachlorobenzene)

Macro-parameters:
- determination of dry matter
- determination of pH (soil)
- determination of silt (soil)
- determination of organic matter (soil)

Inorganic composition:
- determination of metals (soil)
- determination of fluoride, chloride and bromide (soil)
- determination of non-volatile mercury

V6.2 Sample pre-treatment of soil and sludge

V6.2.1 Definition of soil and sludge

The sample pre-treatment for soil and sludge is described separately from the other non-moulded building materials. If soil consists for more than 5% of parts > 4 mm, it is pre-treated according to the sample pre-treatment of non-moulded building materials. After testing, soil can be sub-divided into non-contaminated soil, lightly contaminated soil (category 1 and 2), and highly contaminated soil. Establishing the soil to be non-contaminated occurs on the basis of the protocol for non-contaminated soil, in which the composition of organic and inorganic substances in the soil is investigated. Lightly contaminated soil for use as category 1 or 2 soil in construction projects is analysed for the organic substances composition and the leaching properties of inorganic substances in conformance with the user or enforcement building materials protocol.

Explanation: *This subdivision is based on the required composition analysis for inorganic substances on the one hand, and their cohesive behaviour on the other, which is often different from the other non-moulded building materials. Other sub-sampling methods are (sometimes) prescribed on account of this.*

V6.2.2 Explanation of the diagrams for soil and sludge

A diagram has been drawn up for soil (non-contaminated and contaminated) and sludges (§ V6.2.3).

For soil and sludges, the following analysis samples and sub-samples are taken:

1) four separate sub-samples for the determination of volatile organic substances, and cyanide;
2) sub-sample for the determination of non-polar moderately-volatile organic substances;
3) two sub-samples for the determination of pH, dry matter content, silt content, and organic matter content;
4) sub-sample for the determination of the leaching behaviour;
5) remaining material is used for the analysis sample for metals, including non-volatile mercury, and inorganic substances, excluding cyanides, and a reserve sample.

The diagram shows the general relationship of a sample after sampling, between the various stages of sample pre-treatment and the analyses. For all separate analyses, the diagram must be passed through from the starting point. The numbers near the tasks in the diagram refer to the relevant instruction sheets.

After sub-sampling, the requirements of coding, packaging and storage are applicable for all separate sub-samples. The unused samples can be joined together and disposed of. At least one representative sub-sample must be saved for potential repeat analysis.

As a rule, the sample pre-treatment will be carried out in a laboratory. For sample quantities larger than 20 kg, it may be necessary to perform part of the sample pre-treatment at the sampling site. In this way it is avoided that large quantities of building materials have to be transported as samples. The conditions of sample pre-treatment at the sampling location must be either equivalent to the criteria laid down in NEN-EN-ISO/IEC 17025, or must be carried out in conformance with the relevant protocols and the conditions stated in the instruction sheets.

The sample pre-treatment of volatile substances and polar moderately-volatile substances must be executed in quadruplicate. It is not possible to make mixture-samples. If the analysis method relates to a liquid extraction, a mixture-extract can be made by combining the different samples equivalently.

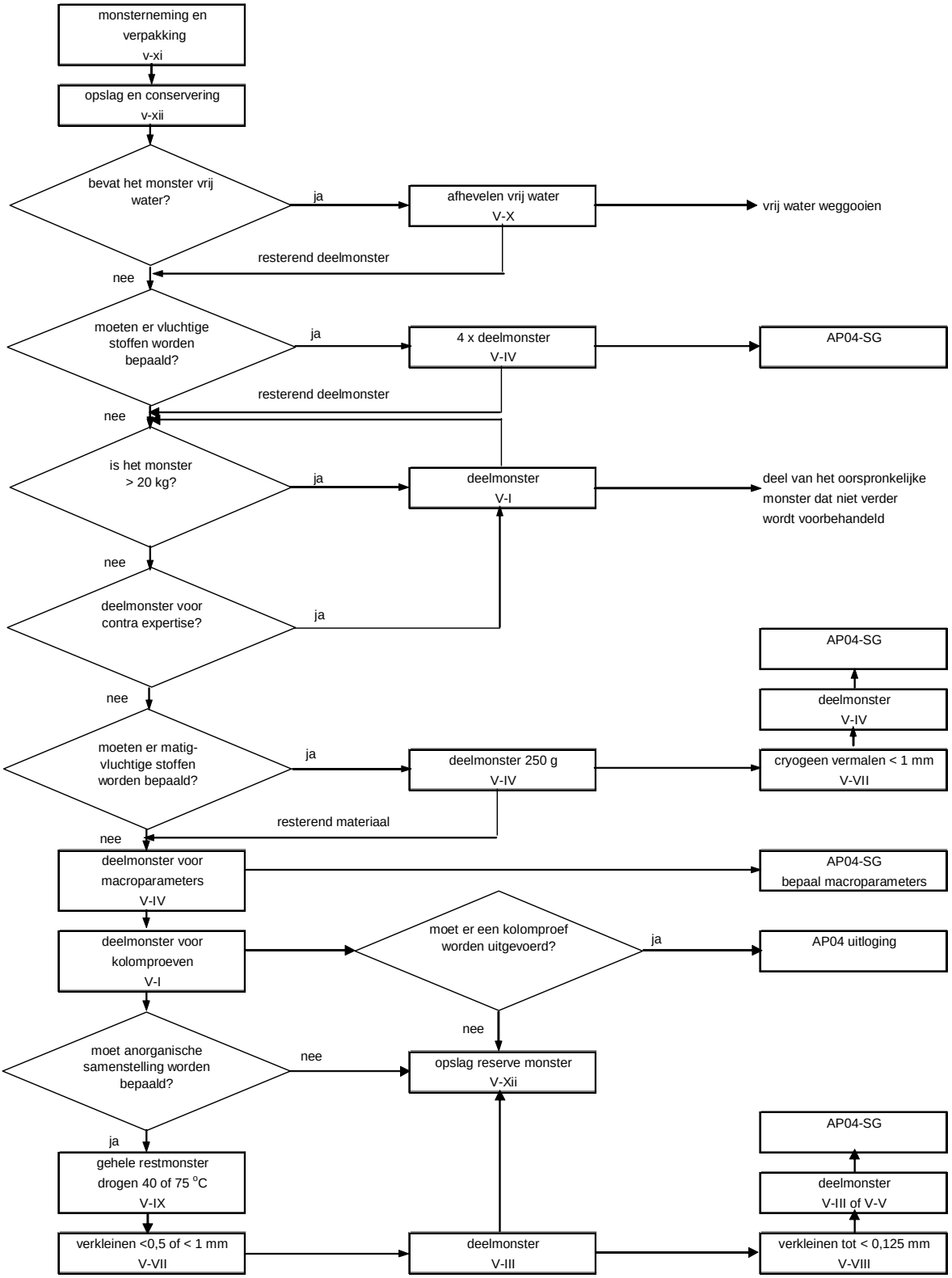
The leaching behaviour in soil may change if the sample is dried. This is why, **deviating** from the enforcement protocol for building materials, sub-sampling of soil and sludge for the leaching analysis is executed with a qualitatively inferior method (quartering), in order to prevent the leaching properties from being influenced.

Remark: *In the situation described, the importance of a representative sub-sampling is secondary to potential differences between the leaching behaviour of field-moist and dried soil.*

For the sample pre-treatment for the inorganic composition analysis, visually perceivable metal parts and other artefacts (plastic etc.) must be manually removed from the sample. The weight percentage must be determined. If the composition cannot be derived from the removed metals, the composition must be determined.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6.2.3 Diagram of soil and sludge



Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6.3 Sample pre-treatment of non-moulded building materials

V6.3.1 Definition of non-moulded building materials

The following building materials fall into the category of non-moulded building materials (NVN 7312):

- powdery building materials;
- almost powdery building materials;
- fine-grained building materials;
- coarse-grained building materials that can be pre-treated at room temperature;
- coarse-grained building materials that must be reduced cryogenically;

with the exception of soil and sludges (for sample pre-treatment of these non-moulded building materials, see § V6.2).

V6.3.2 Explanation of the diagram of non-moulded building materials

The diagram shows the general relationship between the various sample pre-treatment stages and the analyses. For all separate analyses, the diagram must be passed through from starting point. The numbers near the tasks in the diagram refer to the relevant instruction sheets.

For non-moulded building materials (including soil and sludges), the following analysis samples and sub-samples are taken:

1) four separate sub-samples for the determination of volatile organic substances, and polar moderately-volatile organic substances;
2) sub-sample for the determination of non-polar moderately-volatile organic substances;
3) two sub-samples for the determination of the dry matter content;
4) sub-sample for the determination of the leaching behaviour;
5) the remaining sample is treated in its entirety for obtaining a reserve sample.

After sub-sampling, the requirements of coding, packaging and storage are applicable for all separate sub-samples. The unused samples can be joined together and disposed of. At least one representative sub-sample must be saved for potential repeat analysis.

As a rule, the sample pre-treatment is carried out in a laboratory. For sample quantities larger than 20 kg, it may be necessary to perform part of the sample pre-treatment already at the sampling site. In this way it is avoided that large quantities of building materials have to be transported as samples. The conditions of sample pre-treatment at the sampling location must be equivalent to the conditions laid down in the instruction sheets.

Remark: *The sample quantity after sample pre-treatment at the sampling location is therefore between 10 and 20 kg.*

Sub-sampling for the determination of volatile substances must be performed without prior sample pre-treatment by means of core samples.

Remark: *In ANVM project 108/109, the problems regarding the representativeness of the sampling method for the determination of volatile substances have been investigated. Due to the occurrence of losses, the representativeness of building materials larger than 4 mm is considered insufficient. Whenever the concentration is low, the sample pre-treatment in itself will lead to the absence or near-absence of volatile substances. For technical reasons related to the execution of the activity, usually no volatile substances are determined in course-grained building materials. Nevertheless, the analysis can be useful, for instance when high concentrations are expected in account of contamination of the building material. If in this analysis volatile substances are detected, it may be assumed that the actual concentrations will be higher.*

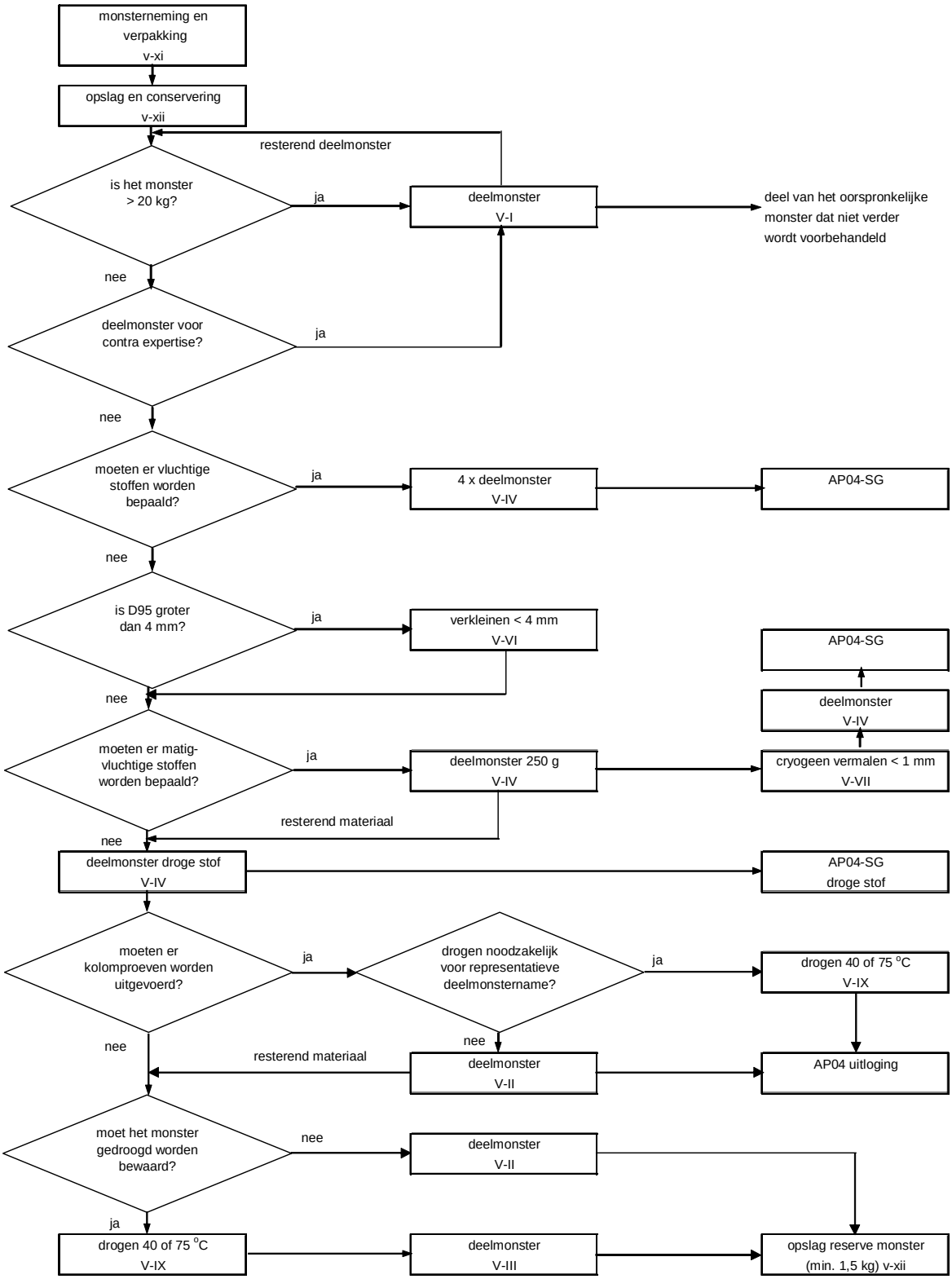
Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

The sample pre-treatment of volatile substances must be executed in quadruplicate. It is not possible to make mixture-samples. If the analysis method concerns a liquid extraction, a mixture-extract can be made by combining the various samples equivalently.

For the sub-sampling of moderately-volatile organic substances, building materials larger than 4 mm must be reduced previously in order to increase the representativeness of this sampling. In order to accomplish this, the building materials must be reduced in field-moist condition.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6.3.3 Diagram of non-moulded building materials



Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6.4 Sample pre-treatment of moulded building materials

V6.4.1 Definition of moulded building materials

The category of building materials consist of monoliths and moulded building materials, that must be pre-treated at room temperature or under cryogenic conditions.

V6.4.2 Explanation of the diagram for moulded building materials

Diagram V6.4.3 shows the general relationship between the various sample pre-treatment stages and the analyses. For all separate analyses, the diagram must be passed through from starting point. The numbers near the tasks in the diagram refer to the relevant instruction sheets, column organic substances.

For moulded building materials, the following analysis samples and sub-samples are taken:

1) four separate sub-samples for the determination of volatile organic substances, and polar moderately-volatile organic substances;
2) sub-sample for the determination of non-polar moderately-volatile organic substances;
3) sub-sample for the determination of the dry matter content;
4) sub-sample for the determination of the leaching behaviour by means of diffusion test[2];

5a) the remaining sample is treated in its entirety for obtaining a reserve sample.
5b) sub-sample for the determination of the leaching behaviour with the column test[3]
5c) sub-sample for the determination of the maximum availability.

After all necessary samples have been taken, the remaining sample is treated in its entirety for obtaining a reserve sample.
.
Preferably, the sample material is not dried before the leaching investigation, as drying can influence the leaching. If drying is required for the stage of division, it is permitted to dry the sample material at 40 °C or 75 °C (see instruction sheet V-IX). Subsequently, a sub-sample must be taken from the dried sample by means of rotatory distribution (see instruction sheet V-III).
NVN 7311 must be followed in order to determine whether it is necessary to dry the storage sample.

After sub-sampling, the requirements of coding, packaging and storage are applicable for all separate sub-samples. The unused samples can be joined together and disposed of. At least one representative sub-sample must be saved for potential repeat analysis

For the determination of volatile substances in moulded building materials is not possible to produce analysis samples directly from sub-samples without the previous execution of sample pre-treatment (reduction). The determination of volatile substances is only performed, if it is to be expected that relatively high concentrations of these substances are present in the building material.

[2] *The sampling for the diffusion test may be carried out before sub-sampling for composition analysis and/or availability test has taken place.*

[3] *If during the diffusion test the leaching behaviour is not checked regarding diffusion, it cannot be tested as a moulded building material. Testing against the Building Materials Decree subsequently takes place as a non-moulded building material, in which the leaching is determined with the column test.*

Remark: *In ANVM project 108/109, the problems relating the representativeness of the sampling method for the determination of volatile substances have been investigated. This is considered insufficient in moulded building materials. Whenever the concentration is low, the sample pre-treatment in itself will lead to the absence or near-absence of volatile substances. For technical reason related to the execution of the activity, usually no volatile substances are determined in moulded building materials. Nevertheless, in this case the analysis can be useful, for instance when high concentrations are expected in account of contamination of the building material. If in this analysis volatile substances are detected, it may be assumed that the actual concentrations will be higher.*

The sample pre-treatment for volatile substances must be performed in quadruplicate. It is not possible to make mixture samples. If the analysis method concerns a liquid extraction, a mixture extract can be made by combining the extracts of the different samples.

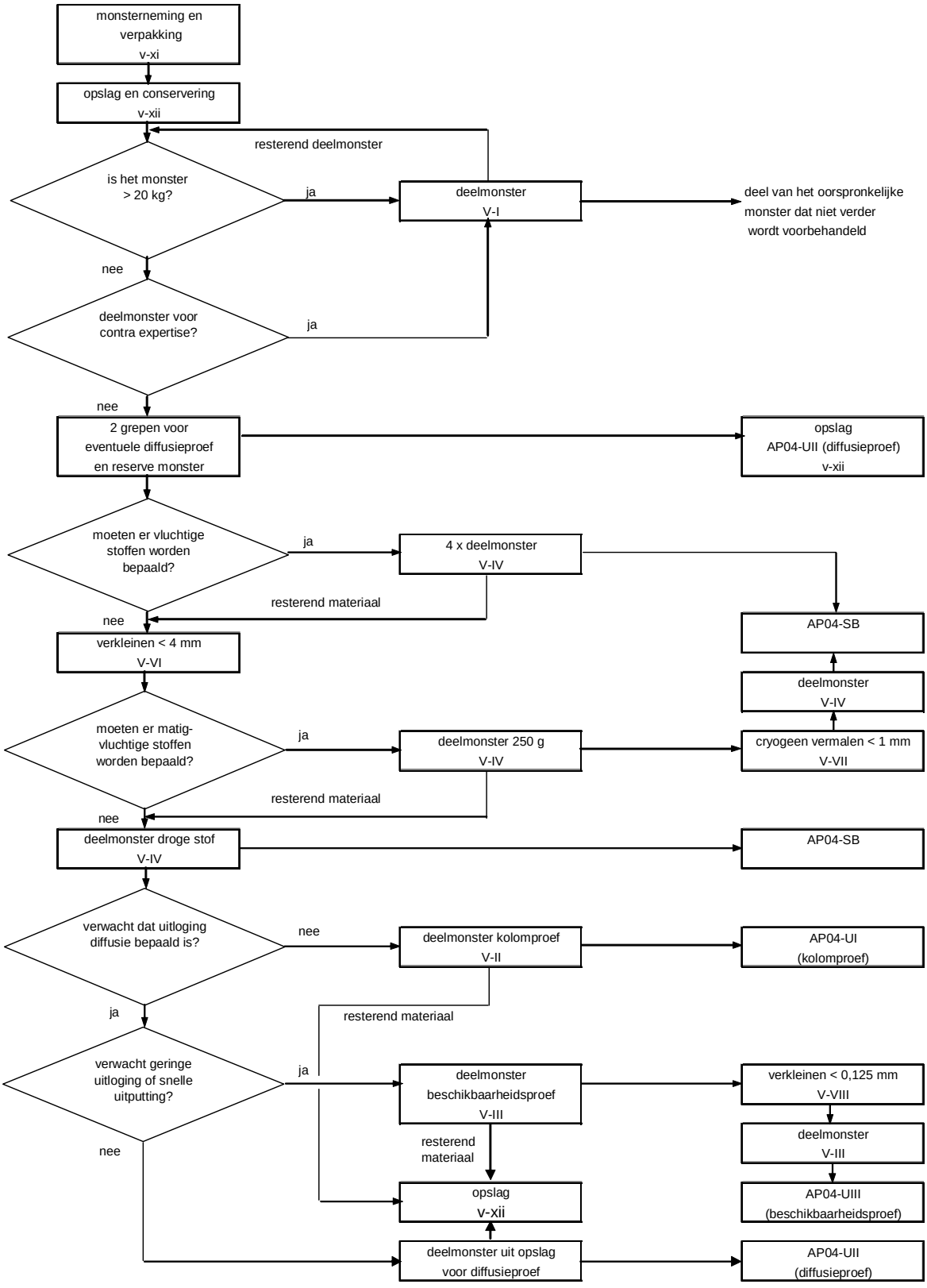
For the diffusion test, the sample must meet the set requirements regarding dimensions and form. Random samples for leaching analysis can be taken from the batch of moulded building materials before sub-samples for composition analysis and/or availability test are taken.

Remark: *Saws can be used for sub-sampling of moulded building materials. This method is less suitable, however, as water is used during the process. Reduction with a hammer or a chisel is not suitable for all samples, as the surface may get an irregular form.*

For the sub-sampling of moderately-volatile organic substances, building materials larger than 4 mm must be reduced previously in order that a sub-sample by means of core samples can be taken.
For moulded building materials, the leaching must either be determined by means of the diffusion test or the availability test. If it turns out that the leaching is not diffusion controlled, the leaching must be determined by means of the column test.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6.4.3 Diagram of moulded building materials



Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V6.5 Instruction sheets

The instruction sheets have been drawn up for each task and contain the references to the relevant NEN-standards and the Enforcement protocols for non-contaminated soil and building materials. Apart from these references, qualitative quality monitoring aspects have been included. Quantitative quality monitoring aspects are useful for the focus points for process control. The quantitative requirements regarding division must be checked for all sub-samples to be processed.

As 'packaging' and 'storage and preservation' are no 'tasks' but 'supportive activities', the numbers of these instruction sheets are rendered in minuscules (v-x and v-xi).
No explicit requirements have been set regarding the quality monitoring of these supportive activities. However, the activities must be incorporated in the institution's quality system.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.I Quartering

Principle

The objective of quartering is to obtain a representative sub-sample. Quartering is a manual division technique. Quartering is applied, if, given the conditions of the material (the moisture content in particular), rotary distribution and static split distribution do not lead to a representative distribution or if the sample is larger than 20 kg. The sample is homogenised as well as possible on a level, inert and clean substratum and is dispersed in a thin layer circle. The sample is divided into four equal parts. Two quarters opposite each other are removed, after which the two remaining quarters are combined into a sub-sample. This task must be repeated if required, until an analysis sample of the right size has been obtained.

Quartering is prescribed when reducing large samples (>20 kg). Given the nature of these activities, quartering is often carried out at the sampling site immediately after sampling.

Method for samples > 20 kg

Category	Tasks to be executed				Process control	
	Enforcement Protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	General	Requirement
Soil and sludge	n/a	-	§ 7.7.3	n/a	Equipment	Non-contaminated inert
Non-moulded building materials	n/a	-	§ 7.7.3	n/a	facilities	Dry Limiting free floating matter inert
Moulded building materials	n/a	-	n/a	n/a	Distribution	-

Method for samples < 20 kg

Category	Task to be executed				Process control	
	Enforcement protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	General	Requirement
Soil and sludge	n/a	-	§ 7.7.3	n/a	Equipment	Non-contaminated inert
Non-moulded building materials	n/a	-	§ 7.7.3	n/a	facilities	Dry Limiting free floating matter inert
Moulded building materials	n/a	-	n/a	n/a	Distribution	(50 ± 10)% per individual distribution stage

Process control

Distribution frequency: every sample

Remarks: *For practical reasons, no quantitative requirements are made for distributing a sample larger than 20 kg. However, the sub-sample must be obtained based on the principle of quartering, which means that the estimated size of the sub-samples must meet the (50± 10%) requirement. Sub-sampling by means of coring is not permitted (if quartering has been prescribed).*

If the sample quantity is smaller than 20 kg after a quartering action, and another quartering action must be performed, for the next steps, the requirements for the distribution must be met.

Distributing identical, moulded building materials must be executed with random grips, with which each specimen should have an equal chance to land in the sample.

If the distribution criterion is exceeded, both sub-samples must be combined again and the distribution procedure must be repeated.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.II Static split distribution

Principle

The objective of static split distribution is to obtain a representative sub-sample. Static split distribution is applied to 'field-moist' samples, for which the conditions for rotatory distribution for a representative distribution cannot be met without performing additional tasks. With static split distribution, a sample is split into two samples of equal size by spreading the sample across a splitter with a fixed split-width. One of the sub-samples obtained in this way is put to one side. The other sub-sample must be re-divided if necessary by means of static split distribution, until an analysis sample of the desired size has been obtained.

After the desired sub-sample has been obtained through static split distribution, the remaining sample material is joined.

Method

Category	Tasks to be executed				Process control	
	Enforcement protocol		Other protocol			
	Non-contaminated soil	Building materials	NVN 7312	NVN 7313	General	Requirement
Soil and sludge Non-moulded building materials Moulded building materials	n/a n/a n/a	§6.3.1.4 §6.3.1.4 §6.3.1.4	§ 7.7.1 § 7.7.1 § 7.7.1	n/a n/a n/a	equipment	Non-contaminated Split width > 3 * D_{95} Minimum of 8 splits per side
					facilities	dry Limiting free floating matter Inert
					loss of mass	< 10%
					distribution	(50 ± 10)% for each individual distribution step

Process control

Distribution frequency: all samples

Loss of mass: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

Remark: *Static split distribution is carried out on "field-moist" samples. The distribution is right, if all grains have an equal chance to land in the sample. The cohesive behaviour of the sample may not be detrimental to the distribution process. If this is the case, the sample must still be dried and subsequently subjected to rotatory distribution in order to obtain a representative sub-sample.*
If the criterion of distribution ((50± 10)%) has not been met, the distribution is non-representative.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.III Rotary distribution

Principle

The objective of rotatory distribution is to obtain a representative sub-sample. Rotatory distribution can be performed if the sample shows (almost) no cohesive behaviour. For this procedure, the samples must often be dried. For rotatory distribution, a sample is introduced into the opening of the distributor at a constant rate of material flow. By the rotating of a fixed number of receptacle vessels the sample is split into a number of identical samples. The material required can also be divided off by a variable – to be determined prior to the splitting - split-aperture with rotating sample flow. This allows a relatively small section of the original sample to be separated from the bulk. If necessary, the sub-sample obtained must be split again by rotating, until an analysis sample of the desired size has been obtained. Depending on the split diagram used, it is allowed to deviate from this 25% the most.

Method

Category	Tasks to be executed				Process control	
	Enforcement protocol		other Protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	General	Requirement
Soil and sludge	§ 5.5	§6.3.1.3	§ 7.7.2	n/a	Equipment	Non-contaminated Split width > 3 * D_{95} distribution time [min] > 1000/ rpm distribution steps) Explanation: distribution steps: concerns the number of vessels in the event of a fixed-number distributor (for example: 8 distribution steps for 8 vessels) or the mathematic number in the event of a rotatory splitter (e.g. 11 distribution steps with a split ratio of 1: 10).
Non-moulded building materials	n/a	§6.3.1.3	§ 7.7.2	n/a		
Moulded building materials	n/a	n/a	n/a	n/a		
					Facilities	Limiting free floating matter (localised) extraction
					Loss of mass	< 10%
					Distribution	fixed number: (average mass of the contents of the sample bottles) +/- (5% of the average mass of the content of the sample bottles) adjustable split-width / rotatory splitter: (mass of the content of the sample bottle) <5% deviation with respect to mass, calculated in the basis of the set split-width.

Process control

Distribution frequency: all samples
rotation: each samples
Loss of mass: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V IV Sub-sampling by means of coring

Principle

Sub-sampling by means of coring is a sampling method by which a representative sub-sample is obtained by taking a number of core samples from the entire (sub) sample. Sub-sampling by means of coring is only applied if:

- this is necessary due to the nature of the substance to be analysed (e.g. volatility) ;

or:

- the spread of the concentration in the sample may be regarded as small (e.g. dry matter).

Method

Category	Task to be executed				Process control	
	Enforcement protocol		Other Protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	requirement
soil and sludge	§ 5.1 § 5.2.1 § 5.2.2 § 5.3	§ 6.2.1 § 6.2.2 § 6.2.3	-	§ 7.5	Core size	- for cryogenic grinding max 25 grams - minimum grasp size see NVN 7302 § 7.5.1.
Non-moulded building materials	n/a	§ 6.2.1 § 6.2.2 § 6.2.3	-	§ 7.5	representativeness	- core sample distribution across the whole sample i.e. grasps - minimum 10 cores per sub-sample
Moulded building materials	n/a	§ 6.2.1 § 6.2.2 § 6.2.3	-	§ 7.5		

Remarks: *For the determination of volatile substances in moulded building materials, a section of the sample is to be manually reduced to < 1 cm, to which the extraction medium is immediately added after sub-sampling by means of coring.*

10 cores per analysis sample must be taken for volatile compounds. Therefore, a total of 4 x 10 cores are taken.

Distribution of identical moulded building materials must be executed randomly, during which each individual specimen has an equal chance.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.V Manual sub-sampling

Principle
When a sample is properly homogenised and the grain size is sufficiently small, a representative sub-sample can be manually taken. Sample material obtained after grinding a sample in one cup by means of the ball grind meets this requirement. The sample material is properly stirred with a clean inert spatula, after which the spatula is used to take sub-samples of the right size for the various analyses.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.VI Reduction < 4 mm

Principle

Building materials with D > 4 grindimetre must be reduced before the following pre-treatment step or analysis can be executed. The input aperture of the (laboratory) crusher is ± 80 mm the most. Larger pieces must be pre-reduced with a hammer and chisel. The sample is reduced further using the jawbreaker. The adjustable split-width is used to regulate the sample grain size to < 4 mm (95%). The cruscher is used to reduce building materials to less than 4 mm.

Method for manual pre-reduction

Category	Tasks to be executed				Process control	
	Enforcement Protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	requirement
soil and sludge	n/a	n/a	n/a	n/a	equipment	Non-contaminated
Non-moulded building materials	n/a	-	§ 7.6.1	§ 7.4.1	facilities	dry
Moulded building materials	n/a	§ 6.2.5	§ 7.6.1	§ 7.4.1	Loss of mass	< 1 m/m %

Method for reducing grains to D_{95} < 80 mm and > 4 mm

Category	Tasks to be executed				Process control	
	Enforcement protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	requirement
soil and sludge	n/a	n/a	n/a	n/a	Grain size	95% m/m < required diameter in conformance with NEN 5753
Non-moulded building materials	n/a	§7.3.1.2	§ 7.6.2	§ 7.4.2	facilities	dry Limiting free floating matter
Moulded building materials	n/a	§7.3.1.2	§ 7.6.2	§ 7.4.2	Loss of mass	< 5 m/m %

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Method for reduction < 4 mm

Category	Task to be executed				Process control	
	Enforcement protocol		Other Protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	requirement
soil and sludge	n/a	n/a	n/a	n/a	Grain size	95% m/m < 4 mm in conformance with NEN 5753
Non-moulded building materials	n/a	§7.3.1.2	§ 7.6.2	§ 7.4.2	facilities	dry limiting free floating matter
Moulded building materials	n/a	§7.3.1.2	§ 7.6.2	§ 7.4.2	Loss of mass	< 5 m/m %

Process control

Frequency control D_{95}: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

Loss of mass: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.VII Reduction < 1 mm and < 0.5 mm

Principle
As a rule, building materials have to be reduced prior to the composition analysis. The building materials are reduced using grinding equipment, suitable for reduction at room temperature and/or under cryogenic conditions. The sample can be continually fed into the reduction equipment, whereby the sample is reduced by means of rotating rotors or discs.

For the determination of the lightly-volatile substances content (and during the pre-treatment of bituminous building materials), the sample is ground to < 1 mm under cryogenic conditions (NEN 5730). The sample is chemically dried where required.
For the determination of the inorganic composition, the sample is ground to < 0.5 mm (soil only).

Method for reduction < 1 mm

Category	Tasks to be executed				Process control	
	Enforcement Protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	requirement
soil and sludge	§ 5.5	§ 6.2.2	§ 7.6.4	§ 7.4.3	Loss of mass	< 10%
Non-moulded building materials	n/a	§ 6.2.2	§ 7.6.4	§ 7.4.3	facilities	dry Limiting free floating matter (localised) extraction
Moulded building materials	n/a	§ 6.2.2	§ 7.6.4	§ 7.4.3		

* see also NEN 5730 § 8.2.1.

Method for reduction < 0.5 mm

Category	Tasks to be executed				Process control	
	Enforcement protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	requirement
soil and sludge	§ 5.5	§6.2.4.2	-	n/a	contamination	According to table 2
Non-moulded building materials	n/a	n/a	n/a	n/a	Loss of mass	< 10%
Moulded building materials	n/a	n/a	n/a	n/a	facilities	dry Limiting free floating matter (localised) extraction

Process control

Frequency control D_{95}: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

Loss of mass: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

contamination: Six-monthly or when replacing parts.

Remark: *Addition of sodium sulphate and talc during cryogenic grinding is carried out on samples or grinding procedures for which chemical drying must have been performed previously.*

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.VII Reduction < 0.125 mm

Principle

The samples for the availability test and the inorganic composition analysis in soil must be reduced to < 0.125 mm. Reduction takes place by reducing with suitable grinding equipment, such as an agate mortar.

Method for reduction < 0.125 mm

Category	Tasks to be executed				Process control	
	Enforcement protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	general	Requirement
soil and sludge	§ 5.5	§6.2.4.2	§ 7.6.3	n/a	equipment	Inert
Non-moulded building materials	n/a	§6.2.4.2	§ 7.6.3	n/a	contamination *	25% of the concentration target value
Moulded building materials	n/a	§6.2.4.2	§ 7.6.3	n/a	Loss of mass	< 10 m/m %
					Grain size	Check in conformance with NEN 5753

* Only applies to the determination of the metal composition.

Process control

Frequency control D_{95}: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

Loss of mass: 1 in 20 samples, with a minimum of once a month and a maximum of once a week.

contamination: Biannually or when replacing parts, except if the reduction equipment consists of the reference method.

Remark: *The reduction of non-moulded and moulded building materials to < 0.125 mm may be preceded, if required, by reduction to a larger grain size and sub-sampling by means of rotatory distribution.*

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.IX Drying

Principle

For specified situations, for obtaining a representative sub-sample or reducing the grain size, the sample must be dry. The sample is dried in a drying oven at a temperature of 40 to 75°C, so that it is possible to obtain a representative sub-sample or to reduce the grain size.

Method

Category	Tasks to be executed				Process control	
	Enforcement protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7312	NVN 7313	General	requirement
soil and sludge	§ 5.4	§6.3.1.1	§ 7.4	n/a	Temperature	(40 ± 2) °C of (75 ± 2) °C
Non-moulded building materials	n/a	§6.3.1.1	§ 7.4	n/a		
Moulded building materials	n/a	§6.3.1.1	§ 7.4	n/a		

Process control

frequency temperature: continuously

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.X Siphoning off free water

Principle
If a sample contains free water, this water may be removed according to NEN 5719. As it is not feasible to centrifuge a 9-kilo sample, instead, a sedimentation period of at least 1 hour is employed. After sedimentation, the clarity of the water above it is checked. If it is not clear, the sample including free water is either being treated, or the sedimentation time is extended. When the free water is clear, it can be siphoned off, for instance by means of a pipette.
The siphoned water is disposed of.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.XI Packaging

Principle

Packaging concerns all materials a sub (sample) of which is stored for a short or long time.

Packaging of the eluates obtained during leaching analysis and destruates obtained during the composition analysis do not fall under the section sample pre-treatment. These are described in the Accreditation Programme: sections "leaching analysis and composition".

Method

Category	Tasks to be executed				Process control	
	Enforcement Protocol		Other protocol			
	Non-contaminated soil	Building Materials	NVN 7311		general	requirement
soil and sludge	§ 4	§ 5	§ 7.1		material	airtight inert
Non-moulded building materials	§ 4	§ 5	§ 7.1			minimum headspace for (moderately) volatile compounds
Moulded building materials	§ 4	§ 5	§ 7.1			light-proof (or store in the dark) *Remark: a plastic bucket, if closed, is permitted*

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

Instruction sheet V.XII Storage and preservation

Principle

The sample preservation of soil and building materials consists of sealing the sample from environmental factors, or of adjusting the environmental factors, in order that they cannot influence the properties of the sample. Examples of this are storing in the dark, refrigerated storage and airtight packaging. The preservation required depends on the sample and the determinations to be carried out.
Preservation of the eluates and destruates obtained during leaching analysis does not fall under the section sample pre-treatment. They are described in the Accreditation Programme: sections "leaching analysis" and "composition".

Method for storage

Category	Tasks to be executed			Process control	
	Enforcement Protocol		Other protocol		
	Non-contaminated soil	Building Materials	NVN 7311	general	requirement
soil and sludge	§ 4	§ 5	§ 7.2/7.3/7.4	storage period	- see SIKB-protocol 3001
Non-moulded building materials	n/a	§ 5	§ 7.2/7.3/7.4		- other: < 8 weeks
Moulded building materials	n/a	§ 5	§ 7.2/7.3/7.4		

Method for preservation

Category	Tasks to be executed			Process control	
	Enforcement Protocol		Other protocol		
	Non-contaminated soil	Building Materials	NVN 7311	general	requirement
soil and sludge	§ 4	§ 5	§ 7.2/7.3/7.4	Temperature	1 °C to 5 °C
Non-moulded building materials	n/a	§ 5	§ 7.2/7.3/7.4	facility	Dark (or light-tight material)
Moulded building materials	n/a	§ 5	§ 7.2/7.3/7.4		

Explanation: *A longer storage period is only permitted if the composition or leaching of non-volatile inorganic substances of a sample is to be determined. In all other cases, and when it is not yet clear beforehand which substances are to be determined, only short-term storage under refrigerated conditions is permitted. The storage period for leaching analysis may not exceed 8 weeks, due to possible changes in the building material.*

As a rule, all building materials for preservation must be stored under refrigeration. Refrigerated storage is not required for building materials other than soil, sieve sand, rubble granulate, black mine-stone, asphaltic granulate, AVI-base ash, asphaltic concrete and cement stabilised mixtures. However, if the volatile or moderately volatile substances in these building materials are to be determined, they nevertheless must be stored under refrigeration.

Foundation Infrastructure for Quality Assurance of Soil Management
Büchnerweg 1 PO Box 420 NL-2800 AK Gouda, The Netherlands
Telephone +31 (0)182 540675 Fax +31 (0)182 540676

V7 Literature

Draft- NEN 5709	2004	Soil – Sample Pre-treatment for the determination of organic and inorganic parameters in soil
NEN 5719	1999	Soil – Pre-Treatment of water-bed samples.
NVN 5730	1991	Soil – Sample Pre-treatment for the determination of organic parameters in soil.
Draft- NEN 5753	2004	Soil – Determination of the silt content and grain size of soil samples using a sieve and pipette.
NVN 7302	1997	Leaching properties of moulded ground and stone-like building materials and waste materials – Sampling – Sampling granular materials from static batches.
NVN 7303	1997	Leaching properties of moulded ground and stone-like building materials and waste materials – Sampling – Sampling moulded and monolithic materials.
NEN 7310	1995	Leaching properties of moulded soil and stone-like building materials and waste materials – Sample Pre-treatment – General instructions.
NVN 7311	1995	Leaching properties of moulded soil and stone-like building materials and waste materials – Sample Pre-treatment – Sample storage and preservation.
NVN 7312	1995	Leaching properties of moulded soil and stone-like building materials and waste materials – Sample Pre-treatment – Sample Pre-treatment for the determination of the leaching properties and the levels of inorganic components.
NVN 7313	1995	Leaching properties of solid soil- and stone-like building materials and waste materials – Sample Pre-treatment – Sample Pre-treatment for determining the leaching behaviour and the content of organic components.
Draft-NEN 7331	2004	Bitumen and materials that contain bitumen – Determination of the content of polycyclic aromatic hydrocarbons (PAH) and benzene, toluene, ethyl-benzene and xylene (BTEX) – Gas chromatography method.