ACCREDITATION PROGRAMME BUILDING MATERIALS DECREE

Section: Soil Composition

AP04 - SG

INSTRUMENTEN
VOOR EENVOUDIGER
EN BETER
BODEMBEHEER

SIKB

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SG1 Introduction

The Accreditation Programme "Building Materials Decree, section composition; soil" (hereafter called AP04-SC) describes the tasks and the performance characteristics of those tasks that must be applied when carrying out a composition investigation of soil within the scope of the Building Materials Decree. Furthermore, the testing procedures, to which a task must comply, for the determination of the performance characteristics and the corresponding criteria have been defined.

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-SG. In accordance with the standardised regulations, a number of tasks have been strictly regulated. In respect of those tasks that are not strictly regulated, the reference point with national and international standardised regulation is defined. If a task is not executed in conformance with defined national and international regulations, the equivalence of the measuring method has to be proven.

In this document, the tasks relating to composition investigation of soil that fall within the scope of the Accreditation Programme are stated in Section SG2. In Section SG3, the concepts and parameters being used are defined and, in Section SC4, the validation of a task through widely acknowledged procedures is provided. In Section SG5, the first, second and third line controls that must be applied at the very least during the execution of tasks that fall under the Building Materials Decree Accreditation Programme are described. The technical data for the tasks and the corresponding performance characteristics are given in Section SG6.

SG2 Overview of tasks

This section of the Accreditation Programme includes all the tasks that are required for the execution of soil composition analysis within the scope of the “Building Materials Decree”. For the specification of Composition; soil, the tasks have been divided into packages. For a breakdown of the packages please refer to AP04-A.

In APO4-SG the following tasks are described:

- Determination of the pH of soil;
- Determination of the dry matter content in soil;
- Determination of the clay content in soil;
- Determination of the organic matter content in soil;
- Determination of the elements present in soil;
- Determination of the mercury content of soil;
- Determination of the cyanides content (total-free and total-complex pH>5) in soil;
- Determination of the content of volatile aromatic and volatile halogenated hydrocarbons in soil;
- Determination of the polycyclic aromatic hydrocarbons content in soil;
- Determination of the extractable organo-halogen compounds (EOX) content in soil[1];
- Determination of the mineral oil content in soil[1].
- Determination of the bromide content in soil;
- Determination of the fluoride content in soil;
- Determination of the chloride content in soil;
- Determination of the chlorophenol content in soil;
- Determination of the PCB and OCB content in soil;
- Determination of the chlorobenzene content in soil;
- Determination of the organo-nitrous pesticide content in soil.
- Investigation protocol for other analytes.

For the pre-treatment of soil samples, please refer to the relevant section of the Accreditation Programme AP04-V.

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[1] *this parameter is method dependent, see also the relevant Performance Sheet.*

SG3 Concepts/parameters

Several concepts and terms regularly recur in the Accreditation Programme.
In order to avoid confusion, the concepts and terms are defined below.
Definitions and testing procedures are wherever possible in line with the following standards that are generally applied in the Netherlands: NEN 3114, ISO 3534-1, ISO 6879, ISO 8466-1, Dev. Draft. NEN-EN-ISO 9169, NEN 7777 and NEN 7778.

SG3.1 Definitions of terms

In this section, the terms used in the definitions and testing procedures for performance characteristics in Sections SG3.2, SG4 and SG5 are defined.

True value (NEN 3114, NEN 7777)

Value of a precisely defined unit.

Explanation: *The true value that would be obtained during a perfect measurement.*

Measurement unit (NEN 7777)

Characteristic of a phenomenon or body that is suitable for qualitative distinction and quantitative determination.

Example: *The unit "mass concentration of nitrate"; note that "nitrate" is in itself not a measurement unit.*

Measurement value (NEN 3114, NEN 7777)

A value obtained by measuring.

Explanation 1: *The "measurement value" may have been defined as the average of a multiple analysis.*
Explanation 2: *The "measurement value" may be the result of a measurement followed by one or more operations such as correction for "procedure blank".*
Explanation 3: *"Measurement value" is synonymous with the "analysis result" (to be reported).*

Measurement prognosis (NEN 3114)

Value that the average measurement value approaches after an increasing number of measurement values.

Measuring method (NEN 3114, NEN 7777)

Manner in which measurements are obtained under clearly defined circumstances.

Explanation: *"Measuring method" is synonymous with "analysis method", "determination method" or "task".*

Systematic deviation (NEN 3114, NEN 7777)

Difference between the measurement prognosis and the true value.

Explanation: *"Systematic deviation" refers to "trueness".*

Accidental deviation (NEN 3114, NEN 7777)

Difference between a measurement value and the measurement prognosis.

Control sample

Homogeneous material which, in terms of composition and form, is accepted as being representative of the samples and which is intended for use in monitoring one or more performance characteristics of the measuring method.

Calibration line/calibration curve (formulated according to IUPAC)

Graphic representation of the signal as a function of the measurable quantity.

Measurement signal (NEN 7777)

Unit that represents the measurement quantity and that is connected to it functionally.

Explanation 1: *The "electrical charge" on the output of an measurement instrument is an example of the unit "measurement signal".*
Explanation 2: *"Response" is synonymous with "measurement signal".*

Conventional true value (NEN 3114, NEN 7777)

Value that deviates from the true value by such a small extent that the difference between the two values is negligible.

Explanation: *The conventional true value is the value that is conventionally measured in a practical situation and which approximates as close as possible to the true value.*

Remark: The FeNeLab sample that has been developed within the framework of ANVM project 152b contains conventional true values for a large number of parameters.

Calibration function (IUPAC, NEN 7777)

Mathematical correlation between the measurement signal and the measurement quantity.

Example: The peak area as a function of the concentration in chromatographic analysis.

Explanation 1: *In practical situations, the mathematical correlation is often estimated using a regression function.*
Explanation 2: *As a rule, the calibration encompasses the entire measurement method. If, for practical reasons, part of the measurement method cannot be incorporated in the calibration, then, in the definition, the said unit is "an auxiliary unit related to the measurement quantity (e.g. the "concentration in the extraction agent" as an auxiliary unit for the measurement quantity "concentration in soil").*

Uncertainty of measurement (NEN 3114, NEN 7777)

Half the length of the interval within which the true value is expected to be.

Explanation: *The uncertainty of measurement often is characterised as a multiple of the (total) standard deviation or variation coefficient.*

Standard deviation (s) (NEN 3114)
Square root of measured variance, where the variance is the sum of the squared measured accidental deviations divided by the number of measured values minus one.

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$

Variation coefficient (vc) (NEN 3114)
Quotient of the standard deviation and the absolute value of the measurement prognosis.
The variation coefficient is related to the concentration. If the concentration can be taken as constant, then it is defined as follows, with "percent" as the unit:

$$vc = 100 * \frac{s}{\bar{x}}$$

In the event of duplicate determinations, in which the concentration is not constant, but where vc is assumed to be constant, the following formula is used:

$$vc = 100 * \sqrt{\frac{\sum_{i=1}^{n} \left(\frac{x_{i1} - x_{i2}}{0,5(x_{i1} + x_{i2})} \right)^2}{2n}}$$

Conformal (measurement) method
Measurement method in which the execution does not deviate in critical areas from the prescribed tasks. An area is seen as non-critical if a deviation in it has no demonstrable effect on the result.

Equivalent (measurement) method (NEN 7778)
Measurement method that meets the requirements that have been set for the intended use of the reference method from a ‘fitness for purpose’ perspective.

Matrix analysis
Analysis in which the performance of the measurement method is determined for relevant sample composition classification groups.

Remark: *In general, this concerns the various types of matrices (e.g. soil: peat, clay and sand) and known interferences.*

z-score
Deviation of the measured value from the test value, relative to the standard deviation.

$$z_i = \frac{x_i - \bar{x}}{s}$$

where: x_i represents the measured value;
x represents the test value such as:
  - an "assigned value" in an inter-laboratory investigation;

- an assigned value for a reference material;
- a measurement value using a reference method.

s represents : standard deviation.

Explanation 1: *In principle, in this document, testing is always performed according to the variation coefficient stipulated in the Performance Sheet.*

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Explanation 2: *In this document, the z-value is used for testing systematic differences. For the evaluation of the deviations for a series of measurement values, the mean* z^2 *is calculated:*

$$\overline{z^2} = \frac{z_1^2 + z_2^2 + \dots + z_n^2}{n}$$

The threshold values of both z and the average z^2 *are determined, taking into account the risks of accidental violation and the relevance with respect to the total measurement error.*

SG3.2 Definitions of performance characteristics

Detection threshold (NEN 7777)

Lowest concentration of a sample component for which its presence can be determined with a certain degree of certainty.

Explanation 1: *Detection threshold is related to the quality criterion "presence".*

Explanation 2: *The legally required detection threshold,* AG_{eis}*, is the minimum detection threshold that must be achieved. This is laid down in the Performance Sheets for each parameter.*

Explanation 3: *In this document, reference is made to NEN 7777 for the determination of the detection threshold. In this standard, the operational definition is used for the determination threshold, which equates the determination limit to ten times the standard deviation at the relevant level. Therefore, the determination threshold is the value of the measurement quantity at which the variation coefficient by convention amounts to 33%.*

Determination threshold (NEN 7777, Appendix C)

Lowest concentration of the component in the sample, for which the presence can still be determined with a certain degree of certainty.

Explanation 1: *Determination threshold is related to the quality criterion "measurement value".*

Explanation 2: *The term determination threshold is not used further in this document, the definition and the explanations have been included here only in order to illustrate the relation to the detection limit.*

Explanation 3: *In this document, reference is made to NEN 7777 for the determination of the detection threshold. In this standard, the operational definition is used for the determination threshold, which equates the determination limit to ten times the standard deviation at the relevant level. Therefore, the determination threshold is the value of the measurement quantity, in which the variation coefficient by convention amounts to 10%.*

Repeatability (NEN 3114, NEN 7777)

Standard for the spread in the measured values obtained using the same method in an identical material under the same conditions.

Explanation 1: *"Repeatability" is a form of "precision" (see under "Precision”).*

Explanation 2: *"Repeatability" is quantified in practice as "lack of repeatability".*

Explanation 3: *The only remaining variable in a practical situation is time.*

Explanation 4: *The maximum permitted variation coefficient for repeatability* ($vc_{r.eis}$) *is specified for each parameter in the Performance Sheets.*

Reproducibility (NEN 7777)

Standard for the spread in measured values obtained using the same method in identical material under different conditions.

Explanation 1: *Reproducibility is a form of precision (see under "Precision").*

Explanation 2: *"Different conditions" may relate to "executor", "laboratory" (equipment, chemicals, standards), "environmental factors" (temperature, humidity), "time".*

Explanation 3: *It is often useful to make a distinction between "intra-laboratory reproducibility and "inter-laboratory reproducibility"; in these cases, the "different conditions" relate to the variability within the laboratory or within a group of laboratories. If the group of laboratories is representative for all, then "inter-laboratory reproducibility" equals "reproducibility of the methods".*

Explanation 4: *The maximum permitted variation coefficient for the intra-laboratory reproducibility ($vc_{W.eis}$) is specified for each parameter in the Performance Sheets.*

Linearity

The relation between measurement quantity and measurement signal as characterised by a straight line.

Explanation 1: *"Linearity" is a theoretical concept. There will always be some deviation from the linear relationship. Sometimes, this deviation is so small that it cannot be determined because of the spread in the measurements. In such cases, linearity is assumed.*

Explanation 2: *As the number of measurements for certain values of the measurement quantity increases, smaller deviations from the linearity can be determined (the spread of the average measured value decreases in line with the number of measured values).*

Explanation 3: *"Lack of fit" is a more general concept for the difference between the measured relationship and the assumed (mathematical) relationship for the calibration function. Thus, non-linearity is a deviation from the assumed linear relationship.*

Recovery (NEN 7777)

Fraction of the measurement component that is recovered during analysis after adding a known quantity of the measurement component to a sample under defined conditions, or the quotient of the measurement value for a sample and the conventional true value.

Explanation 1: *Recovery determined by the addition of the measurement component often generates over optimistic results because it is difficult, or impossible, to include the added component in the sample in the same way as the original presence.*

Precision

Degree of conformity between measurement results in repeated measurements under prescribed conditions.

Explanation 1: *"Prescribed conditions" usually refer to differences in laboratories, executors and equipment.*

Explanation 2: *"Precision" is an umbrella concept. It is quantified in the form of "repeatability" and "reproducibility".*

Trueness/systematic deviation (NEN 7777)

Difference between the measurement prognosis and the true value.

Explanation: *"Trueness" is generally defined and quantified in actual practice as "untrueness".*

Accuracy (NEN 3114)

Degree to which the measurement value obtained using a specific measurement method approximates the true value.

Explanation 1: *The Dutch term "Nauwkeruigheid" is synonymous with the English term "accuracy".*

Explanation 2: *"Accuracy" is the combination of the performance characteristics "trueness" and "reproducibility".*

Selectivity (ISO 6879, NEN 7777)

Subject to the measurement value of a variable other than the measurement quantity.

Explanation 1: *The said variables primarily relate to the sample, e.g. the concentration of a substance in the sample.*

Explanation 2: *The measurement method becomes increasingly selective as the influence of other variables on the measurement value decreases.*

Measurement range (NEN 7777)

Range of the measurement unit within which the performance characteristics meet the defined requirements.

Explanation: *The measurement range is defined for the entire measuring method. If dilution of the sample forms an explicit part of the established measurement method, then the dilution must be included in the quantification of the measurement range.*

Regarding the choice of concentration levels within the framework of validation and quality assurance, the measurement range of a task within the scope of AP04 refers to the range between the demonstrability thresholds stated in the Performance Sheets and the highest value of the measurement unit that can be measured according to the regulations, between which the performance characteristics meet the defined requirements.

Trennzahl or separation number (NPR 6405)

The Trennzahl, or separation, number Tz can be used as a measure of the separating power of the chromatographic system. The Trennzahl number is defined as follows:

$$Tz = \frac{(t_{R,j} - t_{R,i})}{w_{1/2,i} + w_{1/2,j}} - 1$$

where:
$t_{R,i}$ and $t_{R,j}$: are the retention times of compounds i and j respectively;
$w_{1/2,i}$ and $w_{1/2,j}$: the half-height breadths for compounds i and j respectively.

Explanation 1: *The Trennzahl number is a measure of the efficiency and selectivity of a chromatographic system.*
Explanation 2: *In general, the Trennzahl number corresponds to the number of analytes (peaks) that can be separated between the analytes i and j.*

Time lapse (ISO 6879)

Systematic change in the time of the measurement value for the same value of the (measurement) quantity.

Explanation: *If not specified further, "time lapse" only refers to a change in the calibration function.*

SG4 Validation of a task

An intra-laboratory validation investigation should be performed when introducing or modifying a task.
During a validation investigation, the following performance characteristics should be assessed:

- demonstrability limit;
- measurement range;
- linearity/lack of fit;
- trueness/recovery;
- repeatability;
- intra-laboratory reproducibility.

The investigation should demonstrate that the performance characteristics are equal to those of the set standard applicable as the reference point within the Accreditation Programme.
NEN 7777 is used as a basis for the determination of the performance characteristics. Testing against the criteria should be in accordance with the procedure given in this standard. The criteria for the various performance characteristics are stated in the performance sheets. These values are regarded as estimated limiting values.
If a task is performed in conformance with a prescribed standard, the performance characteristics should only be tested against the performance characteristics in the performance sheets.

In principle, the performance characteristics apply to the entire task as described in the instructions, which includes sample pre-treatment. If this is not the case, the performance sheet of the validation investigation will explicitly state for which part of the task the performance characteristics apply.

In the event of a deviation from the standard, it must be determined by means of practical samples if the selectivity of the task is equal to the standardised performance. For those parameters that are determined by the method, the measurement principle must not be deviated from, and the method must conform with the defined task.

The validation must be repeated if there is a change in either the equivalent task or the standard. This should conform to NEN 7777 “Introduction of a different method”.
Validation investigations executed according to the procedures described in previous versions of AP04 remain valid. A new validation in accordance with the procedure stated in this version (Version 7) onwards, is not necessary.

The matrices that fall within AP04-SG are regarded as a single field of application.

If not stated otherwise, the performance characteristics are determined in a soil sample (silt 1- 3%, organic matter 1- 3% and particle size: 97% < 210 μm), after addition of the analytes to be investigated.

Concentration levels for validation are chosen in accordance with NEN 7777 in the lowest decile of the measurement range (see definition measurement range SG3) and in the highest decile of the measurement range.

SG4.1 Test procedure and quantification of performance characteristics

SG4.1.1 Demonstrability limit (AG)

The determination of the demonstrability limit is described in NEN 7777. The demonstrability limit must be determined on the basis of repeatability conditions in a sand sample (silt < 3%, organic matter < 3% and particle size: 97% < 210 µm).

Criterion:

- The demonstrability limit must meet the requirements stated in the performance sheet.

SG4.1.2 Determination of the measurement range of the task

The measurement range is the domain between the demonstrability limit and the highest value (minimal intervention value) of the measurement variable that, following the instructions, can still be measured while the performance characteristics meet the defined requirements.

Explanation 1: *If dilution of the samples is part of the set measurement method, the dilution must be included in the quantification of the measurement range.*

Explanation 2: *"Demonstrability limit" must be replaced by "lowest value" if due to other limitations (e.g. non-linearity) a performance characteristic (e.g. linearity) fails to meet the defined requirement for values above the demonstrability limit.*

SG4.1.3 Testing of the linearity / "lack of fit" of the calibration function of the analysis-instrument

Testing the practicability of the chosen calibration function for the entire measurement range is explained in NEN 7777. The testing should take place on the basis of absolute limiting values.

The following formula can be used to determine, for c, the requirement in model deviation ($\delta_{\text{c,model,abslim}}$):

$$\delta_{\text{c,model,abslim}} = \sqrt{\frac{AG_{eis}^2}{9} + c^2 * \left(\frac{vc_{W,eis}}{100}\right)^2}$$

Calculate the deviation of the theoretical concentration:

$$\delta_{\text{c,model}} = \bar{x} - theoreticalconcentration$$

where: $\bar{x}$ = mean measurement result in mg/kg.ds

The result is satisfactory if:

$$\left|\delta_{c,\mathrm{mod}\,el}\right| \le \delta_{c,\mathrm{mod}\,el,abs\lim} - \frac{t_{0,975} * s_w}{\sqrt{n}}$$

where: s_w = intra-laboratory reproducibility standard deviation of the measurement results in mg/kg.ds
n = number of measurement results

SG4.1.4 Determination of the intra-laboratory trueness / recovery (δc, Tv)

The intra-laboratory trueness / recovery of the task can be determined – in decreasing order of preference – in the following ways:

- certified reference material (or sample with a conventionally true value);
- "spiked samples".

SG4.1.4.1 Intra-laboratory trueness by means of a certified reference material *(or sample with a conventionally true value)*

The determination of the intra-laboratory trueness according to this method is explained in NEN 7777.

Criterion:

- The intra-laboratory trueness must meet the requirement for recovery specified in the performance sheet.

SG4.1.4.2 Recovery using addition

Recovery investigations can be performed using "spiked samples" where a certified reference sample is not available for the task and for a specific matrix. The sample material is "spiked" after pre-treatment of the sample in accordance with AP04-V.

The method for the determination of recovery from addition is defined in NEN 7777.

Explanation 1: *If possible, the samples should be stored for 48 hours under the conditions given in the performance sheet. After 48 hours, the samples are analysed according to the instructions. This is not possible with volatile compounds (for the definition of volatile compounds, see AP04-V).*

Criterion:

- The recovery must fulfil the requirements stated in the performance sheet.

SG4.1.5 Determination of repeatability standard deviation (sr, vcr)

The determination of the repeatability standard deviation is defined in NEN 7777. It can be achieved in two ways: duplicate analysis of different samples, or repeated analysis of the same laboratory sample. Both methods are acceptable.
In the determination, the complete measurement procedure must be completed, that is, inclusive of any sample pre-treatments.
This means that a sample must be pre-treated in accordance with APO4-V before it is analysed using either duplicate or multiple approaches. If an analyte is to be added to a sample in order to obtain an appropriate level of concentration, this may be done either before or after the sample is pre-treated.

Unless stated otherwise, the repeatability is expressed in the performance sheet as the variation coefficient (vc_r).

Criterion:

- The repeatability variation coefficient must fulfil the requirements stated in the performance sheet.

SG4.1.6 Determination of intra-laboratory reproducibility standard deviation (sW, vcW)

The determination of the intra-laboratory reproducibility standard deviation is explained in NEN 7777. It can be executed in two ways: duplicate analysis of different samples or repeated analysis of the same laboratory sample. Both methods are acceptable.
In the determination, the complete measurement procedure must be completed, that is, inclusive of any sample pre-treatments.
This means that a sample must be pre-treated in accordance with APO4-V before it is analysed using either duplicate or multiple approaches. If an analyte is to be added to a sample in order to obtain an appropriate level of concentration, this may be done either before or after the sample is pre-treated.

Explanation 1: *In determining the intra-laboratory reproducibility standard deviation, the control sample from the first-line control may be used.*

Unless stated otherwise in the performance sheet, the intra-laboratory reproducibility is expressed as variation coefficient (vc_W).

Criterion:

- The intra-laboratory reproducibility variation coefficient must meet the requirements specified in the performance sheet.

SG4.2 Equality investigation of a non-standardised task

An equality investigation is not relevant for parameters that are determined by the approved method, but must be carried out if a task is not performed in accordance with the reference method. Any equality investigation must conform to NEN 7778.

SG4.3 Reporting of activities

A validation investigation should be concluded with a validation report. The conclusions of the investigation should be relevant to APO4 and be presented in a performance sheet in which the validation result and the APO4 requirements are included.
Complete documentation of all validation investigations carried out should be available during the accreditation assessment.

SG5 Quality assurance of a task

The quality assurance of a task is subdivided as follows:
First-line control - Performance checking by the executors themselves.
Second-line control - A check within the institute’s organisation but independent of the executors.
Third-line control - Independent external check, ring tests.

The quality assurance of the institute must be laid down in first-, second- and third line documents that should cover the following aspects:

- statistical supervision of the quality assurance;
- resolution of out-of-control situations;
- use or preparation of samples;
- responsibilities of officials.

The defined first-, second- and third-line controls do not represent additional quality assurance, but rather the minimum quality assurance to be applied.

Usually, tasks are executed routinely, but a task may be non-routine for various reasons. For example, if:

- it turns out in practice that over long periods no samples are presented that require a specific task;
- work is performed under a clustering arrangement where, according to the schedule, a task is not performed by a certain laboratory over a lengthy period, followed by a period in which it does perform this task.

There is no precise dividing line between a routine task and a non-routine one. It may be that a task should be considered non-routine over lengthy periods, but routine during other periods when there is a regular demand (such as in the example of the cluster arrangement).
Overall guidelines are:

- for first-line control (SG5.1): follow the procedure for a non-routine task if, over a period of four consecutive quarters, a full control chart cannot be obtained.
- for second-line control (SG5.2) and third-line control (SG5.3): pursue the procedure for a non-routine task if, over a period of four consecutive quarters, during three of these four quarters no samples are presented.

The first-, second- and third-line controls differ when it comes to routine and non-routine tasks. Below, for each level, the quality assurance activities are outlined that should be executed for routine and for non-routine tasks.
Quality assurance can only be executed for non-routine tasks, after accreditation and definitive approval have been obtained on the basis of a validation investigation and quality assurance of a routine task.

SG5.1 First-line control

First-line control should involve periodic performance investigation and a quality check during the execution of a task.
During the periodic performance investigation, the compliance of the task with the guidelines in the performance sheet must be checked (Section SG6). Quality assurance during the performance of a task is achieved using control samples.
A different schedule applies for non-routine tasks.

SG5.1.1 Periodic performance investigation

The basic materials with which the investigation should be carried out are:
Soil: sand sample (silt < 3%, organic matter < 3%, grain size: 97% < 210 μm), unless stated otherwise.

SG5.1.1.1 Recovery

Analytes : all parameters.
Concentration range : "spiked samples" with concentrations within the measurement range of the task
Method : see SG4.1.4.
Frequency : 1 out of every 100 samples, with a minimum of once a month and a maximum of once a week.
Number : individual.
Criterion : See the criterion stated in the performance sheets.

Periodic performance investigation of recovery is not applicable for non-routine tasks (see SG5.1.3).

Explanation : *During multi-component analysis, the critical parameters are recorded in a control chart. The recovery of the other components should be determined every six months.*

SG5.1.1.2 Repeatability variation coefficient (vcr)

Analytes : all parameters.
Concentration range : within the measurement range of the task
Method : routine investigation:
1 in every 100 practical samples presented should be analysed in duplicate, with a minimum of 1 practical sample per month and a maximum of 1 practical sample per week. However, for determinations that are carried out using a multiple approach as standard, a daily test in accordance with SG5.1.4.1 suffices.
: non-routine investigation:
see SG4.1.5.
Frequency : routine investigation:
quarterly evaluation.
non-routine investigation:
once a year, provided the task has been performed.

Criteria:
routine investigation : the repeatability variation coefficient vc_r , as determined in accordance with SG4.1.5.1, must satisfy the requirements stated in the performance sheet.
non-routine investigation : satisfy the requirements stated in the performance sheet.

If desirable for practical reasons, the duplicate analysis may be spread over two days rather than completed within a single day. In this situation, the intra-laboratory reproducibility variation coefficient vc_w requirements, stated in the performance sheet, must be fulfilled.

SG5.1.1.3 Intra-laboratory reproducibility variation coefficient (vcW)

The intra-laboratory reproducibility variation coefficient must be determined if:

- a control chart is not being used,
- a control chart is used but one from which the intra-laboratory reproducibility cannot be determined for all the parameters in the desired concentration range.

Analytes : parameters.
Concentration range : within the measurement range of the task
Method : see SG4.1.6.
Frequency : routine investigation:
once a year.
non-routine investigation:
once a year, provided the task is carried out.
number : at least ten samples.
Criteria : requirements stated in the performance sheet.

SG5.1.1.4 "Lack of fit" / linearity of the calibration function of the analysis instrument

The validity of the calibration function must be tested, if:

- the routine determination of the function in accordance with the measurement instructions is not overdimensioned[2], and
- the sensitivity (the inclination of the calibration function) has changed by more than 20%, or the instrument is re-introduced after a major intervention or after a long period of non-use.

In a multi-component analysis those analytes that are decisive for the measurement range are tested.

Analytes : all parameters;
Concentration range : the measurement range of the analysis instrument;
Method : see SG4.1.3
Checking the “lack of fit” as part of quality assurance is performed as an indicative measure of model deviation. This may be carried out under repeatability conditions. The testing may be based on the repeatability standard deviation of the measurement results in mg/kg.ds. It is recommended that multiple measurements are taken not only of the measurement standards, but also of the calibration standards.
Number : see SG4.1.3
Criteria : see SG4.1.3

Periodic performance investigation of “Lack of Fit” is not applicable for non-routine tasks (see SG5.1.3).

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[2] *Overdimensioned in a linear function means that calibration takes place on more than two levels, using a second-order polynomial, or on more than three, etc.*

SG5.1.1.5 Demonstrability limit (AG)

The demonstrability limit of the task must be assessed if the sensitivity (measurement signal/number of analytes) decreases to such a degree that it becomes likely that the required demonstrability limit will not be reached.
The criterion is that the demonstrability limit must be determined if

$$B_{gev} < \frac{AG_{vst}}{AG_{eis}} * B_{vst}$$

where: B_{gev} : the found sensitivity;
AG_{eis}. : the demonstrability limit specified on the performance sheet;
AG_{vst} : the demonstrability limit in the previous determination investigation;
B_{vst} : the sensitivity in the previous determination investigation of the demonstrability limit.

Explanation 1: *During a multi-component analysis, those components are chosen whose found demonstrability limits are closest to the requirement stated in the performance sheet.*
Explanation 2: *The demonstrability limit must be assessed if, after the removal of a defect or optimisation of the analysis instrument, the analysis instrument does not fulfil the specifications/requirements.*

Analytes : all parameters, or the critical parameters.
Method : see SG4.1.1.
Criterion : the demonstrability limit must satisfy the requirements given in the performance sheet.

SG5.1.2 Quality assurance during the execution of a routine task

The institute must check the quality of the task during its execution, in such a way that the quality is demonstrably assured in all aspects of the task (preparation activities for the analysis as well as the analysis itself). This quality assurance must be carried out on an actual soil sample (unless indicated otherwise on the performance sheet) in which the measurement value lies within the measurement range of the task.

The results must be checked statistically by means of control charts (Shewhart chart), unless otherwise stated on the performance sheet.

Explanation 1: *If an equivalent measurement method is employed, the institution must record those parameters for which the measurement method is being assured in a control chart.*

SG5.1.2.1 Producing a control sample

For a control sample, a practical sample or a mixed sample, preferably a mixture of types of soil, should be used. The practical sample must contain the analytes to be determined (see performance sheet) in the desired concentrations. The sample must be homogenised in such a way, that any inhomogenity in the sample has no significant influence on the intra-laboratory reproducibility. For practical reasons (limited storage life) the control sample may be replaced by a recovery experiment, such as the determination of volatile components in soil. Instead of the control sample, a reference sample may also be used provided it meets the set criteria (see SG4.1.4.1).

Note: In practice, creating a satisfactory control sample may present difficulties. If one or two analytes do not lie within the concentration range required, they may be added to the sample (see SG4.1.4).

SG5.1.2.2 Creating a control chart

A control chart is created by performing the task at least ten times under intra-laboratory reproducibility conditions.

An outlier test is carried out on these first 10 observations if the performance characteristics found do not fulfil the requirement for the intra-laboratory reproducibility deviation set in the performance sheet. In order to identify the outliers, a Grubbs test is executed no more than twice. Once the outliers have been removed, the mean and the standard deviation are determined once again and the outlier test repeated. This is repeated until outliers are no longer detected.
At least eight observations must remain if they are to be used to start a control chart. The mean and the 2s and 3s limits are calculated on the basis of these experiments.
Grubbs-test:

$$G_p = \frac{|x_p - \bar{x}|}{s},$$ where x_p is the individual observation being tested

if G_p > critical value, then the value is an outlier. For n=10 $G_p = 2.482$
n=9 $G_p = 2.387$
n=8 $G_p = 2.274$

SG5.1.2.3 Completing the control charts (NPR 6603)

When the task is carried out, one independent measurement value (not a mean value of a multiple measurement or determination) is recorded each working day, on the control chart. If several control samples are analysed in a working day, the values are tested against the control chart data.

SG5.1.2.4 Evaluation against a control chart (Shewhart)

The following events indicate an “out-of-control” situation:
- a single crossing of the 3s limit;
- successive crossings of the 2s limit on the same side of the mean;
- eleven consecutive values on the same side of the mean.

An out-of-control situation is also indicated if during additional testing of several control sample on a single working day one of the following events occurs:
- the 3s limit on the control chart is exceeded;
- the 2s limit is successively exceeded on the same side of the mean under identical measurement conditions (executor and/or equipment).

In the event of an out-of-control situation, the following measures should be taken:
1) An investigation into the cause should be carried out.
2) The measurement results of the relevant series, day or period are withheld until the cause of the fault has been established. Analysis results that are lower than the reporting limit are an exception to this rule where the out-of-control quality is due to an overestimation of the true value. These analysis results can be reported.
3) After the cause of the fault has been rectified, the relevant series of samples is usually re-analysed.
4) When an out-of-control event due to 11 observations on one side of the mean occurs, the cause of the shift should be investigated immediately. The contents of the samples that are part of the eleventh observation can nevertheless be reported.
   If the problem cannot be solved, (no cause is found or the cause cannot be removed), the recovery must be re-determined and assessed against the criteria laid down in the performance sheets. The control chart is closed immediately and the charts are combined. The new chart begins with n=0, sum x=0 and sum x_i^2=0. When the new control chart is closed, the cumulative standard deviation, determined in the previous periods, is incorporated.
   During the determination of the relevant performance characteristics, samples may well be analysed and reported. To avoid an out-of-control situation, the results of the first line control samples must satisfy the characteristics of the most

recent control chart.

SG5.1.2.5 Testing a control chart

When a control chart is full, it is assessed in accordance with NPR 6603. The following issues are also important:

- When the mean and the standard deviation of a full control chart are calculated, out-of-control results, caused by exceeding the 3s limit, should be excluded.
- When a control chart is closed, the mean and standard deviation are assessed with respect to the historical data on previous charts and the requirements for trueness (recovery) and intra-laboratory reproducibility laid down in the performance sheets.
- NPR 6603 describes how the assessment related to the historical data of previous charts should be executed for the standard deviation, but not for the mean. The mean should be tested using a t-test:

$$t = \frac{\left|X_1 - X_2\right|}{\sqrt{(A+B)}}$$

where X_1 and X_2 are the means of the previous control chart(s) and the present control chart respectively,
and

$$A = \frac{s_1^2}{n_1} \text{ and } B = \frac{s_2^2}{n_2}$$

where s_1 and s_2 are the standard deviations of the previous and the present control charts respectively.

The number of degrees of freedom is $v = \dfrac{(A+B)^2}{\dfrac{A^2}{(n_1+1)} + \dfrac{B^2}{(n_2+1)}} - 2$

Subsequently, the testing value of t(0.95;v) can be determined.

- In the event that the means and/or the standard deviations cannot be combined, the cause of this should be investigated immediately. If the quality of the analysis has improved (recovery closer to 100% or a smaller standard deviation), there is no need to further investigate the cause. If there has been an increase in the standard deviation or a deterioration in the recovery rate but the requirements laid down in the performance sheet are still met, then the cause needs not be investigated and the charts may be combined. In this situation, consideration should be given to calculating a new mean or standard deviation.

In the event that the quality has not improved or the recovery rate or standard deviation fails to meet the requirements of the performance sheet, a further investigation must be carried out. If this does not solve the problem (no cause found or the cause cannot be removed), the relevant performance characteristics must be re-determined and again assessed against the criteria laid down in the performance sheets. In the situation of a deviation in the mean, the trueness/recovery must once again be determined; and in the case of a deviation in the standard deviation, the intra-laboratory reproducibility standard deviation must be again determined. The control chart is then closed and the charts combined. The new chart starts with n=0, sum x=0 and sum $x_i^2=0$. During the determination of the relevant performance characteristics, it is still allowable to analyse samples and report. In order to avoid a situation of an out-of-control quality, the results of the first-line control samples must satisfy the characteristics of the most recent control chart.

Once it has been concluded that no cause can be found, the performance characteristics of the new control chart must be determined within 10 measuring days. Further, these 10 measuring days must fall within a period of 3 months.

- When combining control chart data, in order to ensure that the latest data have a sufficient influence on the values, the previous four charts (approximately 120 observations) are made full use of. When creating a new chart, the mean and the 1s, 2s and 3s limits are based on the means and the standard deviation of the previous five charts.

SG5.1.3 Quality assurance when executing a non-routine task

The quality of a non-routine task is assured by means of a limited validation of the task. During a limited validation investigation, the following performance characteristics must be assessed at the very least:

- recovery;
- "lack of fit" / linearity, provided the determination of the calibration function is not overdimensioned (see note 5.1.1.4);
- repeatability variation coefficient;
- demonstrability limit.

These performance characteristics must be tested for each investigation. The performance characteristics must minimally be determined with the experiment below:

1) one blank test,
2) two recovery tests in the lowest decile of the measurement range.
3) two recovery tests in the highest decile of the measurement range.

SG5.1.3.1 Recovery

Analytes : all parameters;
Method/assessment : see SG4.1.4. under repeatability conditions
Frequency : per investigation;
Criterion : the mean recovery (experiments 2 and 3) must meet the requirement stated on the performance sheet.

SG5.1.3.2 Repeatability variation coefficient

Analytes : all parameters;
Frequency : per investigation;
Criterion : the variation coefficient is established through the recovery experiments (experiments 2 and 3). This variation coefficient must be lower than the repeatability requirement stated in the performance sheet.

SG5.1.3.3 "Lack of fit" / linearity of the calibration function

Analytes : all parameters;
Frequency : per investigation;
Calculation : see SG4.1.3
Criterion : see SG4.1.3

SG5.1.3.4 Demonstrability limit

Analytes : all parameters;
Frequency : per investigation;
Criterion : see SG5.1.1.5.

SG5.1.4 Additional daily quality assurance issues

If additional quality assurance points are listed in the standardised regulation then these must be checked. Further, in the performance sheet of the corresponding task, a number of additional quality assurance points may be listed (specific points of interest). In the event that a quality assurance point is referred to in both, the strictest criterion applies.

Additionally, it is part of good practice to add standard and (procedural) blanks to the measurement series. The standards and procedure blanks should meet the following guidelines:

- If no dilution step is applied, a 1-point calibration solution should ideally be higher than 20 times the demonstrability limit and lower than 0.6 times the linear measurement range of the measurement system or method.
- If no dilution step is applied, a calibration curve must be built up from a number of measurement points that are distributed proportionately across the entire measurement range (e.g. 3 measurement points per decile) of the measurement system or method.
- A procedure blank must fulfil the requirements stated on the performance sheet or, if the reporting limit applied by the laboratory is lower, it must be smaller than this reporting limit.

The institution should have laid down in a document how it deals with heightened blanks, with regard to:

- increased reporting values;
- correction for the blank;
- the limiting value for "out-of-control" with the task.

SG5.1.4.1 Multiple determination (duplicate determination)

Analyse for which sub-samples have been taken, must be executed in duplicate before sample preparation has taken place. The duplicate determination is executed under repeatability conditions.

If the measurement value is higher than 20 times the demonstrability limit, a duplicate determination must meet the following criterion:

- The measurement results of the duplicate determinations may not differ from each other by more than 4 times the requirements of the repeatability standard deviation. If this requirement is relative, the difference between them in terms of percentage of the duplicate results is calculated on the basis of the mean result.

Remark: *The above comments do not relate to tasks for which, prior to the measurement, the part samples are separately reprocessed and then combined (volatile compounds, cyanide).*

With duplicate determinations, the mean value is reported.

SG5.1.5 Additional periodical quality assurance points

In order to prevent gross errors, an institution should introduce an additional periodical quality assurance point. For a number of tasks, this additional quality assurance point is prescribed in the performance sheet.

SG5.1.5.1 The application of procedural internal standards

Where possible, an institution may use internal standard(s) in order to detect gross errors.

Conditions for applying internal standard(s):

- The internal standard must be added (to the sample or extract of the sample) at an early stage, in order that it passes through the whole process.
- The internal standard must be representative of the analytes to be determined. These include labelled, dated compounds or a congener of the analytes to be determined.
- An internal standard can be applied where the task permits it.

A measurement value may only be corrected using an internal standard if this is stated in the defined task. The institution must establish, in a validation investigation, the limiting value the internal standard must meet; this must be reasonably aligned with the data for the relevant task as stated in the performance sheet.

SG5.1.5.2 Additional confirmation

If, during a determination, a non-specific detector is applied then, if possible, additional confirmation should be carried out, for example by using another detector. As a rule, instructions concerning this aspect are included in the stated standard under ´Method´ in the performance sheet. Criteria regarding additional confirmation are stated in the performance sheets.

Explanation: *An example of a specific detector is a mass-selective detector (MS).*

The purpose of additional confirmation is not to confirm results quantitatively but rather to confirm that the compound found has been justifiably and correctly identified.

If indicated on the performance sheet, additional confirmation of a determination must be executed on 2% of the samples. For this additional confirmation, a sample containing an analyte must be chosen.

The additional confirmation may either be executed on the destruate/extract, on the ascertained analysis sample for the analysis already performed, on the pre-treated sample material or on the field-moist sample material.

If the identification of the compounds is not confirmed by the additional confirmation, the institution should perform a more detailed investigation.

SG5.1.5.3 Consistency analysis of calibration solutions

Monitoring the trueness of a calibration solution occurs during the production of new calibration standards: this applies to both stock solutions and calibration solutions.
The trueness should be checked daily if new calibration solutions are prepared for each measuring series. Often, this is already being done with a control chart (control sample). However, if a calibration solution is used over a longer period of time, a once-only check of the trueness of the calibration solution following preparation will suffice.
In the consistency check, the newly prepared calibration solution is compared with the "old" calibration solution used up till then.

Criterion:
- The relative difference between the "old" and the newly-prepared calibration solutions must not be greater than 7.5%. In multi-component or multi-element calibration solutions, the relative difference may amount to 7.5%-12.5% for not more than one-fifth of the components or elements.

SG5.2 Second-line control

The laboratory must regularly check its tasks using a second-line control sample, the identity of which is as far as possible unknown. The frequency should be such that the sum of the second- and third-line checks is at least 4 in any year. If possible, at least one of these should be a third-line control (ring test).

For the second-line control, the preparation of the samples and the statistical processing should be laid down in working guidelines. The procedural aspects should also be recorded in a separate procedure.
For a second-line control, the following materials can be used:
- (internal) certified reference materials;
- samples with a conventional true value;
- spiked samples;
- blank material.

During the second-line control, repeatability and trueness must be tested against the requirements stated in the performance sheet. If the second-line control results do not correspond to the validation data, the institution should undertake corrective action.

Second-line control is necessary for non-routine task during those periods when the task is being performed.

SG5.3 Third-line control

If possible, the institution should regularly subject its accredited tasks to third-line control sample testing. For this, ring tests can be used.
The frequency should be such that a total of at least four second- and third- line checks are completed each year. Wherever possible, at least one of these should be a third-line (ring) test.
For the third line control, the procedural aspects should be laid down in a separate document.

For non-routine tasks, third line control is only necessary during those periods when the task is being performed.

SG5.3.1 Assessing the results of a ring test

The results of a ring test are evaluated in the following manner.

For each component, the z-score is calculated as follows:

$$z_i = \frac{x_i - x_{ref}}{s}$$

where:

x_i	the found value
x_{ref}	the assigned value (exclusive of outliers; in accordance with ISO 5725-2 and, for instance, a Grubbs test)

If the number of participants in a ring test is greater than 6, the standard deviation, s, is determined according to:

If $s_{W,eis} > s_{ring}$ then $s = s_{W,eis}$
If $s_{W,eis} < s_{ring}$ then $s = s_{ring}$

where:

$s_{W,eis}$	the requirement in terms of the intra-laboratory reproducibility standard deviation;
s_{ring}	the standard deviation in the assigned value across the ring test.
n	number of laboratories that participated in the ring test.

If the number of participants is less than 6, the standard deviation, s, is equal to $s_{W,eis}$. The z values are only calculated and evaluated for components with a level higher than five-times the determined AG. The result for a component, or group component, is significantly different from the assigned value, if:

- The absolute value of the z-score of one the observations is larger than 3 ($|z| > 3$).
- In a ring test consisting of 1 or 2 samples of the same matrix, the absolute value of the z-scores of one specific component is larger than 2 and lies on the same side of the mean for more than 2 samples in the most recent two ring tests, or for 2 samples within the most recent ring test.
- In a ring test consisting of 3 samples of the same matrix, the absolute values of the z-scores of one specific component is larger than 2 and lies on the same side of the mean for more than 2 samples within the most recent two ring tests or within one of these ring tests.
- In a ring test consisting of 4 samples of the same matrix, the absolute values of the z-scores of one specific component is larger than 2 and lies on the same side of the mean for more than 3 samples within the most recent two ring tests or within one of these ring tests.

In the table below, by way of illustration, the borderline cases have been entered. Firstly, an examination is performed of the latest ring test. Here, the maximum number of exceedances must be lower than that given in Column 2 and Column 3. If no significant deviation is found within the latest ring test or the previous one, then the most recent two investigations are tested against the requirements given in Columns 2 and 3.

The order given in the table is arbitrary and is of no significance in testing a significant deviation. The evaluation only concerns the number of exceedances and their type (II or III).

Table: Criteria for the evaluation of ring tests[1]

Number of samples to be evaluated	Allowed[2]	Not allowed[3]
1	II	III
2	I, II	II, II
		I, III
3	I, II, II	II, II, II
		I, I, III
4	I, I, II, II	I, II, II, II
		I, I, I, III
5	I, I, I, II, II	I, I, II, II, II
		I, I, I, I, III
6	I, I, I, I, II, II	I, I, I, II, II, II
		I, I, I, I, I, III
7	I, I, I, I, I, II, II, II	I, I, I, I, II, II, II, II
		I, I, I, I, I, I, III
8	I, I, I, I, I, II, II, II	I, I, I, I, II, II, II, II
		I, I, I, I, I, I, I, III

[1]) **I** = |z| < 2 sigma (content lies within the 95% reliability range), **II** = 2 sigma < |z| < 3 sigma (content lies outside the 95% reliability range) and **III** = |z| > 3 sigma (an exceedance, determine the cause); [2]) z-scores per parameter that are allowed within a single ring test; [3]) z-scores for each parameter from which the cause of the deviation must be determined.

When evaluating the table and determining whether the laboratory has undertaken timely action, the reporting time of the ring tests should be taken into account. It may be that the results of ring test n are only reported after the results of ring test n+1 have already been submitted. In this situation, it can only be determined whether the results of a given component in both ring test n and ring test n+1 contained deviations after the submission of ring test n+2 results. It is possible that the same deviation will also occur in ring test n+2 but, in the situation described, without the laboratory being at fault for not undertaking timely action.

By way of an illustration, the following diagram shows this graphically.

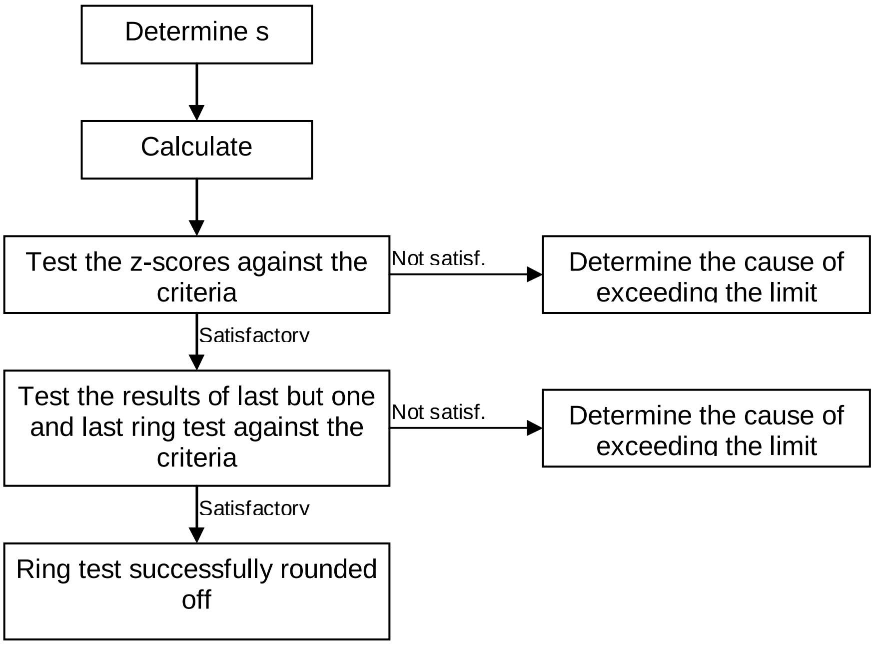

Figure Diagram of assessing ring test results

If a result deviates significantly, then the laboratory needs to investigate the possible cause.

During this investigation, the following actions should be undertaken:

- analysis of the quality problem, based on the results of the most recent successful ring tests, internal data on quality assurance and the relevant measurements;
- draw up a plan for corrective action;
- record the performance of the corrective action;
- check if the corrective action has been successful.

One of the following actions should be undertaken if, during the investigation into the deviation(s), no cause could be established:

- carry out a second-line control;
- if the self-reprocessed destruate/extract is still available, arrange for its contents to be determined by another laboratory with AP04 accreditation.

SG6 Technical description and quality assurance of tasks

Performance sheet SG.I Determination of the pH-$CaCl_2$ in soil

Principle

The analysis sample is extracted using a $CaCl_2$ solution in a ratio of 1:5 (v/v). The pH value is measured using a pH meter. A saturated calomel electrode is used as a reference.

Conformity with the standard

An execution based on this performance sheet is fully in line with the standard as stated here under ´Method´. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Method and quality assurance

	Soil	
METHOD		
Sampling	AP04-M, applicable VKB protocols	
Storage conditions	SIKB protocol 3001	
Storage period	SIKB protocol 3001	
Sample pre-treatment	AP04-V	
Storage conditions	SIKB protocol 3001	
Storage period	SIKB protocol 3001	
Task	Draft NEN 5750 ($CaCl_2$)	
Sample size	> 5 ml	
Multiple	N/A	
FIRST-LINE CONTROL		
Demonstrability limit	N/A	
Water	conductivity < 0.2 mS/m and pH > 5.6	
Control sample	soil sample with a pH between 6 and 8	
Recovery	N/A	
Repeatability standard deviation (expressed in s_r)		
	pH < 7.00	< 0.15
	7.00 < pH < 7.50	< 0.20
	7.50 < pH < 8.00	< 0.30
	pH > 8.00	< 0.40
Intra-laboratory reproducibility standard deviation (expressed in s_W)		
	pH < 7.00	< 0.15
	7.00 < pH < 7.50	< 0.20
	7.50 < pH < 8.00	< 0.30
	pH > 8.00	< 0.40

Additional quality assurance points

Proc. internal standards.	N/A
Confirmation	N/A

Specific points of interest
- Daily calibration

Comparative investigation

Samples containing analyte yes
in at least 12 samples the pH must lie between 4 and 6,
in at least 12 samples the pH must lie between 6 and 8,
in at least 12 samples the pH must lie between 8 and 10.
anomalous soil characteristics no
additional comparative experiments no

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample: between pH 2 and pH 12

Next performance sheet : pH

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : N/A
Concentration range of the samples : N/A
Soil : for institutions accredited for ring tests.

Reference materials
Concentration of the samples : N/A
Soil matrix : sand, clay, peat or a mixture of these types of soil
Soil samples from : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen (NMI-NBR),
National Institute of Standards and Technology (NIST).
Samples with a conventional true value,
Certified materials that have been analysed in line with the task as described in the performance sheet.

Performance sheet SG.II Determination of the dry matter content in soil

Principle

Air-dried soil

The analysis sample is dried to a constant mass for 6 hours at 105°C. The difference in mass before and after drying is a measure of the moisture content and also of the dry matter content.

Field-moist sample and ground sample

The analysis sample is dried to a constant mass for 16 hours at 105°C. The difference in mass before and after drying is a measure of the moisture content and also of the dry matter content.

Conformity with the standard

An execution based on this performance sheet is fully in line with the standard as stated here under ´Method´. If the work is carried out in conformance with the performance sheet, conformity with the standards may be claimed.

Method and quality assurance

	Field-moist soil	Air-dried soil
METHOD		
Sampling	AP04-M, applicable VKB protocols	AP04-M, applicable VKB protocols
Storage conditions	SIKB protocol 3001	SIKB protocol 3001
Storage period	SIKB protocol 3001	SIKB protocol 3001
Sample pre-treatment	AP04-V	AP04-V
Storage conditions	SIKB protocol 3001	SIKB protocol 3001
Storage period	SIKB protocol 3001	SIKB protocol 3001
Task	NEN 5747	Draft NEN 5748
Sample size	35 ± 5g	12.5 ± 2.5 g
Multiple	in duplicate	in duplicate (if not homogenised)
FIRST-LINE CONTROL		
Demonstrability limit	N/A	N/A
Blank	N/A	N/A
Control sample		
	2% in duplicate of Addition of H_20 (<20%) to sand	2% in duplicate of Addition of H_20 (<10%) to sand
Recovery	N/A	N/A
Repeatability standard deviation		
20 - 50% (m/m)	< 5 %	
50 - 80% (m/m)	< 2.5 %	< 2.5 %
80 - 100% (m/m)	< 2.5 %	< 2.5 %
Intra-laboratory reproducibility standard deviation		
20 - 50% (m/m)	< 5 %	
50 - 80% (m/m)	< 2.5 %	< 2.5 %
80 - 100% (m/m)	< 2.5 %	< 2.5 %
Additional quality assurance points		
Proc.internal standards.	N/A	N/A
Confirmation	N/A	N/A
Specific points of interest	N/A	

Continuation of performance sheet: Dry matter

Comparative investigation

Samples containing analytes yes, with anomalous soil characteristics
Anomalous soil characteristics yes

	Field-moist soil		Air-dried soil	
Dry matter	silt	organic matter	silt	organic matter
85 - 100 % (m/m)	< 15 %	< 20 % (m/m)	< 15 %	< 20 % (m/m)
70 - 85 % (m/m)	< 20 %	> 20 % (m/m)	< 20 %	> 20 % (m/m)
70 - 85 % (m/m)	> 20 %	< 20 % (m/m)	> 20 %	< 20 % (m/m)
50 - 70 % (m/m)	10 - 45 %	< 20 % (m/m)	10 - 45 %	< 20 % (m/m)
50 - 70 % (m/m)	10 - 45 %	> 20 % (m/m)	10 - 45 %	> 20 % (m/m)
20 - 55 % (m/m)	< 45%	> 50 % (m/m)	< 45 %	> 50 % (m/m)

All the combinations noted above must be tested with at least 10 different samples in accordance with Section 4.2.2.

Additional comparative experiments no

SECOND- AND THIRD-LINE CONTROL N/A

Performance sheet SG.III Determination of the silt content of soil

Principle

The analysis sample is pre-treated with hydrogen peroxide in order to oxidise any organic matter present. Subsequently, hydrochloric acid is added to remove the carbonates and adhesive constituents present.
A suspension is made of the sample, to which a peptisator (a solutions of sodium pyrophosphate) is added. The silt fraction is determined using the pipette method.

Conformity with the standard

An execution based on this performance sheet is fully in line with the standard as stated here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard can be claimed.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	N/A (SIKB protocol 3001)
Storage period	N/A (SIKB protocol 3001)
Sample pre-treatment	AP04-V
Storage conditions	N/A (SIKB protocol 3001)
Storage period	N/A (SIKB protocol 3001)
Task	Draft NEN 5753
Related task	Draft NEN 5750 (pH-CaCl$_2$), draft NEN 5754 (organic matter) and NEN-ISO 10693 (calcite)
Sample size	>20 g (see criteria in draft NEN 5753)
Multiple	in duplicate

Task is determined by method.

FIRST-LINE CONTROL	
Demonstrability limit	< 1 wt.%
Control sample	
silt content	7 - 15 wt.% silt
Recovery	
silt (<10 wt.%)	60 - 120 %
silt (>10 wt.%)	90 - 110%
Repeatability variation coefficient	
silt	< 10%
Intra-laboratory reproducibility variation coefficient	
silt	< 15%
Additional quality assurance points	
proc. internal standards	N/A
confirmation	N/A

Specific points of interest

- **Method-determined parameter**
- **During validation tests, sedimentation step with certified reference material or reference material with a conventionally true value**

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample: 2 - 15 wt.%

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : 1 wt.%
Concentration range of the samples : 2 - 15 wt.%
Soil : for institutions accredited for ring tests.

Continuation of performance sheet: Silt content

Reference materials

Concentration of the samples : 2 - 15 wt.%
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples from : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
National Institute of Standards and Technology (NIST),
Samples with a conventionally true value,
Certified materials that have been analysed following the task described in the performance sheet.

Performance sheet SG.IV Determination of the organic matter content in soil

Principle

After having been dried at 105 °C, the analysis sample is incinerated at 550 °C. The loss of mass at 550 °C is a measure of organic matter content. The result is corrected for the water bound to clay minerals and, if more than 5 % (m/m) free iron (expressed as Fe_2O_3) is anticipated, also for the water bound to the iron.

Conformity with the standard

An execution based on this performance sheet is fully in line with the standard as stated here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard can be claimed.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB protocols
Storage conditions	SIKB protocol 3001
Storage period	SIKB protocol 3001
Sample pre-treatment	AP04-V
Sample size	>8 kg
Storage conditions	SIKB protocol 3001
Storage period	SIKB protocol 3001
Task	Draft NEN 5754 with the exception of pre-treatment
Related task	NEN 5739 (determining free iron: for this, measurement of iron using ICP-AES is allowed):
	Draft NEN 5753 (silt)
Sample size	10 ± 2g
In multiple	duplicate

FIRST-LINE CONTROL

Demonstrability limit	< 0.2%
Blank (absolute value)	N/A
Control sample (control of the dispersion)	2% of samples in duplicate or soil sample with an organic matter content between 5 and 15%
Recovery	N/A
Repeatability standard variation coefficient	
0 - 5%	< 0.75% (absolute)
5 - 15%	< 7 % (vc_r)
> 15%	< 6 % (vc_r)
Intra-laboratory reproducibility standard variation coefficient	
0 - 5%	< 1.0 % (absolute)
5 - 15%	< 10 % (vc_W)
> 15%	< 8 % (vc_W)
Additional quality assurance points	
Proc. internal standards.	N/A
Confirmation	N/A
Specific point of interest	N/A
Comparison investigation	
samples containing analytes	yes
deviating soil characteristics	yes

Field-moist soil

silt	organic matter
< 15 %	< 20 % (m/m)
< 20 %	> 20 % (m/m)
> 20 %	< 20 % (m/m)
10 - 45 %	< 20 % (m/m)
10 - 45 %	> 20 % (mm)
< 45 %	> 50 % (m/m)

additional comparison experiments no

SECOND- and THIRD-LINE CONTROL

N/A

Performance sheet SG.V Determination of elements in soil

Principle

The analysis sample is rendered soluble with aqua regia. The contents of elements are measured with atom absorption spectrometry (reverberatory furnace, graphite furnace or hydrid technique), ICP-AES or ICP-MS.

Conformity with the standard

The execution based on this performance sheet is completely in line with the standard as stated here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds) target value-[1]	intervention value-[2]	AG $_{eis}$
Lead	7439-92-1	52	530	13
Cadmium	7440-43-9	0,54	12	0,17
Zinc	7440-66-5	53	720	17
Nickel	7440-02-0	10	210	3
Arsenic	7440-38-2	15,8	55	4
Chromium	7440-47-3	50	380	15
Copper	7440-50-8	16,2	190	5
Molybdenum	7439-98-7	10	200	1,5
Antimony	7440-36-0	3	15	1
Barium	7440-39-3	38,7	625	15
Selenium	7782-49-2	0,7	100	10
Vanadium	7440-62-2	42	250	1
Tin	7440-31-5	20	-	6
Cobalt	7789-43-7	4,44	240	1

[1] The target value is based on soil containing 2% organic matter and 0% silt

[2] The intervention value is based on soil containing 10% organic matter and 25% silt

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	N/A (SIKB-protocol 3001)
Storage period	N/A (SIKB-protocol 3001)
Sample pre-treatment	AP04-V
Sample size	> 8 kg
Storage conditions	N/A (SIKB-protocol 3001)
Storage period	N/A (SIKB-protocol 3001)
Task	NEN 5758, NEN 5759, NEN 5760, NEN 5761, NEN 5762, NEN 5765, NEN 5767, draft-NEN 6427, NVN 7321 and NVN 7322.
Related task	NEN 6465, NVN 5770 or draft-NEN 6961 (dissolution)
Sample size	> 5 g[1]
FIRST-LINE CONTROL	
Demonstrability limit	< AG$_{eis}$ (see above under "Analytes to be determined")
Blank	< AG$_{eis}$
Control sample	
Analyte(s)	all

Continuation of performance sheet: Elements in soil

Recovery

Zinc	80 - 110%
Lead	80 - 110%
Cadmium	80 - 110%
Nickel	80 - 110%
Arsenic	80 - 110%
Chromium	80 - 110%
Copper	80 - 110%
Molybdenum	80 - 110%
Antimony	80 - 110 %
Barium	80 - 110%
Selenium	80 - 110 %
Tin	80 - 110%
Vanadium	80 - 110 %
Cobalt	80 - 110 %

Repeatability variation coefficient

Zinc	< 7,5 %
Lead	< 7,5 %
Cadmium	< 7,5 %
Nickel	< 10 %
Arsenic	< 7,5 %
Chromium	< 10 %
Copper	< 7,5 %
Molybdenum	< 7,5 %
Antimony	< 7,5 %
Barium	< 10 %
Selenium	< 7,5 %
Tin	< 7,5 %
Vanadium	< 10 %
Cobalt	< 7,5 %

Intra-laboratory reproducibility variation coefficient

Zinc	< 10 %
Lead	< 10 %
Cadmium	< 10 %
Nickel	< 15 %
Arsenic	< 10 %
Chromium	< 15 %
Copper	< 10 %
Molybdenum	< 10 %
Antimony	< 10 %
Barium	< 15 %
Selenium	< 10 %
Tin	< 10 %
Vanadium	< 15 %
Cobalt	< 10 %

Additional quality assurance points

Proc. internal stand.	Yes (5.1.5.1)
Confirmation	perform measurement with CP-AES at different wavelengths

Specific points of interest

N/A

Comparison investigation

<u>samples containing analytes</u>	yes (all)
<u>deviating soil characteristics</u>	N/A
<u>additional comparison experiments</u>	N/A

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly for : all

Continuation of performance sheet: Elements in soil

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
National Institute of Standards and Technology (NIST),
Nederlands Instituut voor referentiematerialen(NMI-NBR),
Samples with a conventional true value
Certified materials that have been analysed according to the task stated in the performance sheet.

[1] If the maximum amount of organic matter stated in the standard (0,25 or 0,5 gram) is exceeded, proportionally more acid must be applied. If in a closed dissolution the maximum amount of the sample per vessel is exceeded, more vessels must be used. After dissolution, the extracts must be combined.

Performance sheet SG.VI Determination of non volatile mercury in soil

Principle

The analysis sample is rendered soluble with aqua regia. In the destruate, mercury is reduced to volatile atomic mercury with tin (II)chloride. The mercury is brought out of the solution by vaporization.
The content of mercury is measured by means of atom absorption spectrometry or fluorescence spectrometry.

Conformity with the standard

The execution based on this performance sheet is completely in line with the standard as stated here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		
		target value-1)	intervention value-2)	$AG_{,eis}$
Mercury	7439-92-1	0,20	10	0,05

1) The target value is based on soil containing 2% organic matter and 0 % silt.
2) The intervention value is based on soil containing 10% organic matter and 25% silt.

Method and quality assurance

		Soil
METHOD		
Sampling		AP04-M, applicable VKB-protocols
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Sample pre-treatment		AP04-V
	Sample size	> 8 kg
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Task		NEN-ISO 16772
	Related task	NEN 6465, NVN 5770 or draft-NEN 6961 (dissolution)
	Sample size	> 5 g1)
FIRST-LINE CONTROL		
Demonstrability limit		< AG_{eis} (see above under “Analytes to be determined”)
Blank		< AG_{eis}
Control sample		
	Analyte(s)	soil sample containing mercury
Recovery		
	mercury	80 - 110%
Repeatability variation coefficient		
	mercury	< 10 %
Intra-laboratory reproducibility variation coefficient		
	mercury	< 15 %
Additional quality assurance points		
	Proc. internal stand.	N/A
	Confirmation	N/A
Specific points of interest		N/A
Comparison investigation		
<u>samples containing analytes</u>		yes
<u>deviating soil characteristics</u>		N/A
<u>additional comparison experiments</u>		N/A

Continuation of performance sheet: Kwik

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly on : mercury

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiesamples (NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

1) If the maximum amount of organic matter stated in the standard (0,25 or 0,5 gram) is exceeded, proportionally more acid must be applied. If in a closed dissolution the maximum amount of the sample per vessel is exceeded, more vessels must be used. After dissolution, the extracts must be combined.

Performance sheet SG.VII Determination of cyanides (free and total) in soil

Principle

Pre-treatment of soil

The analysis sample is extracted with caustic soda. The content of free and/or total cyanide is measured by means of photometry.

Determination of the total content of cyanide

Under the influence of UV-light, complex bound cyanide is converted into free cyanide-ions. At a pH of 3,8, these are, together with the free cyanide-ions already present, overdistilled at a pH of 3,8. The content of cyanide is photometrically measured after the conversion of the cyanide by means of pyridine-barbituric acid.

Determination of the content of free cyanide

Free cyanide-ions are overdistilled at a pH of 3,8. The content of cyanide is photometrically measured after conversion of the cyanide by means of pyridine-barbituric acid.

Conformity with the standard

The execution based on this performance sheet is completely in line with the standard as stated here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		
		target value-1)	intervention-value-1)	AG_{eis}
Cyanide (total-free)		1	20	1
Cyanide (total-complex)		5	50	1

---

1) The target- and intervention values are based on 10% organic matter and 0% silt

Method and quality assurance

		Soil
METHOD		
Sampling		AP04-M, applicable VKB-protocols
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Sample pre-treatment		**AP04-V**
	Sample size	> 8 kg
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Task		NEN 6655
	Related task	-
	Sample size	> 45 g
	Multiple	extraction in quadruplicate, analysis simple

FIRST-LINE CONTROL
Demonstrability limit < AG_{eis} (see above under "Analytes to be determined")
Blank < AG_{eis}
Control sample
Analytes $K_3Fe(CN)_6$, Thiocyanate, KCN
Recovery
Free cyanide
$K_3Fe(CN)_6$ 0 - 7 %
Thiocyanate < 5 µg/l (< 0,5 %, starting from 1000 µg/l in the extract)
KCN 80 - 110 %
Total cyanide
$K_3Fe(CN)_6$ 80 - 110 %
Thiocyanate < 10 µg/l (< 1 %, starting from 1000 µg/l in the extract)

Continuation of performance sheet: Cyanides

Repeatability variation coefficient
Free cyanide < 10 %
Total cyanide < 10 %
Intra-laboratory reproducibility variation coefficient
Free cyanide < 15 %
Total cyanide < 15 %
Additional quality assurances points
Internal standard N/A
Confirmation N/A
Specific points of interest
N/A
Comparison investigation
samples containing analytes yes
for free cyanide and total cyanide
deviating soil characteristics no
additional comparison experiments yes
10 samples with 100 mg/l sulfide.

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly on : $K_3Fe(CN)_6$, Thiocyanate, KCN

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.VIII Determination of volatile aromatic hydrocarbons and volatile halogened hydrocarbons in soil

Principle

The analysis sample is extracted in quadruplicate with methanol. The extracts are blended into one extract mixture. Part of the extract is diluted with water. The volatile compounds are pushed out of the solution with nitrogen gas. The content of volatile compounds are measured with a gaschromatograph with FID- and ECD-detection or with a mass spectrometer.

Conformity with the standard

The execution based on this performance sheet is different (optionally)from the standard as laid down here under ´Method´. If the work is performed in conformance with the performance sheet, conformity with the standard may only be claimed, if the measurement is carried out with FID/ECD-detectors. If the measurement is performed with a MS-detector, equivalence may be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (μg/kg.ds) target value-[1]	intervention-value-[2]	AG $_{eis}$
Volatile aromatic hydrocarbons				
Benzene	71-43-2	10	1000	50
Toluene	108-88-3	10	130000	100
Ethylbenzene	100-41-4	10	50000	50
o-Xylene	95-47-6	-	-(2000)[3]	100
m-Xylene	108-38-3	-	-(2000)[3]	100
p-Xylene	106-42-5	-	-(2000)[3]	100
Sum of xylenes		10	25000	
Styrene	100-42-5	20	100000	100
Volatile halogened hydrocarbons				
Dichloromethane	75-09-2		20000	500
Trichloromethane	67-66-3	0,2	10000	50
Tetrachloromethane	56-23-5	0,2	1000	50
Trichloro-ethene	79-01-6	0,2	60000	50
Tetrachloro-ethene	127-18-4	2	4000	10
1,1-Dichloro-ethane	75-74-3	-	-(1000)[3]	500
1,2-Dichloro-ethane	107-06-2	-	4000	500
Sum of dichloro-ethanes				
1,1-Dichloro-ethene[4]	75-35-4	-	-(1000)[3]	500
cis 1,2-Dichloro-ethene	156-59-2	-	-(1000)[3]	500
trans 1,2-Dichloro-ethene	156-60-5	-	-(1000)[3]	500
Sum of dichloro-ethenes				
1,1,1-Trichloro-ethane	79-01-6	-	-(1000)[3]	50
1,1,2-Trichloro-ethane	79-00-5	-	-(1000)[3]	50
Sum of trichloro-ethanes		0,2		

[1] The target value is based on soil containing 2% organic matter and 0% silt.
[2] The intervention value is based on soil containing 10% organic matter and 25% silt.
[3] In the building materials decree no intervention value is given. The value given is applied in validation and quality assurance.
[4] The component has not been validated for the standard in question.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001

Continuation of performance sheet: Hydrocarbons

Sample pre-treatment AP04-V
Related task NEN 5747 (dry matter)
Storage conditions N/A (SIKB-protocol 3001)
Storage period N/A (SIKB-protocol 3001)
Task NEN-ISO 15009
Sample size 50 ± 2 g
Multiple extraction in quadruplicate,
analysis simple.

FIRST-LINE CONTROL
Demonstrability limit $< AG_{eis}$ (see above under "Analytes to be determined")
Blank $< AG_{ag}$
Control sample
Analyte(s) dichloromethane, benzene
Tetrachloroethene
Recovery
Dichloromethane 70 - 120%
Other 70 - 115%
Repeatability variation coefficient
All < 20%
Intra-laboratory reproducibility variation coefficient
All < 25%
Additional quality assurance points
Internal standard Yes (5.1.5.1)
Confirmation Yes, 2 % of the samples if FID- and/or ECD-detection is applied (see 5.1.5.2). Confirmation by means of. GCMS or column with different polarity (see NEN-ISO 15009).
Specific points of interest
- **Chromatografic analysis**

Selectivity: See NEN-ISO 15009, if GC-MS is applied, oly for analytes with the same m/z
Comparison investigation
samples containing analytes yes
for benzene, toluene, xylenes, trichloro-ethene and tetrachloro-ethene
deviating soil characteristics N/A
additional comparison experiments yes
For the other analytes the equivalence is determined by means of addition to soil(SG4.2) with an extraction agent.

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample : within the measurement range of the task
Investigate biannually on : benzene, toluene, dichloromethane and monochlorobenzene

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration range of the samples : within the measurement range of the task
Matrix : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau van Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.IX Determination of Polycyclic Aromatic Hydrocarbons (PAH) in soil

Principle

For determining the PAH-compounds, two methods are possible.

NVN 5731:

The analysis sample is extracted with aceton and petrol ether. If disturbing compounds are expected, the extract is purified over a column of aluminium oxide. The content of PAH-compounds is measured with a HPLC with UV- and/or fluorescence detection.

Draft-NVN 5710:

The analysis sample is extracted with aceton. The aceton extract is brought over a solid-phase column. After in-line elution, the content of PAH-compounds is measured with an HPLC by means of UV- and/or fluorescence detection.

Conformity with the standard

The execution based on this performance sheet is completely in line with the standard as stated here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the related standard can be claimed.

Analytes to be determined

name	CAS-number	soil(mg/kg.ds) target value-[1]	intervention-value-[2]	AG $_{eis}$
naphtalene	91-20-3	-	-	0,01
phenantrene	85-01-8	-	-(2)[3]	0,01
antracene	120-12-7	-	-	0,01
fluorantene	206-44-0	-	-	0,01
benzo(a)antracene	56-55-3	-	-(2)[3]	0,01
chrysene	218-01-9	-	-	0,01
benzo(k)fluorantene	207-08-9	-	-	0,01
benzo(a)pyrene	50-32-8	-	-(2)[3]	0,01
benzo(ghi)perylene	191-24-2	-	-(2)[3]	0,01
indeno(1,2,3-cd)pyrene	193-39-5	-	-	0,01
PAH (sum 10)		1,0	40	

[1] The target value is based on soil containing 2% organic matter and 0% silt.
[2] The intervention value is based on soil containing 10% organic matter and 25% silt.
[3] In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

Method and quality assurance

		Soil
METHOD		
Sampling		AP04-M, applicable VKB-protocols
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Sample pre-treatment		AP04-V
	Related task	NEN 5747 (dry matter)
	Sample size	250 ± 13 g
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Task		NVN 5731, draft- NVN 5710
	Sample size	> 20 g
	In multiple	N/A
FIRST-LINE CONTROL		
Demonstrability limit		< AG $_{eis}$ (see above under “Analytes to be determined”)
Blank		< AG $_{eis}$

Continuation of Performance sheet: Polycyclic aromatic hydrocarbons

Control sample

Analyte(s)	naphtalene, phenantrene, benzo(k)fluorantene and indeno (1,2,3-cd)pyrene

Recovery

Naphtalene	60 - 110%
Phenantrene	60 - 110%
Acenaphtylene and acenaphtene	60 - 110%
Other	85 - 110%

Repeatability variation coefficient

Naphtalene	< 15%
Phenantrene	< 15%
Acenaphtylene and acenaphtene	< 15%
other	< 15%

Intra-laboratory reproducibility variation coefficient

Naphtalene	< 20%
Phenantrene	< 20%
Acenaphtylene and Acenaphtene	< 20%
Other	< 20%

Additional quality assurance points

Proc.internal stand.	Yes (5.1.5.1)
Confirmation	Yes, 2 % of the samples if UV or fluorescence detection is used (see 5.1.5.2). Confirmation with MS- or fluorescence detection if UV-detection is used. Confirmation with MS- or UV-detection if fluorescence detection is used. See NVN 5731 or draft-NVN 5710.

Specific points of interest

- Chromatografic analysis

Selectivity:

See NVN 5731 or draft-NVN 5710.

Comparison investigation

samples containing analytes	yes (all)
deviating soil characteristics	N/A
additional comparison experiments	N/A

SECOND-LINE CONTROL

Soil

Concentration range of the analytes in the sample :within the measurement range of the task
Investigate analytes (at least) quarterly on :naphtalene, phenanthrene , benzo(k)fluorantene and indeno(1,2,3-cd)-pyrene
investigate biannually on :all

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration range of the samples : within the measurement range of the task
Matrix : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Bureau of Standards (NBS),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials, that have been analysed according to the task described in the performance sheet.

Performance sheet SG.X Determination of Extractable Organohalogene compounds (EOX) in soil

Principle

The analysis sample is extracted with aceton and petrol ether. The extract is condensed. The content of organically bound chlirde is measured with a microcoulometer.

Conformity with the standard

The performance based on this performance sheet is completely in line with the standard as mentioned here under ´Method´. If a procedure in conformance with the performance sheet is pursued, conformity with the related standard may be claimed.

Analytes te be determined

name	CAS-number	soil/sediment (mg/kg.ds) target value1)	intervention-value2)	AG eis
EOX (Aldrin)	(390-00-2)	0,02	-(6)3)	0,1

1) The target value is based on soil containing 2% organic matter and 0% silt.
2) The intervention value is based on soil containing 10% organic matter and 25% silt.
3) In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

Method and quality assurance

Soil

METHOD

Sampling AP04-M, applicable VKB-protocols
- Storage conditions SIKB-protocol 3001
- Storage period SIKB-protocol 3001

Sample pre-treatment AP04-V
- Related task NEN 5747 (dry matter)
- Sample size 250 ± 13 g
- Storage conditions SIKB-protocol 3001
- Storage period SIKB-protocol 3001

Task NEN 5735
- Sample size > 20 g
- multiple N/A

Task determines the method.

FIRST-LINE CONTROL

Demonstrability limit < AG_{eis} (see above “Analytes to be determined”)

Blank < AG_{eis}

Control sample
- Analyte(s) soil sample containing EOX

Recovery
- EOX(Aldrin) 80 - 110%

Repeatability variation coefficient
- EOX(Aldrin) < 15%

Intra-laboratory reproducibility variation coefficient
- EOX(Aldrin) < 20%

Additional quality assurances points
- Internal standard N/A
- Confirmation N/A

Specific point of interest

Parameter determining the method

Calibration equipment

Daily check of Aldrin standard solution (control chart if necessary).

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample :within the measurement range of the task

Analytes to be investigated every quarter on :Aldrin spiked soil sample
Continuation of Performance sheet: EOX

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Bureau of Standards (NBS),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet

Performance sheet SG.XI Determination of mineral oil in soil

Principle

The analysis sample is extracted with aceton and petrol ether. The extract is purified by means of florisil and condensed if necessary. The content of mineral oil is measured with a gaschromatograph with FID-detection.

Explanation:

Definition of mineral oil: compounds that can be isolated with petrol ether under the conditions of this standard and that can be chromatographed with reference times that lying between the reference times of n-decane and n-tetracontane.

Conformity with the standard

The execution based on this performance sheet is completely in line with the standard as mentioned here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	soil/sediment(mg/kg.ds) target value-[1]	intervention-value-[2]	AG $_{eis}$
Mineral oil	-	10	5000	20

[1] The target value is based on soil containing 2% organic matter and 0% silt.
[2] The intervention value is based on soil containing 10% organic matter and 25% silt.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Sample pre-treatment	AP04-V
Related task	NEN 5747 (dry matter)
Sample size	250 ± 13 g dry matter in accordance with NEN 5747
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Task	NEN 5733
Sample size	> 20 g
In multiple	N/A

Task is determined by the method.

FIRST-LINE CONTROL	
Demonstrability limit	< AG $_{eis}$ (see above under “Analytes to be determined”) Based on mixture of gas- and motoroil
Blank	< AG $_{eis}$
Control sample	
Analyte(s)	soil sample containing gas- and motoroil
Recovery	70 - 110%
Repeatability variation coefficient	< 15%
Intra-laboratory reproducibility variation coefficient	
Mixture of gas- and motoroil	< 20%
Additional quality assurance points	
Proc.internal stand.	N/A
Confirmation	N/A

Specific points of interest

- **parameter determined by method**
- **Calibration equipment**

  Respons ratio C40/C20 must be higher than 0,85.

Quantification on RIVM-standard.

Continuation of performance sheet: Mineral oil

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes (at least) quarterly on : gasoil spiked soil sample

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value,
Certified materials that have been analysed according to the task described in the performance sheet

Performance sheet SG.XII Determination of bromide in soil

Principle

100 ml water is added to the analysis sample. This is being shaken for 1 hour. The extract is filtered over a folding filter, after which the content of bromide is photometrically determined by means of an ionchromatograph.

Conformity with the standard

The execution based on this performance sheet partly differs from the standard mentioned here under ´Method´. As the standard has a different application range (water), no conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment(mg/kg.ds)		
		target value-[1]	intervention-value-[1]	AG_{eis}
Bromide		20	-(600)[2]	5

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[1] The target- and intervention value are based on soil containing 10% organic matter and 25% silt.

[2] In the Building materials decree no intervention value is given. The value given has been applied to the validation and quality assurance.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Sample pre-treatment	AP04-V
Related task	-
Sample size	> 8 kg
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Task	VPR C85-06 and NEN-EN-ISO 10304-2 (measurement). For filtration a 0.45 µm filter must be applied.
Sample size	> 10 g (see AP04-V)
multiple	N/A
FIRST-LINE CONTROL	
Demonstrability limit	< AG_{eis} (see “Analytes to be determined” above)
Blank	< AG_{eis}
Control sample	
Analytes	bromide
Recovery	
Bromide	85 - 110%
Repeatability variation coefficient	
Bromide	< 7,5%
Intra-laboratory reproducibility variation coefficient	
Bromide	< 10%
Additional quality assurance points	
Internal standard	N/A
Confirmation	N/A
Specific points of interest	
	N/A
Comparison investigation	
samples containing analytes	yes
deviating soil characteristics	N/A
additional comparison experiments	N/A

Continuation of performance sheet: Bromide

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly on : All

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value,
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XIII Determination of total inorganic fluoride in soil

Principle

10 g soil is suspended in 100 ml water. The suspension is distilled. Subsequently, the distillate is determined potentiometrically. Both free and complex bound fluoride is determined with this method.

Conformity with the standard

The execution based on this performance sheet partly differs from the standard described here under ´Method´. As the standard has a different application range (water), no conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		
		target value-1)	intervention-value-2)	AG$_{eis}$
Fluoride		175	-(2000)3)	50

1) The target value is based on soil containing 2% organic matter and 0% silt.
2) The intervention value is based on soil containing 10% organic matter and 25% silt.
3) In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Sample pre-treatment	AP04-V
Related task	-
Sample size	> 8 kg
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Task	VPR C85-03 (distillation), NEN 6483 (meting) of NEN 6589 (meting)
Sample size	> 10 g
In multiple	N/A
FIRST-LINE CONTROL	
Demonstrability limit	< AG$_{eis}$ (see “Analytes to be determined” above)
Blank	< AG$_{eis}$
Control sample	
Analytes	fluoride
Recovery	
Fluoride	70 - 110%
Repeatability variation coefficient	
Fluoride	< 5%
Intra-laboratory reproducibility variation coefficient	
Fluoride	< 7,5%
Additional quality assurance points	
Internal standard	N/A
Confirmation	N/A
Specific points of interest	N/A
Comparison investigation	
samples containing analytes	yes
deviating soil characteristics	N/A
additional comparison experiments	N/A

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly on : All

Continuation of performance sheet: Fluoride

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XIV Determination of the content of chloride in soil

Principle

100 ml water is added to the analysis sample. This is shaken for 1 hour. The extract is filtered through a folding filter, after which the chloride content is determined by means of potentiometric titration, by means of an ionchromatograph or with flow analysis.

Conformity with the standard

The execution based on this performance sheet partly differs from the standards described here under ´Method´. As the standards have a different application range (water), no conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		
		target value-[1)]	intervention-value-[1)]	AG $_{eis}$
Chloride		200	-(2000)[2)]	50

[2)] The target- and intervention values are based on soil containing 10% organic matter and 25% silt.

[3)] In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

Method and quality assurance

METHOD

Sampling		AP04-M, applicable VKB-protocols
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Sample pre-treatment		AP04-V
	Related task	dry matter in accordance with NEN 5747
	Sample size	> 8 kg
	Storage conditions	SIKB-protocol 3001
	Storage period	SIKB-protocol 3001
Task		VPR C85-06 (extraction), NEN 6476, NEN-EN-ISO 10304-2 and NEN-EN-ISO 15682 (measurement). For filtration a 0,45 µm filter must be applied.
	Sample size	> 10 g
	In multiple	N/A

FIRST-LINE CONTROL

Demonstrability limit		< AG $_{eis}$ (see “Analytes to be determined” above)
Blank		< AG $_{eis}$
Control sample		
	Analytes	chloride
Recovery		
	Chloride	90 - 110%
Repeatability variation coefficient		
	Chloride	< 5%
Intra-laboratory reproducibility variation coefficient		
	Chloride	< 7,5%
Additional quality assurance points		
	Internal standard	N/A
	Confirmation	N/A
Specific points of interest		
		N/A

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly on : All

Continuation of performance sheet: Chloride

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value.
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XV Determination of chlorophenols in soil

Principle

Extraction of soil

The analysis sample (ca. 50 g) is suspended in 50 ml 1 mol/l hydrochloric acid. The suspension is extracted twice, each time with 50 ml of toluene. The extracts are combined.

Clean-up extract

The toluene extract is shaken three times with a potassium carbonate solution. This inorganic phase is shaken with a solution of hydrochloric acid anhydride in hexane. In this way, derivatising of the chlorophenols occurs. The content of chlorophenols is measured from the hexane phase by means of a gaschromatograph with ECD-detection.

Conformity with the standard

The procedure based on this performance sheet is completely in line with the practical guideline as described here under ´Method´. If the work is carried out in accordance with the performance sheet, conformity with the practical guideline may be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		
		target value-[1]	intervention value-[2]	AG $_{eis}$
2-Chlorophenol	95-57-8		-(2)[3]	0,01
3-Chlorophenol	108-43-0		-(2)[3]	0,01
4-Chlorophenol	106-48-9		-(2)[3]	0,01
sum of monochlorophenols		0,0005	-	
2,3-Dichlorophenol	576-24-9		-(2)[3]	0,001
2,4-Dichlorophenol	120-83-2		-(2)[3]	0,001
2,5-Dichlorophenol	583-78-8		-(2)[3]	0,001
2,6-Dichlorophenol	87-65-0		-(2)[3]	0,001
3,4-Dichlorophenol	95-77-2		-(2)[3]	0,001
3,5-Dichlorophenol	591-35-5		-(2)[3]	0,001
sum of dichlorophenols		0,0006	-	
2,3,4-Trichlorophenol	15950-66-0		-(2)[3]	0,0005
2,3,5-Trichlorophenol	933-78-8		-(2)[3]	0,0005
2,3,6-Trichlorophenol	933-75-5		-(2)[3]	0,0005
2,4,5-Trichlorophenol	95-95-4		-(2)[3]	0,0005
2,4,6-Trichlorophenol	88-06-2		-(2)[3]	0,0005
3,4,5-Trichlorophenol	609-19-8		-(2)[3]	0,0005
som of trichlorophenols		0,0002	-	
2,3,4,5-Tetrachlorophenol	4901-51-3		-(2)[3]	0,0005
2,3,4,6-Tetrachlorophenol	58-90-2		-(2)[3]	0,0005
2,3,5,6-Tetrachlorophenol	935-95-5		-(2)[3]	0,0005
som of tetrachlorophenols		0,0002	-	
Pentachlorophenol	87-86-5	0,0004	5	0,0005
sum of chlorophenols			10	

[1] The target value is based on soil containing 2% organic matter and 0% silt.
[2] The intervention value is based on soil containing 10% organic matter and 25% silt.
[3] In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

Continuation of performance sheet: chlorophenols

Method and quality assurance

Soil

METHOD

Sampling AP04-M, applicable VKB-protocols
Storage conditions SIKB-protocol 3001
Storage period SIKB-protocol 3001
Sample pre-treatment AP04-V
Related task NEN 5747 (dry matter)
Storage conditions SIKB-protocol 3001
Storage period SIKB-protocol 3001

Task VPR C85-14
Sample size > 45 g
In multiple Yes, extraction in quadruplicate, analysis simple

FIRST-LINE CONTROL
Demonstrability limit < AG_{eis} (see "Analytes to be determined" above)
Blank < AG_{eis}
Control sample
Analyte(s) 2-chlorophenol, 2,4,5-trichlorophenol and pentachlorophenol
Recovery
Monochlorophenols 65 - 110 %
Other 70 - 110 %
Repeatability variation coefficient
All < 10 %
Intra-laboratory reproducibility variation coefficient
All < 15 %
Additional quality assurance points
Internal standard N/A
Confirmation Yes, 2 % of the samples (see 5.1.5.2). Confirmation regarding GCMS.
Specific points of interest
- Gaschromatografic analysis
Selectivity:
Trennzahl (between the monochlorophenols) > 0.0
Trennzahl between analytes not mentioend must be at least > 2,0, except the one between 2,4- and 2.5-dichlorophenol.
Comparison investigation
samples containing analytes N/A
deviating soil characteristics N/A
additional comparison experiments N/A
soil characteristics of paragraph 4.2 with addition of all components (see 4.1.4.2)

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes (at least) quarterly on : 2-chlorophenol, 2,4,5-trichlorophenol and pentachlorophenol
Investigate analytes biannually on : all

THIRD-LINE CONTROL
Ring tests
Reporting limit ringtest : $AG_{, eis}$
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration range of the samples : within the measurement range of the task
Matrix : sand, clay, peat or mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR)
National Institute of Standards and Technology (NIST),
Samples with a conventional true value.
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XVI Determination of polychlorobiphenyles (PCB) and organochloride pesticides (OCP) in soil

Principle

The analysis sample is extracted with aceton and petrol ether. The extract is purified over an aluminiumoxide and, if PCBs are present, a silicagel column. The content of OCP amd PCB is measured by means of gaschromatography and ECD-detection.

Conformity with the standard

The procedure based on this performance sheet is completely in line with the standard as described here under 'Method´. If the work is carried out in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds) target value-[1]	intervention-value-[2]	AG_{eis}
PCB 28 (2,4,4'-trichloorbifenyl)	7012-37-5	0,0002	-(0,3)[3]	0,002
PCB 52 (2,5,2',5'- tetrachlorobiphenyl)	35693-99-3	0,0002	-(0,3)[3]	0,002
PCB 101 (2,4,5,2',5'-pentaCB)	37680-37-2	0,0008	-(0,3)[3]	0,002
PCB 138 (2,3,4,2',4',5'-hexaCB)	35065-28-2	0,0008	-(0,3)[3]	0,002
PCB 153 (2,4,5,2',4',5'-hexaCB)	35065-27-1	0,0008	-(0,3)[3]	0,002
PCB 180 (2,3,4,5,2',4',5'-heptaCB)	35065-29-3	0,0008	-(0,3)[3]	0,002
Sum of polychlorobiphenyle		0,004	1	
PCB 118 (2,4,5,3',4'-pentaCB)	31508-00-6	0,0008	-(0,3)[3]	0,002
Hexachlorobenzene (HCB)[4]	188-74-1			
alfa-Hexachlorocyclohexane (α-HCH)	319-84-6	0,0005	-(0,3)[3]	0,001
beta-Hexachlorocyclohexane (β-HCH)	319-85-7	0,0002	-(0,3)[3]	0,001
gamma-Hexachlorocyclohexane (γ-HCH)	58-89-9	0,00001	-(0,3)[3]	0,001
Sum of HCHs			2	
Aldrin	390-00-2	0,0005	-(0.3)[3]	0,001
Dieldrin	60-57-1	0,0001	-(0.3)[3]	0,001
Endrin	72-20-8	0,0002	-(0.3)[3]	0,001
Sum of drins			4	
p,p'-DDE	72-54-9	-	-(0,3)[3]	0,001
o,p'-DDD	53-19-0	-	-(0,3)[3]	0,001
o,p'-DDT	784-02-6	-	-(0,3)[3]	0,001
p,p'-DDD	72-54-8	-	-(0,3)[3]	0,001
o,p'-DDE	3424-82-6	-	-(0,3)[3]	0,001
p,p'-DDT	50-29-3	-	-(0,3)[3]	0,003
Sum of DDT,DDE,DDD		0,0005	4	
Heptachloride	76-44-8	0,0005	-(0,3)[3]	0,001
α-Endosulfan	959-98-7	0,0005	-(0,3)[3]	0,001
cis-Heptachloro-epoxide	280044-83-9	-	-(0,3)[3]	
trans-Heptachloro-repoxide	1024-5703	-	-(0,3)[3]	0,001
Sum of heptachloro-epoxide		0,0005		
Chlorodane (cis & trans)	87-66-3	0,0005	-	0,001

[1] The target value is based on soil containing 2% organic matter and 0% silt.

[2] The intervention value is based on soil containing 10% organic matter and 25% silt.

[3] In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

[4] See performance sheet SG-XVII.

Method and quality assurance

Soil

METHOD

Sampling AP04-M, applicable VKB-protocols
Storage conditions SIKB-protocol 3001
Storage period SIKB-protocol 3001

Continuation of performance sheet: polychlorobiphenyls (PCB) en organochloro pestides (OCB)

Sample pre-treatment AP04-V
Related task NEN 5747 (dry matter)
Sample size 250 ± 13 g
Storage conditions SIKB-protocol 3001
Storage period SIKB-protocol 3001
Task NEN-ISO 10382
Sample size > 20 g
In multiple N/A

FIRST-LINE CONTROL
Demonstrability limit $< AG_{eis}$ (see "Analytes to be determined" above)
Blank $< AG_{eis}$
Control sample
Analyte(s) HCB, PCB 52, β-HCH, α-HCH,
p.p-DDT en α-endosulfan
Recovery
HCB 60 - 110%
α-endosulfan 60 - 110%
others 75 - 110%
Repeatability variation coefficient
all < 20%
Intra-laboratory reproducibility variation coefficient
all < 25%
Additional quality assurance points
Internal standard Yes (5.1.5.1)
Confirmation Yes, 2 % of the samples if ECD-detection is applied (see 5.1.5.2). Confirmation by means of GCMS of column with other polarity (see NEN-ISO 10382).
Specific points of interest
<u>- Chromatografic analysis</u>
Selectivity: See NEN-ISO 10382, if GC-MS is applied, only for analytes with the same m/z.
Comparison investigation
<u>samples containing analytes</u> yes
for PCB 138, PCB 153, hexachloorbenzene, γ-HCH and p,p'-DDT.
<u>deviating soil characteristics</u> N/A
<u>additional comparison experiments</u> yes
other analytes by addition to soil samples or paragraphe 4.2 (see 4.1.4.2)

SECOND-LINE CONTROL
Soil
Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes (at least) quarterly: , α-HCH, β-HCH, PCB 52, α-endosulfan en p.p'-DDT
Investigate biannually on : all

THIRD-LINE CONTROL
Ring tests
Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.
Reference materials
Concentration range of the samples : within the measurement range of the task
Matrix : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards(NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards & Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XVII Determination of the content of chlorobenzenes in soil

Principle

Mono-and dichlorobenzenes

See performance sheet SG-VIII. The volatile chlorobenzenes are determined according to NEN-ISO 15009. The chlorobenzenes are not explicitly stated in the application range of this standard. The standard is applicable nevertheless.

Tri-, tetra-, penta- and hexachlorobenzene

See performance sheet SG-XVI. The moderately-volatile chlorobenzenes are determined according to NEN-ISO 10382. The chlorobenzenes are not explicitly stated in the application range of this standard. The standard is applicable nevertheless.

Conformity with the standard

The analytes have not been listed in the scope of the standards as stated under´Method´. Therefore no conformity with the relevant standard may be claimed. N.B. Should a Dutch preface be published for the standards referred to, in which the standard is declared applicable for the determination of the analytes referred to, then conformity with the relevant standard may be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (µg/kg.ds) target value-[1]	intervention-value-[2]	AG eis
Monochlorobenzene	108-90-7	(d)	-(6000)[3]	500
1,2-Dichlorobenzene	95-50-1	-	-(6000)[3]	300
1,3-Dichlorobenzene	541-73-1	-	-(6000)[3]	300
1,4-Dichlorobenzene	106-46-7	-	-(6000)[3]	300
Sum of dichlorobenzenes		2		
1,2,3-Trichlorobenzene	87-61-6	-	-(600)[3]	3
1,2,4-Trichlorobenzene	120-82-1	-	-(600)[3]	3
1,3,5-Trichlorobenzene	108-70-3	-	-(600)[3]	3
Sum of trichlorobenzenes		2		
1,2,3,4-Tetrachlorobenzene	634-66-2	-	-(600)[3]	1
1,2,3,5-Tetrachlorobenzene	634-90-2	-	-(600)[3]	1
1,2,4,5-Tetrachlorobenzene	95-94-3	-	-(600)[3]	1
Sum of tetrachlorobenzenes		2		
Pentachlorobenzene	608-93-5	0,5	-(600)[3]	0,4
Hexachlorobenzene	188-74-1	0,5	-(600)[3]	1
Sum of Chlorobenzenes			30000	

[1] The target value is based on soil containing 2% organic matter and 0% silt.
[2] The intervention value is based on soil containing 10% organic matter and 25% silt.
[3] In the Building materials decree no intervention value is given. The value given is applied in validation and quality assurance.

Method and Quality Assurance

METHOD

Sampling	AP04-M, applicable VKB-protocols	
Storage conditions	SIKB-protocol 3001	
Storage period	SIKB-protocol 3001.	
	Mono-, dichlorobenz.	**Tri-, tetra-, penta-, hexachlorobenz.**
Sample pre-treatment	see perf.sheet SG-VIII	see performance sheet SG-XVI
Task	see perf.sheet SG-VIII	see performance sheet SG-XVI

FIRST-LINE CONTROL

Demonstrability limit	< AG eis (see "Anal.to be determ.")	< AG eis (see "Anal. to be determ.")
Blank	< AG eis	< AG eis

Control sample

Analytes	see perf.sheet SG-VIII	see perf.sheet SG-XVI en 1,2,3,4-Tetrachlorobenzene

Continuation of performance sheet: Chlorobenzenes

Recovery

Mono-, dichlorobenzene	70 - 110%
Trichlorobenzenes	60 - 110%
Tetrachlorobenzene	55 - 110%
Pentachlorobenzene	65 - 110%
Hexachlorobenzene	see performance sheet SG-XVI

Repeatability variation coefficient

All	< 20%

Intra-laboratory reproducibility variation coefficient

All	< 25%

Additional quality assurance points

Internal standard	see performance sheets SG-VIII en SG-XVI
Confirmation	see performance sheets SG-VIII en SG-XVI

Specific points of interest

N/A

SECOND-LINE CONTROL

Soil

Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes quarterly on : All

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration of the samples : within the measurement range of the task
Matrix soil : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XVIII Determination of organonitrogen-pesticides in soil

Principle

The analysis sample is extracted with aceton and dichloromethane. The extract is shaken with water, dried and condensed. If interfering compounds are present, a clean-up over a silicagel column is performed. The content of organonitrogen pesticides is measured by means of a gaschromatograph with NPD-detection or a mass selective detector.

Conformity met de standard

The procedure based on this performance sheet differs from the standard as stated here under´Method´ in respect of sample extraction. As the standard has a different application range (water), no conformity with the standard can be claimed.

Analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds) target value-[1]	intervention value-[2]	AG $_{eis}$
Atrazine	1912-24-9	0,00001	6	0,002
Propazine	139-40-2			
Simazine	122-34-9			
Terbutryn	886-50-0			

[1] The target value is based on soil containing 2% organic matter and 0% silt.
[2] The intervention value is based on soil containing 10% organic matter and 25% silt.

Method and quality assurance

	Soil
METHOD	
Sampling	AP04-M, applicable VKB-protocols
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Sample pre-treatment	AP04-V
Related task	NEN 5747 (dry matter)
Sample size	250 ± 13 g
Storage conditions	SIKB-protocol 3001
Storage period	SIKB-protocol 3001
Task	VPR C85-17, of NEN-EN 10695 (measurement)
Sample size	> 20 g
In multiple	N/A

FIRST-LINE CONTROL	
Demonstrability limit	< AG $_{eis}$ (see "Analytes to be determined" above)
Blank	< AG $_{eis}$
Control sample	
Analyte(s)	Atrazine
Recovery	
All	70 - 110 %
Repeatability variation coefficient	
All	< 15 %
Intra-laboratory reproducibility variation coefficient	
All	< 20 %

Additional quality assurance points	
Internal standard	N/A
Confirmation	Yes, 2 % of the samples if NPD-detection is applied (see 5.1.5.2). Confirmation by means of GCMS.

Specific points of interest

- Gaschromatographic analysis

Selectivity: if GC-MS is applied, only for analytes with the same m/z
Trennzahl (simazine and atrazine) > 1,5
Trennzahl (propazine and atrazine) > 1,5

Continuation of performance sheet: Organonitrogen pesticides

Comparison investigation

samples containing analytes N/A
deviating soil characteristics N/A
additional comparison experiments no
soil characteristics of paragraph 4.2 with addition of all components (see 4.1.4.2)

SECOND-LINE CONTROL

Soil
Concentration range of analytes in the sample : within the measurement range of the task
Investigate analytes (at least) quarterly on : Atrazine
Investigate biannually on : all

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test : AG_{eis}
Concentration range of the samples : within the measurement range of the task
Soil : institution accredited for ring tests.

Reference materials

Concentration range of the samples : within the measurement range of the task
Matrix : sand, clay, peat or a mixture of these types of soil
Soil samples of : Bureau Communautaire de Reference (BCR),
National Bureau of Standards (NBS),
Nederlands Bureau voor Referentiematerialen(NMI-NBR),
National Institute of Standards and Technology (NIST),
Samples with a conventional true value
Certified materials that have been analysed according to the task described in the performance sheet.

Performance sheet SG.XIX Investigation protocol for other parameters

The investigation protocol is valid for all parameters not mentioned in the Accreditation programme as well as for those parameters for which no task has been defined in the Accreditation programme. Validation and quality assurance of a task must take place in accordance with Accreditation programme.
For the pre-treatment of samples document AP04-V must be followed.

1 Choosing a task

A task which is executed under the Investigation protocol, should possess the performance characteristics listed in the table below.

Tabel: Quality requirements for a quality task

	Soil: Organic components	**Soil: Inorganic components**
Demonstrability limit	< 0,33 times the target value[1]	< 0,33 times the target value[1]
Measurement range		
all	up to intervention value	up to intervention value
Recovery		
all	50 - 110 %	70 - 110 %
Repeatability variation coefficient		
all	< 15 %	< 15 %
Intra-laboratory reproducibility variation coefficient		
all	< 20 %	< 20 %

[1] starting from 2 % organic matter and 0 % silt.

A task is chosen according to the following preferential order:

- In accordance with Regulations for implementing the Building materials decree Appendix G (organic parameters).
- In accordance with a NEN-standard (compound determination); the parameter has been determined in accordance with this standard, if the standard meets the desired performance characteristics.
- In accordance with a CEN- or ISO-standard; if for the relevant parameter there is a NEN-standard, the parameter must be determined in accordance with it.
- Base don a NEN-standard for another matrix than soil (watersoil or building material; compound determination); the parameter has been determined in accordance with this standard, if the standard meets the desired performance characteristics.

Organic parameters:

- Can the parameter be determined in accordance with the methods mentioned in Appendix G or the Regulations for implementing the Building materials decree?
- Does a validated RIZA-, IVM-, RIVM- or RIKILT-measurement method exist which meets the desired performance characteristics?
- In accordance with ASTM- or EPA-standard; which meets the desired performance characteristics.
- An "in-house" measurement method (literature research) which can meet the desired performance characteristics.

Inorganic parameters:

- Does a validated RIZA-, IVM-, RIVM- or RIKILT-measurement method exist: if it does, does this procedure meet the desired performance characteristics?

- In accordance with the EPA-standard; if it possesses the desired performance characteristics.
- An "in-house" measurement method (literature research) which can meet the desired performance characteristics.

If no procedure is able to meet the desired performance characteristics, a procedure may be applied with less performance characteristics in respect of repeatability (at most 25%). If the repeatability is >15%, the procedure must be performed in multiple.

2 Validation of a task not laid down

The task is validated in accordance with the test procedures given in Chapter 4. The measurement range, the demonstrability limit, the recovery and the repeatability must be determined during the validation investigation

3 Quality assurance of a task not laid down

The quality assurance of the task is performed in accordance with Chapter 5.

SG7 Literature

NEN 3114	1990	Nauwkeurigheid van metingen - Termen en definities.
ISO 3534-1	1993	Statistics - Vocabulary and symbols - Part 1: Probability and general statistical terms.
Ontw. NVN 5710	2003	Bodem, bouwmaterialen en waterbodem – Bepaling van de gehalten aan tien (zestien) polycyclische aromatische koolwaterstoffen (PAK) in grond met on-line zuivering en hogedruk-vloeistofchromatografie (HPLC).
NVN 5731	1998	Bodem - Bepaling van de gehalten aan tien polycyclische aromatische koolwaterstoffen met behulp van hogedruk-vloeistofchromatografie.
NEN 5733	1997	Bodem - Bepaling van het gehalte aan minerale olie in grond en water-bodem met gaschromatografie.
NEN 5735	1999	Bodem - Bepaling van het halogeengehalte afkomstig van niet-vluchtige, met aceton en petroleumether extraheerbare organohalogeenverbindingen (EOX).
NEN 5739	1996	Bodem - Bepaling van het gehalte aan vrij ijzer met atomaire-absorptiespectrometrie.
NEN 5747	1990	Bodem - Bepaling van het vochtgehalte en het gehalte aan droge stof van veldvochtige grond.
Ontw. NEN 5748	2004	Bodem - Bepaling van het vochtgehalte en het gehalte aan droge stof van luchtdroge grond en waterbodem.
Ontw. NEN 5750	2004	Bodem - Bepaling van de pH in grond-, sediment-, slib- en bouwstofmonsters.
Ontw. NEN 5753	2005	Bodem - Bepaling van lutumgehalte en korrelgrootte van grondmonsters met behulp van zeef en pipet.
Ontw. NEN 5754	2004	Bodem - Bepaling van het gehalte aan organische stof in grond en waterbodem volgens de gloeiverliesmethode.
NEN 5758	1990	Bodem - Bepaling van het gehalte aan koper in grond met behulp van atomaire-absorptiespectrometrie (vlamtechniek) na ontsluiting met salpeterzuur en zoutzuur.
NEN 5759	1990	Bodem - Bepaling van het gehalte aan zink in grond met behulp van atomaire-absorptiespectrometrie (vlamtechniek) na ontsluiting met salpeterzuur en zoutzuur.
NEN 5760	1991	Bodem - Bepaling van het gehalte aan arseen in grond met behulp van atomaire-absorptiespectrometrie (hydridegeneratietechniek) na ontsluiting met salpeterzuur en zoutzuur.
NEN 5761	1990	Bodem - Bepaling van het gehalte aan lood in grond met behulp van atomaire-absorptiespectrometrie (vlamtechniek) na ontsluiting met salpeterzuur en zoutzuur.

NEN 5762	1990	Bodem - Bepaling van het gehalte aan cadmium in grond met behulp van atomaire-absorptiespectrometrie (vlamtechniek) na ontsluiting met salpeterzuur en zoutzuur.
NEN 5765	1991	Bodem - Bepaling van het gehalte aan nikkel in grond met behulp van atomaire-absorptiespectrometrie (vlamtechniek) na ontsluiting met salpeterzuur en zoutzuur.
NEN 5767	1991	Bodem - Bepaling van het gehalte aan chroom in grond met behulp van atomaire -absorptiespectrometrie (vlamtechniek) na ontsluiting met salpeterzuur en zoutzuur.
NVN 5770	1993	Bodem en slib - Monstervoorbehandeling van grond en slib voor bepaling van elementen met atomaire-spectrometrie. Ontsluiting met salpeterzuur en zoutzuur in een microgolfoven.
Ontw. NEN 6427	1999	Water – Bepaling van 66 elementen met inductief gekoppeld plasma massaspectrometrie.
NEN 6465	1992	Water, lucht en bodem - Monstervoorbehandeling van slib, slibhoudend water, luchtstof en grond voor de bepaling van elementen met atomaire-absorptiespectrometrie - Ontsluiting met salpeterzuur en zoutzuur.
NEN 6476	1981	Water - Bepaling van het gehalte aan chloride door potentiometrische titratie.
NEN 6483	1982	Water - Potentiometrische bepaling van het totale gehalte aan fluoride.
NEN 6589	1990	Regenwater - Potentiometrische bepaling van het gehalte aan totaal anorganisch fluoride met behulp van een doorstroominjectiesysteem.
NPR 6603	1988	Water en slib - Richtlijnen voor interne kwaliteitsbeheersing met controlekaarten bij chemische analyses.
NEN 6611	1997	Water en slibhoudend water. Bepaling van het antimoongehalte met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6655	1997	Water en bodem - Fotometrische bepaling van het totale gehalte aan cyanide en het gehalte aan vrij cyanide met behulp van een doorstroomanalysesysteem.
ISO 6879	1995	Air quality - Performance characteristics and related concepts for air quality measuring methods.
Ontw. NEN 6961	2001	Ontsluiting voor de bepaling van 28 geselecteerde elementen met koningswater. Betreft de elementen Ag, Al, As, B, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Hg, Mg, Mn, Mo, Na, Ni, K, P, Pb, Sb, Se, Sn, Sr, Tl, V, Zn.
NVN 7321	1997	Uitloogkarakteristieken van vaste grond- en steenachtige bouwmaterialen en afvalstoffen - Bepaling van het gehalte van anorganische componenten - Bepaling van het gehalte van 11 elementen met atomaire-absorptiespectrometrie (grafietoventechniek).

NVN 7322	1997	Uitloogkarakteristieken van vaste grond- en steenachtige bouwmaterialen en afvalstoffen - Bepaling van het gehalte van anorganische componenten - Bepaling van het gehalte van 14 elementen met atomaire-emissiespectrometrie (inductief gekoppeld plasma).
NEN 7777	2003	Milieu – Prestatiekenmerken van meetmethoden.
NEN 7778	2003	Milieu – Gelijkwaardigheid van meetmethoden.
ISO 8466-1	1993	Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics - Part 1: Statistical evaluation of the linear calibration function.
Ontw. NEN-EN-ISO 9169	2004	Luchtkwaliteit - Toepassing en bepaling van de prestatiekenmerken van een automatische meetsysteem.
NEN-EN-ISO 10304-2	1996	Bepaling van opgeloste anionen met vloeistofchromatografie. Deel 2: Bepaling van bromide, chloride, nitraat, nitriet, orthofosfaat, en sulfaat in afvalwater.
NEN-ISO 10382	2003	Bodem - Bepaling van organochloorbestrijdingsmiddelen en polychloorbifenylen - Gaschromatografische bepaling met elektronen-invangdetectie.
NEN-ISO 10693	2004	Bodem - Bepaling van het gehalte aan carbonaten - Volumetrische methode.
NEN-EN-ISO 10695	2000	Water – Bepaling van het gehalte aan geselecteerde organostikstof- en organofosforverbindingen – Gaschromatografische methoden.
NEN-ISO 15009	2002	Bodem - Gaschromatografische bepaling van het gehalte aan vluchtige aromatische koolwaterstoffen, naftaleen en vluchtige gehalogeneerde koolwaterstoffen - "Purge-and-trap"-methode met thermische desorptie.
NEN-EN-ISO 15682	2001	Water – Bepaling van het gehalte aan chloride met doorstroomanalyse (CFA en FIA) en fotometrische of potentiometrische detectie.
NEN-ISO 16772	2004	Bodem - Bepaling van het gehalte aan kwik in koningswater bodemextracten met behulp van atomaire-absorptiespectrometrie met koude damp of atomaire fluorescentiespectrometrie met koude damp.
VPR C85-06	1985	Voorlopige praktijkrichtlijn bodem - grondwater en grond, opwerking en analyse - Bromide (opgelost c.q. oplosbaar).
VPR C85-14	1985	Voorlopige praktijkrichtlijn bodem - grondwater en grond, opwerking en analyse – Chloorfenolen.
VPR C85-17	1985	Voorlopige praktijkrichtlijn bodem – grondwater en grond, opwerking en analyse – Organo-stikstofbestrijdingsmiddelen (triazines).