

**ACCREDITATION PROGRAMME
BUILDING MATERIALS DECREE**

Section: Analysis of eluates

AP04 - E



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E 1 Introduction

The Accreditation Programme "Building Materials Decree, section: analysis of eluates" (hereafter called AP04-E), describes the tasks and the performance characteristics of the tasks that must be applied within the scope of the Building Materials Decree. Furthermore, the testing procedures for the determination of the performance characteristics and the corresponding criteria have been defined, to which a task must comply.

The (inter)nationally standardised tasks that are applied in investigations within the scope of the Building Materials Decree are reference points for the Accreditation Programme AP04-E. For the prescribed regulations the reference point of (inter)nationally standardised regulations is defined. If a task is not executed in conformance with defined (inter)national regulations, the equivalence of the measuring method has to be proven.

In this document, the tasks relating to the analysis of eluates that fall under the Accreditation Programme are stated in Chapter E2. In Chapter E3, the concepts and parameters being used are defined and in Chapter E4 the validation of a task is rendered by generally acknowledged procedures. In Chapter E5, the first-, second- and third-line controls are described that must be applied at the very least during the execution of tasks that fall under the Accreditation Programme Building Materials Decree. The technical data of the tasks and the corresponding performance characteristics are given in Chapter E6.



E 2 Overview of tasks

This section of the Accreditation Programme includes all tasks required for the analysis of eluates from the leaching investigation within the scope of the "Building Materials Decree". All these tasks have been included in the classification of packages for the specification Leaching investigation (see APO-A).

In APO4-E the following tasks are described:

- Determination of lead in eluates;
- Determination of cadmium in eluates;
- Determination of zinc in eluates;
- Determination of nickel in eluates;
- Determination of arsenic in eluates;
- Determination of chromium in eluates;
- Determination of copper in eluates;
- Determination of mercury in eluates;
- Determination of molybdenum in eluates;
- Determination of barium in eluates;
- Determination of tin in eluates;
- Determination of cobalt in eluates;
- Determination of antimony in eluates;
- Determination of selenium in eluates;
- Determination of vanadium in eluates;
- Determination of cyanides (free and complex) in eluates;
- Determination of bromide in eluates;
- Determination of chloride in eluates;
- Determination of sulphate in eluates;
- Determination of fluoride in eluates;
- Determination of sodium in eluates;
- Determination of potassium in eluates.



For the leaching investigation, please refer to the relevant section in the Accreditation Programme AP04-U.

For all leaching tests, the requirements for the eluate analyses are the same, with the exception of the required detection limits (AG_{eis}). The AG_{eis} in non-moulded materials stated in the performance sheet applies to the column test; the AG_{eis} for moulded materials applies to the diffusion- and availability tests.

E 3 Concepts/parameters

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

E 3.1 Definitions of terms

In this paragraph, the terms used in the definitions and test procedures of performance characteristics in paragraph E3.2, Chapter E4 and 5 are defined.

True value (NEN 3114, NEN 7777)

Value of a precisely defined quantity.

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

(Measurement) variable (NEN 7777)

Characteristic of a phenomenon or a body that can be distinguished qualitatively and determined quantitatively.

Example: The (measurement) variable "mass concentration of nitrate"; "nitrate" is not in itself a (measurement) variable.

Measurement value, measurement result (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 a measurement value approaches in an increasing number of measurement values.

Measuring method (NEN 3114, NEN 7777)

Manner in which measurements are conducted 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, as regards composition and form, is accepted as being representative for samples and which is intended for 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 (measurement) quantity.

Measurement signal (NEN 7777)

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

Explanation 1: *The "electrical charge" on the output of a 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 to such a small degree, that the difference between both values is negligible.

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

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 analyses.

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 an 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, in which variance is: sum of the squared measured accidental deviations divided by the number of measurement 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 this concentration can be taken as constant, it is defined as follows, where "percent" is the unit:

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

In the event of duplicate determinations, in which the concentration is not constant, but in which vc is taken 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, the execution of which does not deviated from the prescribed task (standard), and in which any deviations in non-crucial parts do not have a demonstrable effect on the result.

Equal (measurement) method (NEN 7778)

Measurement method that meets the requirements that have been set for the intended use of the reference method from the perspective of effectiveness ('fitness for purpose').

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 different types of matrices (e.g. eluates of the column test and diffusion test) and known interferences.

Maximally Allowed Leaching Concentrations (MAU)

This refers to that concentration in an eluate, from which the maximally allowed emission can be calculated.

Remark: The required detection limit (AG_{eis}) is determined at 30% of the MAU. For calculating the MAU, please refer to Attachment E1.

z-score

Deviation of the measurement value from the testing value, in relation to the standard

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

deviation.

In which: x_i represents: measurement value;
 \bar{x} represents: testing value such as:
- "assigned value" in an inter-laboratory investigation;
- assigned value for a reference material;
- measurement value of 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.

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 average z^2 is calculated:

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

The limiting values of both z and the average z^2 are determined by taking into account the risks of accidental exceedings and their relevance with respect to the total measurement error.

E3.2 Definitions of performance characteristics

Detection limit (NEN 7777)

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

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

Explanation 2: The legally required detection limit, AG_{eis} , is the detection limit that must be achieved at the very least. This is laid down in the Performance sheets per parameter.

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

Quantification limit (NEN 7777, Appendix C)

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

Explanation 1: Quantification limit is related to the quality criterion "measurement value".

Explanation 2: The term quantification limit 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, for the determination of the detection limit reference is made to NEN 7777. In this standard, the operational definition is used for the quantification limit, which equates the quantification limit to ten times the standard deviation at the relevant level. Therefore, the quantification limit is the value of the measurement quantity, in which the variation coefficient conventionally amounts to 10%.

Repeatability (NEN 3114, NEN 7777)

Standard for the spread in 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 determined 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 next page 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 ($v_{CW, eis}$) is determined for each parameter in the Performance sheets.

Linearity

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

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

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

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

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 circumstances, or quotient of the measurement value for a sample and the conventional true value.

Explanation 1: Recovery determined from the addition of the measurement component often generates (too) optimistic results, as it is difficult or impossible to include the added part of the measurement component in the sample in the same way as the original part.

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" has actually been defined and is 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 "Nauwkeurigheid" 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 another variable 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 more 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 detection limits stated in the Performance sheets and the highest value of the measurement quantity that can be measured according to the regulations, and of which the performance characteristics meet the defined requirements.

Trennzahl or separation number (NPR 6405)

As a measure for the separating capacity of the chromatographic system, the Trennzahl or the separation number T_z can be used. The Trennzahl is defined as follows:

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

In which:

$t_{R,i}$ and $t_{R,j}$: the retention times of compounds i and j respectively;
 $w_{1/2,i}$ and $w_{1/2,j}$: the width halfway up for compounds i and j respectively.

Explanation 1: *The Trennzahl is a measure for the efficiency and selectivity of a chromatographic system.*

Explanation 2: *By and large it can be stated that Trennzahl corresponds to the number of analytes (peaks) that still can be separated between the analytes i and j.*

Drift (ISO 6879)

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

Explanation: *If not specified further, "Drift" only refers to the change of the calibration function.*



E 4 Validation of a task

When a task is introduced or altered, an intra-laboratory validation investigation should be performed.

During a validation investigation the following performance characteristics are tested:

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

The investigation should prove 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 occurs according to the procedure stated 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 must 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 instruction, which includes sample pre-treatment. For the eluate analysis, this does **not** include the leaching test.

If the standard is deviated from, it must be determined by means of practical samples if the selectivity of the operation is in conformance with the standardised task.

The validation must be repeated if there is a change in either the equal task or the standard. This must be performed in conformance with 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 according to the procedure stated from this version (version 3) onward, is not necessary.

If not stated otherwise, the performance characteristics are determined on synthetic eluates (the validation matrix) of 1000 µS/cm (matrix I) and 20000 µS/cm (matrix II) after addition of the analytes to be investigated on the right concentration level.

Table 1 Composition of synthetic eluates

Matrix chemicals (free of watervrij) in mg/l	Matrix I all analytes *	matrix I sodium, potassium	matrix II all analytes *	matrix II sodium, potassium	matrix II cyanide	matrix II chloride, fluoride, sulfate, bromide
calcium chloride	117	117	2230	2230	2230	
sodium chloride	382		4400		4400	
potassium chloride			5600		5600	
sodium nitrate			136			136
sodium acetate			664		664	664

* with the exception of the analytes mentioned in the table.

Concentration levels for validation are chosen in accordance with NEN 7777 in the lowest decade of the measurement range (see definition measurement range E3) and in the highest decade of the measurement range.



E 4.1 Test procedure and quantification of performance characteristics

E 4.1.1 Detection limit (AG)

The determination of the demonstrability limit is stated in NEN 7777. The detection limit must be determined on the basis of repeatability conditions.

Criterion:

- The detection limit must meet the requirement stated in the performance sheet.

E 4.1.2 Determination of the measurement range of the task

Give as measurement range the domain between detection limit and the highest value (minimal intervention value) of the measurement variable that according to the instruction can still be measured, while the performance characteristics meet 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: "Detection limit" must be replaced by "lowest value" if on account of other limitations (e.g. a-linearity) a performance characteristic (e.g. linearity) not yet meets the defined requirement for values above the detection limit.

E 4.1.3 Testing of the linearity / "lack of fit" of the calibration function of the analysis-instrument

The testing of the practicability of the chosen calibration function for the entire measurement range is stated in NEN 7777. The testing must be performed on the bases of absolute limiting values.

Determine for c the requirement in model deviation ($\delta_{c,model,abslim}$) by means of the following formula:

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

Calculate the deviation of the theoretical concentration:

$$\delta_{c,model} = \bar{x} - theoretical\ concentration$$

in which: \bar{x} = average measurement result in mg/l

The result is satisfactory, if:

$$|\delta_{c,model}| \leq \delta_{c,model,abslim} - \frac{t_{0,975} * s_w}{\sqrt{n}}$$

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

n = number of measurement results

E 4.1.4. Determination of the intra-laboratory trueness / recovery (δ_c , Tv)

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

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



E 4.1.4.1 Intra-laboratory trueness by means of certified reference material

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

Criterion:

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

E 4.1.4.2 Recovery from addition

Recovery investigations are performed on "spiked samples", if a certified reference sample is not available for the task and for a specific matrix.

The determination of recovery from addition in accordance with this method is stated in NEN 7777.

Explanation 1: *If possible, the samples stored for 48 hours under the storing conditions given in the performance sheet. After 48 hours the samples are analysed according to the task.*

Criterion:

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

E 4.1.5 Determination of repeatability standard deviation (s_r , v_{cr})

The determination of the repeatability standard deviation is stated 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 allowed.

For the analysis, the whole measurement method must be completed, inclusive of the leaching test.

Unless stated otherwise, in the performance sheet the repeatability is expressed as variation coefficient (v_{cr}).

Criterion:

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

E 4.1.6 Determination of intra-laboratory reproducibility standard deviation (s_W , v_{cW})

The determination of the intra-laboratory reproducibility standard deviation is stated in NEN 7777. This can be executed in two ways: duplicate analysis of different samples or repeated analysis of the same laboratory sample. Both methods are allowed.

For the analysis, the whole measurement method must be completed, inclusive of the leaching test.

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

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

Criterion:

- The intra-laboratory reproducibility variation coefficient must fulfil the requirement stated in the performance sheet.

E 4.2 Equality investigation of a non-standardised task

The equality investigation must be executed, if a task is not performed in conformity with the reference method. The equality investigation must be executed in conformance with NEN 7778.

E 4.3 Reporting of activities

The validation investigation is rounded off by a validation report. The conclusion of the investigation must be relevant in relation to APO4 and is to be represented in a performance sheet in which the validation result and the APO4-requirements have also been included.

A complete documentation of each performed validation investigation must be available during the accreditation investigation.



E 5 Quality assurance of a task

The quality assurance of a task is subdivided as follows:

- First-line control - Performance checking of the executors themselves.
- Second-line control - Check within the organisation of the institution, independently of the executors.
- Third-line control - Independent external check, ring tests.

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

- statistical supervision over the quality assurance;
- settlement of out-of-control situations;
- use and/or preparation of samples;
- responsibilities of officials.

The defined first-, second- and third-line controls do not present an additional quality assurance, but the minimal quality assurance to be applied.

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

- it turns out in practice that over longer periods of time no samples are being presented for a specific task;
- work is performed under a cluster arrangement, whereby according to schedule over a longer period of time a task is not performed by a certain laboratory, followed by a period in which it does perform this task regularly.

There is no precise dividing line between a task which is and which is not performed routinely. It may also occur, that a task can be considered non-routine over longer periods of time, but that it must be considered routine during periods, in which there is a regular supply (as in the example of the cluster arrangement).

Overall guiding lines are:

- for the first-line control (E5.1), pursue the procedure for a non-routine task, if over a period of four consecutive quarters no full control chart can be obtained.
- for the second-line control (E5.2) and the third-line control (E5.3), pursue the procedure for a non-routine task, if over a period of four consecutive quarters during three of the four quarters no samples are being presented.

The first-, second- and third-line controls are different when it comes to routine and non-routine tasks. Below, it is indicated for each of them, which quality assurance must be executed for a routine and which for a non-routine task.

The quality assurance can only be executed based on a non-routine task, after the accreditation and definitive appointment have been obtained on the basis of validation investigation and quality assurance of a routine task.

E 5.1 First-line control

The first-line control must be composed of a periodical performance investigation and the quality check during the execution of a task.

During the periodical performance investigation, the compliance of the task with the data stated in the performance sheet must be checked (Chapter E6). The quality assurance during the performance of a task is performed by means of control samples.

A different schedule applies for non-routine tasks.



E 5.1.1 Periodical performance investigation

Unless stated otherwise, the basic materials upon which the investigation is executed are: synthetic eluates of the composition given below (the validation matrix) of 1000 $\mu\text{S}/\text{cm}$ (matrix I) and 20000 $\mu\text{S}/\text{cm}$ (matrix II) after addition of the substances to be investigated in the right concentration level.

Tabel 2: Samenstelling synthetische eluaten

Matrix chemicals (free of water) in mg/l	Matrix I al analytes *	matrix I sodium, potassium	matrix II all analytes *	matrix II sodium, potassium	matrix II cyanide	matrix II chloride, fluoride, sulfate, bromide
calcium chloride	117	117	2230	2230	2230	
sodium chloride	382		4400		4400	
potassium chloride			5600		5600	
sodiumnitrate			136			136
sodiumacetate			664		664	664

* with the exception of the analytes given in the table.

E 5.1.1.1 Recovery

Analytes : all parameters.
 Concentration range : "spiked samples" with concentrations within the measurement range of the task
 Method : see E4.1.4.
 Frequency : 1 in every 100 samples, maximally once a week.
 Number : singular.
 Criterion : See the criterion stated in the performance sheets.

Periodical performance investigation on recovery is not applicable for a non-routine task (see E5.1.3).

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

E 5.1.1.2 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 is analysed in duplicate, with a minimum of 1 practical sample a month and a maximum of 1 practical sample a week; for determinations that are as a rule being performed multiple, a daily test in accordance with E5.1.4.1. suffices.
 : non-routine investigation:
 see E4.1.5.
 Frequency : routine investigation:
 quarterly evaluation.
 non-routine investigation:
 once a year, if the task is performed.
 Criteria:
 routine investigation : the repeatability variation coefficient vc_r is determined in accordance with E4.1.5.1.
 The repeatability variation coefficient must fulfil the requirement stated in the performance sheet.

non-routine investigation : requirement stated in the performance sheet.



If desirable for practical reasons, it is permitted not to perform the duplicate analysis in one day, but spread over two days. In this case, the requirement for the intra-laboratory reproducibility variation coefficient vc_w , stated in the performance sheet, must be met.

E5.1.1.3 Intra-laboratory reproducibility variation coefficient (vcW)

The intra-laboratory reproducibility standard deviation must be determined, if:

- no control chart is used,
- a control chart is used, from which the intra-laboratory reproducibility cannot be determined for all parameters in the desired concentration range.

Analytes	:	parameters.
Concentration range	:	within the measurement range of the task
Method	:	see E4.1.6.
Frequency	:	routine investigation: once a year. non-routine investigation: once a year, if the task is performed.
Number	:	at least ten.
Criterion	:	requirement stated in the performance sheet.

E5.1.1.4 "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 overdimensioned¹ and
- the sensitivity (=inclination of the calibration function) has changed more than 20% or the instrument is re-introduced after a major intervention or after a longer period of not having been used.

In a multi-component analysis, those analytes that are decisive for the measurement range are tested. As a rule, these are the insensitive and sensitive analytes.

Analytes	:	all parameters;
Concentration range	:	the measurement range of the analysis instrument;
Method	:	see E4.1.3 Checking the "lack of fit" as part of the quality assurance is performed as an indicative testing of the model deviation. This may be executed under repeatability conditions. The testing may take place on the basis of the repeatability standard deviation of the measurement results in mg/kg.ds. It is recommended to take a multiple measurement of both calibration standards and the measurement standard(s).
Number	:	see E4.1.3
Criteria	:	see E4.1.3

Periodical performance investigation into "Lack of Fit" is not applicable for a non-routine task (see E5.1.3).

¹ Overdimensioned in a linear function means that calibration is performed on more than two levels, in a second-degree polynomial on more than three, etc.



E5.1.1.5 Detection limit (AG)

The detection limit of the task must be tested, if the sensitivity (measurement signal/quantity of the analyte) decreases to such a degree, that most likely the required detection limit will not be reached.

The criterion is, that the detection limit must be determined, if

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

In which: B_{gev} : the found sensitivity;
 AG_{eis} : the detection limit required in the performance sheet;
 AG_{vst} : the detection limit in the last determination investigation;
 B_{vst} : the sensitivity in the last determination investigation of the detection limit.

Explanation 1: During a multi-component analysis, those components are chosen, of which the detection limits found are closest to the requirement stated in the performance sheet.

Explanation 2: The detection limit must be tested, 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 E4.1.1.
Criterion : the detection limit must correspond to the requirement given in the performance sheet.

E 5.1.2 Quality assurance during the execution of a routine task

The institution 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 and the analysis itself). This quality assurance must be done on the synthetic leaching sample matrix I, whereby 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 stated otherwise in the performance sheet.

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

E 5.1.2.2 Producing a control sample

The synthetic leaching sample matrix I is taken as the control sample, against which the analytes are spiked. The sample must be homogenised in such a way, that the inhomogeneity of the sample has no significant influence on the intra-laboratory reproducibility. For practical reasons (limited storage life), the sample may be replaced by a recovery experiment, such as the determination of volatile components. Instead of the control sample a reference sample may also be used, if it fulfils the set criteria (see E4.1.4.1).

E 5.1.2.2 Starting up a control chart

A control chart is started up by performing the task at least 10 times under intra-laboratory reproducibility conditions.

An outlier-test is performed 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, the Grubbs-test is executed twice at the most. When the outliers have been removed, the average and the standard deviation are determined once again and the outlier-test is repeated. This is repeated until no outliers are detected anymore.

At least 8 observations must remain, in order that the remaining observations can be used to start a control chart. The average and the 2s- and 3s-limits are calculated on the basis of these experiments.



Grubbs-test:

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

if $G_p >$ critical value, then the value is an outlier.

For $n=10$ $G_p = 2,482$

$n=9$ $G_p = 2,387$

$n=8$ $G_p = 2,274$

E 5.1.2.3 Filling in control charts (NPR 6603)

When the task is carried out, each working day, one independent measurement value (no average value of a multiple measurement or determination) is recorded on the control chart. If several control samples are analysed per working day, the values are tested against the control chart data.

E 5.1.2.4 Testing with a control chart (Shewhart)

In the following cases, it concerns out-of-control quality:

- every exceeding of the 3s-limit;
- a surpassing of the 2s-limit on the same side of the average twice on end;
- the eleventh consecutive value on the same side of the average.

In the following cases, it concerns an out-of-control quality during additional testing of several control samples per working day:

- every exceeding of the 3s-limit of control chart;
- when the 2s-limit is exceeded twice on end on the same side of the average under the same measurement conditions (executor and/or equipment).

When it concerns out-of-control quality, the following measures must be taken:

- 1) An investigation into the cause is carried out.
- 2) The measurement results of the relevant series, day or period are blocked, until the cause of the fault has been established. Analysis results that are smaller than the reporting limit are an exception to this, when it concerns out-of-control quality due to an overestimation of the true value. These analysis results can be reported.
- 3) After the cause of the fault has been removed, usually the relevant series of samples is analysed again.
- 4) When an out-of-control quality due to 11 observations on one side of the average is established, the cause of the shift must be investigated immediately. The contents of the samples that are part of the eleventh observation can be reported in any case.

If the problem cannot be solved, (no cause is known or the cause cannot be removed), the recovery must be determined again and tested 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 made use of all the same.

During the determination of the relevant performance characteristics, samples may nevertheless be analysed and reported. To prevent a situation of out-of-control quality, the results of the first-line control samples must meet the characteristics of the last control chart.

E 5.1.2.5 Testing a control chart

When a control chart is full, it is tested in accordance with NPR 6603. Additionally, the following issues are important:

- When the average and the standard deviation of a full control chart are calculated, out-of-control results, caused by the exceeding of the 3s-limits, are not included.
- When a control chart is closed, the average and standard deviation are tested in respect of the historical data of previous charts and the requirements for the



trueness (recovery) and the intra-laboratory reproducibility laid down in the performance sheets.

- NPR 6603 describes how the test in respect of the historical data of previous charts should be executed for the standard deviation, but not for the average. The average must be tested with the t-test:

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

In which: X_1 and X_2 are the averages of the previous control chart(s) and the present control chart respectively

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

in which: s_1 and s_2 are the standard deviations of the previous control chart(s) and the present control chart respectively

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

Subsequently, the testing value of $t(0,95;v)$ can be determined.

- In the event that the average and/or the standard deviation may not be combined, the cause of this should be investigated immediately. In case 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 the standard deviation has increased or the recovery deteriorated, yet the requirements laid down in the performance sheet are still being met, the cause does not have to be investigated and the charts may be combined. In latter case, it may be considered to calculate a new average or standard deviation.

If the quality has not improved or if the recovery or standard deviation does not meet the requirements of the performance sheet, a further investigation must be performed. If this does not solve the problem (no cause known or the cause cannot be removed), the relevant performance characteristics must be determined again and tested again against the criteria laid down in the performance sheets. In case of a deviation of the average, the trueness/recovery must be determined again and in case of a deviation of the standard deviation, the intra-laboratory reproducibility standard deviation must be determined again. The control chart is closed and the charts are 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 allowed to analyse and report samples. In order to avoid a situation of out-of-control quality, the results of the first-line control samples must meet the characteristics of the last control chart.

After it has been established that no cause can be found, the performance characteristics of the new control chart must be determined within 10 measuring days. In addition to this, these 10 measuring days must fall within a period of 3 months.

- In order to make sure that during the combining of the control chart data the latest data have a sufficient influence on the values, during the combining, the previous four charts (approximately 120 observations) are fully made use of. When starting up the new chart, the average and the standard deviation of the previous five charts are used for filling in the average and the 1s, 2s and 3s limits.

E 5.1.3 Quality assurance during the execution of 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 tested at the very least:



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

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

- 1) one blank experiment,
- 2) two recovery experiments between 20 and $80 * C_{ag}$.
- 3) two recovery experiments with a concentration of at least 10 times higher.

E 5.1.3.1 Recovery

Analytes	:	all parameters;
Method/calculation	:	see E4.1.4. under repeatability conditions
Frequency	:	per investigation;
Criteria	:	the average recovery (experiments 2 and 3) must fulfil the requirement stated in the performance sheet.

E 5.1.3.2 Repeatability variation coefficient

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

E 5.1.3.3 "Lack of fit" / linearity of the calibration function

Analytes	:	all parameters;
Frequency	:	per investigation;
Calculation	:	see E4.1.3
Criteria	:	see E4.1.3

E 5.1.3.4 Detection limit

Analytes	:	all parameters;
Frequency	:	per investigation;
Criteria	:	see E5.1.1.5.

E 5.1.4 Additional daily quality assurance points

If additional quality assurance points are mentioned in the standardised regulation, these must be checked. Furthermore, in the performance sheet of the corresponding task, a number of additional quality assurance points are stated (specific points of interest). If a quality assurance point is stated in both, the strictest criterion applies.

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

- If no dilution is applied, a 1-point calibration solution must preferably be higher than 20 times the detection limit and lower than 0,6 times the linear measurement range of the measurement system or measurement method.
- If no dilution is applied, a calibration curve must be built up from a number of measurement points that are distributed proportionally across the entire measurement range (e.g. 3 measurement points per decade) of the measurement system of the measurement method.
- A procedure blank must fulfil the requirement stated in 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 increased blanks, a.o. with regard to:

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

E 5.1.4.1 Multiple determination (duplicate determination)

Analyses, for which the partial samples are taken before sample preparation has occurred, must be executed in duplicate. The duplicate determination is executed under repeatability conditions.

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

- The measurement results of the duplicate determinations may not differ from each other more than 4 times the requirement for the repeatability standard deviation. If this requirement is relative, the deviation between the duplicate results in terms of percentage is calculated starting from the average result.

The average value of the determination in duplicate is reported.

E 5.1.5 Additional periodical quality assurance points

In order to prevent gross mistakes, the institution must introduce an additional periodical quality assurance point for each task. For a number of tasks, this additional quality assurance point is prescribed in the performance sheet.

E 5.1.5.1 The application of (procedural) internal standards

If possible, the institution may apply internal standard(s) in order to detect gross mistakes.

Conditions for applying internal standard(s):

- The internal standard must be added (to the sample or extract of the sample) at an early stage, so that it will pass through the whole task.
- The internal standard must be representative for the analytes to be determined. These include labelled, deuterated compounds or a congeneric of the analytes to be determined.
- If the task permits this, an internal standard is applied.

A measurement value may only be corrected for an internal standard, if this is stated in the defined task. The institution must establish by means of a validation investigation, which internal standard the limiting value must meet and whether this is in a reasonable proportion to the data of the task concerned stated in the performance sheet.

E 5.1.5.2 Additional confirmation

If during a determination a non-specific detector is applied, if possible, additional confirmation must be carried out, for example by means of another detector or another set-up of the detector. As a rule, instructions in respect of this have been included in the standard stated under 'Method' in the performance sheet. Criteria regarding additional confirmation are stated in the performance sheets.

The purpose of additional confirmation is not to confirm results quantitatively. It rather concerns the confirmation, that the compounds found have been justifiably and correctly identified.

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

The additional confirmation may either be executed on the destructure/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.

E 5.1.5.3 Consistency analysis of calibration solutions

Monitoring the trueness of a calibration solution occurs during the production of new calibration standards: this applies for both stock solutions and calibration solutions. The trueness must be checked daily, if new calibration solutions are prepared for each measuring series. Usually, this is already being done with a control chart (control sample). But if a calibration solution is used for a longer period of time, a once-only check of the trueness of the calibration solution after the preparation will suffice. For 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 solution may not amount to more than 7,5%. In multicomponent- or multi-element calibration solutions, for maximally one fifth of the components or elements the relative difference may amount to 7,5%-12,5%.

E 5.2 Second-line control

The laboratory must regularly investigate the tasks with a second-line control-sample, of which the identity is as much as possible unknown. The frequency is such, that the sum of the second- and third-line checks is at least 4 a 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 must be laid down in a working guideline. Also, the procedural aspects must be recorded in a separate procedure.

For the second-line control, the following materials can be used:

- (internal) certified reference materials;
- spiked samples;
- blank material.

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

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

E 5.3 Third-line control

If possible, the institution should subject its accredited tasks to third-line control samples regularly. For this, ring tests can be used.

The frequency is such, that the sum of the second- and third-line checks is at least 4 a year. If possible, at least one of these should be a third-line control (ring test).

For the third-line control, the procedural aspects should be laid down in a separate document.

For a non-routine task, the third-line control is necessary during the periods that the task is being performed.



E 5.3.1 Testing the results of a ring test

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

For each parameter, the z-score is calculated according to:

$$Z_i = \frac{X_i - X_{ref}}{s}$$

in which :

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

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

$$S_{W,eis} > S_{ring} \rightarrow s = S_{W,eis}$$

$$S_{W,eis} < S_{ring} \rightarrow s = S_{ring}$$

in which :

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

If the number of participants is less than 6, the standard deviation, s , will equal $S_{W,eis}$. The z-values are calculated and evaluated only for components with a level higher than 5 times the determined AG. The result of a component or group component is deviates significantly from the assigned value, if:

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

In the table below, by way of illustration the borderline cases have been filled in. Firstly, the test is performed on the latest ring test. The maximum number of exceedings must here be lower than what is given in column 2 and column 3. If no significant deviation is found within the last ring test or the last but one ring test, then the last two investigations are tested against the requirements given in the columns 2 and 3.

The order given in the table has been chosen arbitrarily and is of no importance for testing in respect of a significant deviation. The evaluation only concerns the number of exceedings and the type (II or III) to which they belong.



Table: Criteria for the evaluation of ring tests¹

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** = $|z| < 2 \text{ sigma}$ (content lies within the 95% reliability range), **II** = $2 \text{ sigma} < |z| < 3 \text{ sigma}$ (content lies outside the 95% reliability range) and **III** = $|z| > 3 \text{ sigma}$ (an exceeding, determine its cause); ²) z-scores per parameter that are allowed within one ring test; ³) z-scores per 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 must be taken into account. It may occur, that ring test n is only reported, after the results of ring test n+1 have already been submitted. Only after the submission of ring test n+2 it can be determined whether the results of a given component in both ring test n and in ring test n+1 presented deviations. With this, it is possible that the same deviation also occurs in ring test n+2 (which had already been submitted), without the laboratory being responsible for not having undertaken timely action.

By way of illustration, the following diagram renders this graphically.



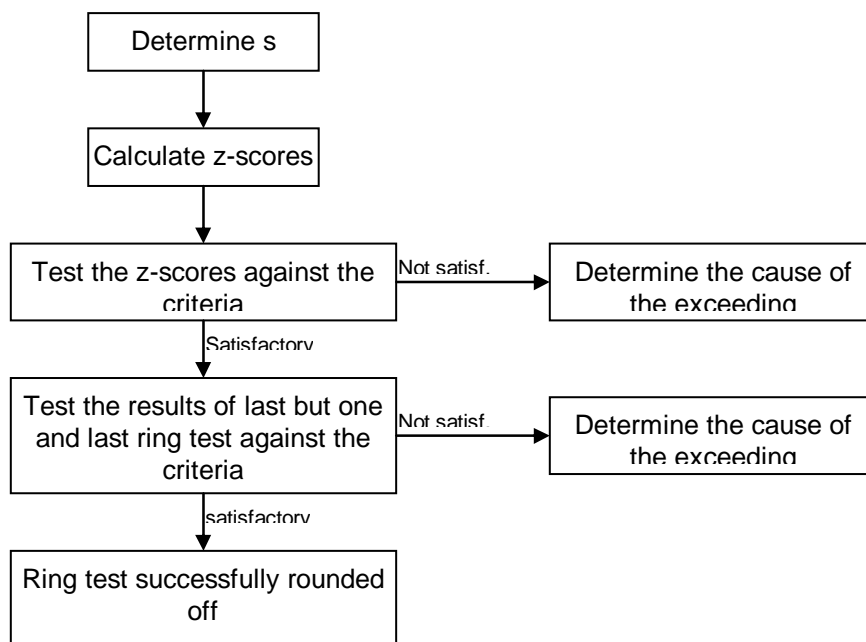


Figure Diagram testing results of ring test

If a result deviates significantly, the laboratory has to investigate the possible cause of this.

During this investigation, the following actions must be undertaken:

- analyse the quality problem, based on the results of the last successful ring tests, internal data of the quality assurance and the relevant measurements;
- draw up a plan for corrective actions;
- record the execution of the corrective action(s);
- check, if the corrective action is/has been successful.

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

- execute a second-line control;
- if the self-reprocessed destructure/extract is still present, let the content be determined by another laboratory accredited for AP04.



E 1 Technical description and quality assurance of tasks

The performance sheet mentions all requirements of the task to be validated. A distinction is made between the AG required for moulded and for non-moulded materials respectively.

The method must be validated for both validation matrices, see also E4. Each of the matrices states the requirements the task must fulfil.

Remark: In most cases, the detection limit of the method for a heavier matrix will be higher than for a relatively clean matrix. In those cases for which it is not certain if the legally required detection limit (AG_{eis}) can be obtained for the heavier matrix, this is indicated by a footnote.



Performance sheet E.I Determination of lead in eluates

Principle

Lead in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated under 'Method' completely. If the work is being performed in conformance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

Name	CAS-number	eluates (µg/l)			
		Maximally allowed leaching	AG _{eis}		
		Non-moulded	Moulded	Non-moulded	Moulded
Lead	7439-92-1	100	41	30	10

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7321, draft-NEN 6964 AAS-grafiphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	NEN 6453, draft-NEN 6965 AAS-flame
	Draft-NEN 6427 ICP-MS

Demonstrating equivalence Twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.

Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 µS/cm) Matrix II (20000 µS/cm)
Detection limit	<AG _{eis} (please refer to "Analytes to be determined" above) <AG _{eis} (please refer to "Analytes to be determined" above)
Blank Control sample)	< AG
Analyte(s)	all (E5.1.2.)
Recovery	
Lead	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Lead	< 7,5 % < 10 %
Intra-laboratory reproducibility variation coefficient	
Lead	< 10 % < 13 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation performance sheet: lead

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement range of the task

Investigate analytes once a quarter on:

lead

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.II Determination of cadmium in eluates

Principle

Cadmium in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the worked is performed in conformance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$)			
		maximally allowed leaching	AG_{eis}		
		Non-moulded	Moulded	Non-moulded	Moulded
Cadmium	7440-43-9	2,3	0,38	0,7	0,1

Method and quality assurance

		Eluates	
METHOD			
Sampling			
Preservation conditions		n/a	
Preservation period		n/a	
Sample pretreatment			
Sample size		n/a	
Preservation conditions		SIKB-protocol 3001	
Preservation period		SIKB-protocol 3001	
Task			
		Standard	technique
		NVN 7321, draft-NEN 6964	AAS-graphite oven
		NVN 7322, draft-NEN 6966	ICP-AES
		NEN 6452, draft-NEN 6965	AAS-flame
		draft-NEN 6427	ICP-MS
Demonstrating equivalence			
		twelve eluates with a concentration in the area, in which equivalence is desirable The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.	
Related task		n/a	
Multiple		n/a	
FIRST-LINE CONTROL			
		Matrix I (1000 $\mu\text{S/cm}$)	Matrix II (20000 $\mu\text{S/cm}$)
Detection limit			
		< AG_{eis} (see "Analytes to be determined" above)	< AG_{eis} (see "Analytes to be determined" above)
Blank			
		< AG	
Control sample)			
Analyte(s)		all (E5.1.2.)	
Recovery			
Cadmium		80 - 110 %	65 - 110 %
Repeatability variation coefficient			
Cadmium		< 7,5 %	< 10 %
Intra-laboratory reproducibility variation coefficient			
Cadmium		< 10 %	< 13 %



Continuation performance sheet: cadmium

Additional quality assurance points

Proc.internal stand.	n/a	
Confirmation	For ICP-AES:	result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).

Specific points of attention n/a

SECOND-LINE CONTROL

Eluates	Concentration range of analytes in the sample	within the measurement range the task
	Investigate analytes once a quarter on:	cadmium

THIRD-LINE CONTROL

Ring tests	
Reporting limit ring test	AG



Performance sheet E.III Determination of zinc in eluates

Principle

Zinc in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$) maximally allowed leaching	AG_{eis}		
			Moulded	Non-moulded	Moulded
Zinc	7440-66-5	240	69	70	20

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	technique
	NVN 7321, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	NEN 6443, draft-NEN 6965 AAS-flame
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$)
	Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above)
Blank	< AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Zinc	80 - 110 %
Repeatability variation coefficient	
Zinc	< 7,5 %
Intra-laboratory reproducibility variation coefficient	
Zinc	< 10 %
Additional quality assurance	
points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length fulfils the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: zinc

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample	within the measurement range of the task
Investigate analytes once a quarter on:	zinc

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test	AG
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Performance sheet E.IV Determination of nickel in eluates

Principle

Nickel in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$)			
		maximally allowed leaching	AG_{eis}		
		Non-moulded	Moulded	Non-moulded	Moulded
Nickel	7440-02-0	72	17	20	5

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7321, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	NEN 6456, draft-NEN 6965 AAS-flame
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$) Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above) < AG_{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Nickel	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Nickel	< 7,5 % < 10 %
Intra-laboratory reproducibility variation coefficient	
Nickel	< 10 % < 13 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: nickel

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement
range of the task
Nickel

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.V Determination of arsenic in eluates

Principle

Arsenic in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (graphite oven or hydride technique), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$) maximally allowed leaching	AG_{eis}		
			Non-moulded	Moulded	Moulded
Arsenic	7440-38-2	83	14	20	5

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7323, draft-NEN 6964 AAS-hydrid
	NEN 6457, draft-NEN 6966 AAS-graphite oven
	NVN 7322, draft-NEN 6965 ICP-AES
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable. In the lowest area, concentrations may range between 4 $\mu\text{g/l}$ and 40 $\mu\text{g/l}$. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$)
	Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Arsenic	80 - 110 %
Repeatability variation coefficient	
Arsenic	< 7,5 %
Intra-laboratory reproducibility standard deviation	
Arsenic	< 10 %
Additional quality assurance points	
Proc.internal stand.	n/a



Confirmation	For ICP-AES:	result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a	

Continuation of performance sheet: arsenic

SECOND-LINE CONTROL

Eluates	Concentration range of analytes in the sample	within the measurement range of the task
	Investigate analytes once a quarter on	arsenic

THIRD-LINE CONTROL

Ring tests	
Reporting limit ring test	AG



Performance sheet E.VI Determination of chromium in eluates

Principle

Chromium in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$)		AG_{eis}	
		maximally allowed leaching			
		Non-moulded	Moulded	Non-moulded	Moulded
Chromium	7440-47-3	41	48	10	10

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7321, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	NEN 6448, draft-NEN 6965 AAS-flame
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$) Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above) < AG_{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Chromium	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Chromium	< 7,5 % < 10 %
Intra-laboratory reproducibility variation coefficient	
Chromium	< 10 % < 13 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: chromium

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement
of the task
chromium

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.VII Determination of copper in eluates

Principle

Copper in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$) maximally allowed leaching	AG_{eis}		
			Moulded	Non-moulded	Moulded
Copper	7440-50-8	35	17	10	5

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	technique
	NVN 7321, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	NEN 6451, draft-NEN 6965 AAS-flame
	draft-NEN 6427 ICP-MS

Demonstrating equivalence twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.

Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$)
	Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above)
	< AG_{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Copper	80 - 110 %
	65 - 110 %
Repeatability variation coefficient	
Copper	< 7,5 %
	< 10 %
Intra-laboratory reproducibility variation coefficient	
Copper	< 10 %
	< 13 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: copper

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample within the measurement range of the task
Investigate analytes once a quarter on: copper

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test AG



Performance sheet E.VIII Determination of mercury in eluates

Principle

Mercury in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2 en 0,2 g/l dichromate. Subsequently, the samples are measured with an AAS (cold vapour).

Conformity with the standard

The execution on the basis of this performance sheet follows the standard as it is stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates (µg/l)		AG _{eis}	
		maximally allowed leaching			
		Non-moulded	Moulded	Non-moulded	Moulded
Mercury	7439-92-1	1,7	0,15	0,5	0,04

Method and quality assurance

Eluates

METHOD

Sampling

Preservation conditions n/a
 Preservation period n/a

Sample pretreatment

Sample size n/a
 Preservation conditions SIKB-protocol 3001
 Preservation period SIKB-protocol 3001

Task

Standard NEN 7324 technique AAS-cold vapour

Demonstrating equivalence

twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.

Related task n/a
 Multiple n/a

FIRST-LINE CONTROL

Matrix I (1000 µS/cm) Matrix II (20000 µS/cm)

Detection limit

<AG_{eis} (see "Analytes to be determined" above) <AG_{eis} (see "Analytes to be determined" above)

Blank

Control sample)

Analyte(s) all (E5.1.2.)

Recovery

Mercury 80 - 110 % 65 - 110 %

Repeatability variation coefficient

Mercury < 7,5 % < 10 %

Intra-laboratory reproducibility variation coefficient

Mercury < 10 % < 13 %

Additional quality assurance points

Proc.internal stand. n/a
 Confirmation n/a

Specific points of attention

n/a

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample within the measurement range of the task
 Investigate analytes once a quarter on: mercury

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test AG



Performance sheet E.IX Determination of molybdenum in eluates

Principle

Molybdenum in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently the samples are measured with an AAS (graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$) maximally allowed leaching	$AG_{\text{requirement}}$		
			Moulded	Non-moulded	Moulded
Molybdenum	7439-98-7	18	4,8	5	1

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7321, draft-NEN 6965 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable. In the lowest area, concentrations may range between 1 $\mu\text{g/l}$ en 10 $\mu\text{g/l}$. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$) Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above) < AG_{eis} (see "Analytes to be determined" above)
Blank Control sample)	<AG
Analyte(s)	all (E5.1.2.)
Recovery	
Molybdenum	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Molybdenum	< 10 % < 13 %
Intra-laboratory reproducibility variation coefficient	
Molybdenum	< 13 % < 17 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: molybdenum

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement range of the task molybdenum

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.X Determination of barium in eluates

Principle

Barium in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates (µg/l) maximally allowed leaching	AG _{requirement}		
			Non-moulded	Moulded	Moulded
Barium	7440-39-3	230	206	60	60

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7321, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	draft-NEN 6427 ICP-MS
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 µS/cm) Matrix II (20000 µS/cm)
Detection limit	<AG _{eis} (see "Analytes to be determined" above) <AG _{eis} (see "Analytes to be determined" above)
Blank Control sample)	<AG
Analyte(s)	all (E5.1.2.)
Recovery	
Barium	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Barium	< 7,5 % < 10 %
Intra-laboratory reproducibility variation coefficient	
Barium	< 10 % < 13 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: barium

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement range of the task
barium

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.XI Determination of tin in eluates

Principle

Tin in the eluate is kept dissolved by acidising it with nitric acid or hydrochloric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$) maximally allowed leaching	AG_{eis}		
			Non-moulded	Moulded	Moulded
Tin	7440-31-5	9.3	10	2	3

Method and quality assurance

		Eluates	
METHOD			
Sampling			
Preservation conditions		n/a	
Preservation period		n/a	
Sample pretreatment			
Sample size		n/a	
Preservation conditions		SIKB-protocol 3001	
Preservation period		SIKB-protocol 3001	
Task			
		Standard	technique
		NVN 7321, draft-NEN 6964	AAS-graphite oven
		NVN 7322, draft-NEN 6966	ICP-AES
		draft-NEN 6427	ICP-MS
Demonstrating equivalence			
		twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.	
Related task		n/a	
Multiple		n/a	
FIRST-LINE CONTROL			
		Matrix I (1000 $\mu\text{S/cm}$)	Matrix II (20000 $\mu\text{S/cm}$)
Detection limit		< AG_{eis} (see "Analytes to be determined" above)	< AG_{eis} (see "Analytes to be determined" above)
Blank		<AG	
Control sample)			
Analyte(s)		all (E5.1.2.)	
Recovery			
Tin		80 - 110 %	65 - 110 %
Repeatability variation coefficient			
Tin		< 10 %	< 13 %
Intra-laboratory reproducibility variation coefficient			
Tin		< 13 %	< 17 %
Additional quality assurance points			
Proc.internal stand.		n/a	
Confirmation		For ICP-AES:	result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention			
		n/a	



Continuation of performance sheet: tin

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement range of the task
tin

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.XII Determination of cobalt in eluates

Principle

Cobalt in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently the samples are measured with an AAS (graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates (µg/l) maximally allowed leaching	AG _{requirement}		
			Non-moulded	Moulded	Moulded
Cobalt	7489-43-7	24	10	7	3

Method and quality assurance

Eluates

METHOD

Sampling

Preservation conditions n/a
 Preservation period n/a

Sample pretreatment

Sample size n/a
 Preservation conditions SIKB-protocol 3001
 Preservation period SIKB-protocol 3001

Task

Standard technique

NVN 7321, draft-NEN 6964 AAS-graphite oven
 NVN 7322, draft-NEN 6966 ICP-AES
 draft-NEN 6427 ICP-MS

Demonstrating equivalence

twelve eluates with a concentration in the area, in which equivalence is desirable. In the laagste gebied mogen concentraties tussen 3 µg/l en 30 µg/l genomen worden. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.

Related task n/a
 Multiple n/a

FIRST-LINE CONTROL

Matrix I (1000 µS/cm) Matrix II (20000 µS/cm)

Detection limit

<AG_{eis} (see "Analytes to be determined" above) <AG_{eis} (see "Analytes to be determined" above)

Blank

Control sample)

Analyte(s) all (E5.1.2.)

Recovery

Cobalt 80 - 110 % 65 - 110 %

Repeatability variation coefficient

Cobalt < 10 % < 13 %

Intra-laboratory reproducibility variation coefficient

Cobalt < 13 % < 17 %

Additional quality assurance points

Proc.internal stand. n/a
 Confirmation For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).

Specific points of attention

n/a



Continuation of performance sheet: cobalt

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample within the measurement range of the task
Investigate analytes once a quarter on: cobalt

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test AG



Performance sheet E.XIII Determination of antimony in eluates

Principle

Antimony in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently the samples are measured with an AAS (hydride of graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates (µg/l) maximally allowed leaching	AG _{eis}		
			Non-moulded	Moulded	Moulded
Antimony	7440-36-0	3	1,3	0,9	0,4

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7323, draft-NEN 5760 AAS-hydride
	NVN 6611, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 µS/cm) Matrix II (20000 µS/cm)
Detection limit	<AG _{eis} (see "Analytes to be determined" above) <AG _{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Antimony	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Antimony	< 10 % < 13 %
Intra-laboratory reproducibility variation coefficient	
Antimony	< 13 % < 17 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: antimony

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement range of the task antimony

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.XIV Determination of selenium in eluates

Principle

Selenium in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (hydride or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$)		AG_{eis}	
		maximally allowed leaching		Non-moulded	Moulded
Selenium	7782-49-2	3,2	0,48	0,9	0,7

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	Standard technique
	NVN 7323, draft-NEN 5760 AAS-hydride
	NEN 6612, draft-NEN 6964 AAS-graphite oven
	NVN 7322, draft-NEN 6966 ICP-AES
	draft-NEN 6427 ICP-MS
Demonstrating equivalence	twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	Matrix I (1000 $\mu\text{S/cm}$) Matrix II (20000 $\mu\text{S/cm}$)
Detection limit	< AG_{eis} (see "Analytes to be determined" above) < AG_{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery	
Selenium	80 - 110 % 65 - 110 %
Repeatability variation coefficient	
Selenium	< 10 % < 13 %
Intra-laboratory reproducibility variation coefficient	
Selenium	< 13 % < 17 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention	n/a



Continuation of performance sheet: selenium

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample

within the measurement range of the task selenium

Investigate analytes once a quarter on:

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



Performance sheet E.XV Determination of vanadium in eluates

Principle

Vanadium in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently, the samples are measured with an AAS (flame- or graphite oven), ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standards as they are stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$) maximally allowed leaching	AG_{eis}		
			Non-moulded	Moulded	Moulded
Vanadium	7440-62-2	110	80	30	20

Method and quality assurance

		Eluates	
METHOD			
Sampling			
Preservation conditions		n/a	
Preservation period		n/a	
Sample pretreatment			
Sample size		n/a	
Preservation conditions		SIKB-protocol 3001	
Preservation period		SIKB-protocol 3001	
Task			
		Standard	technique
		NVN 7321, draft-NEN 6964	AAS-graphite oven
		NVN 7322, draft-NEN 6966	ICP-AES
		draft-NEN 6427	ICP-MS
Demonstrating equivalence			
		twelve eluates with a concentration in the area, in which equivalence is desirable. The other requirements in respect of the matrix investigation (E4.2.1) must be fulfilled.	
Related task		n/a	
Multiple		n/a	
FIRST-LINE CONTROL			
		Matrix I (1000 $\mu\text{S/cm}$)	Matrix II (20000 $\mu\text{S/cm}$)
Detection limit		< AG_{eis} (see "Analytes to be determined" above)	< AG_{eis} (see "Analytes to be determined" above)
Blank		<AG	
Control sample)			
Analyte(s)		all (E5.1.2.)	
Recovery			
Vanadium		80 - 110 %	65 - 110 %
Repeatability variation coefficient			
Vanadium		< 7,5 %	< 10 %
Intra-laboratory reproducibility variation coefficient			
Barium		< 10 %	< 13 %
Additional quality assurance points			
Proc.internal stand.		n/a	
Confirmation		For ICP- AES:	result of second wave length meets the duplicate criterion under repeatability conditions (E5.1.4.1) (results are not averaged).
Specific points of attention			
		n/a	



Continuation of performance sheet: vanadium

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample within the measurement range of the task
Investigate analytes once a quarter on: vanadium

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test AG



Performance sheet E.XVI Determination of cyanides (free and complex) in eluates

Principle

Cyanides (free and complex) in the eluate are kept dissolved by alkalising the solution with 10 M NaOH to a pH>12.

Determination of the total content of cyanide

Complex-bound cyanide is rendered soluble at a pH of 3,8 by the action of UV-light. A UV-B-lamp and a spiral of borosilicate glass are being used for this. UV-light with a wave length smaller than 290 nm is filtered out. Subsequently, the hydrogen cyanide, which is made available at a pH of 3,8 and a temperature of 125 °C, is overdistilled and photometrically determined.

Determination of the content of free cyanide

Preceding the distillation, a zinc sulphate solution is added to the sample stream, as a result of which any iron cyanides present precipitate as zinc cyanoferrate complex. Subsequently, the cyan hydrogen, which becomes available at a pH of 3,8 and a temperature of 125 °C, is overdistilled and photometrically determined.

Conformity with the standard

The execution on the basis of this performance sheet follows the standard as it is stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates (µg/l)		AG _{eis}	
		maximally allowed leaching		Non-moulded	Moulded
		Non-moulded	Moulded	Non-moulded	Moulded
Cyanides (total-free)		0,25	0,48	1	1
Cyanides (total-complex)		1,3	2,4	1	1



Remark:

With the method prescribed, it is not possible to measure to the level of the maximally allowed leaching for cyanide (total-free). This is also shown by the AG_{eis} mentioned above, which is higher than the maximally allowed leaching. At the time of publication of this document, in connection with the standardisation, the improvement of the method was being worked on. In due time, it will therefore be possible to perform measurements up to the level of the maximally allowed leaching of cyanide (total-free).

Continuation of performance sheet: cyanides

Method and quality assurance

	Eluates	
METHOD		
Sampling		
Preservation conditions	n/a	
Preservation period	n/a	
Sample pretreatment		
Sample size	n/a	
Preservation conditions	SIKB-protocol 3001	
Preservation period	SIKB-protocol 3001	
Task		
Related task	NEN-EN-ISO 14403	
Multiple	n/a	
FIRST-LINE CONTROL		
	Matrix I (1000 µS/cm)	Matrix II (2000 µS/cm)
Detection limit	<AG _{eis} (see "Analytes to be determined" above)	<AG _{eis} (see "Analytes to be determined" above)
Blank Control sample)	<AG	
Analyte(s)	K ₃ Fe(CN) ₆ , Thiocyanate, KCN	
Recovery		
Cyanides (total-free)		
K ₃ Fe(CN) ₆	< 7 %	< 7 %
Thiocyanate	< 0,5 % (on the basis of 1000 µg/l)	< 0,5 % (on the basis of 1000 µg/l)
KCN	80 - 110%	65 - 110 %
Cyanides (total-complex)		
K ₃ Fe(CN) ₆	80 - 110 %	65 - 110 %
Thiocyanate	< 1 % (on the basis of 1000 µg/l)	< 1 % (on the basis of 1000 µg/l)
KCN	80 - 110 %	65 - 110 %
Repeatability variation coefficient		
Cyanides (total-free)	< 7,5 %	< 10 %
Cyanides (total-free)	< 7,5 %	< 10 %
Intra-laboratory reproducibility variation coefficient		
Cyanides (total-free)	< 10 %	< 13 %
Cyanides (total-free)	< 10 %	< 13 %
Additional quality assurance points		
Proc.internal stand.	n/a	
Confirmation	n/a	
Specific points of attention	n/a	
SECOND-LINE CONTROL		
Eluates	Concentration range of analytes in the sample	within the measurement range of the task
	Investigate analytes once a quarter on:	K ₃ Fe(CN) ₆ , thiocyanate and cyanide
THIRD-LINE CONTROL		
Ring tests		
Reporting limit ring test	AG	



Performance sheet E.XVII Determination of bromide, chloride and sulfate in eluates

Principle

The eluates are supplied in a non-preserved condition. The different anions (fluoride, bromide, chloride, nitrate and sulfate) are separated on the basis of their relative affinity for a very alkaline anion-exchange columns. The separated anions are detected conductometrically and quantified.

Conformity with the standard

The execution on the basis of this performance sheet follows the standard as it is stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$)		AG_{eis}	
		maximally allowed leaching			
		Non-moulded	Moulded	Non-moulded	Moulded
Chloride ¹⁾	7782-50-5	56400	6200	10000	1000
Bromide ¹⁾	7726-95-6	270	10	80 ²⁾	3 ²⁾
Sulfate ¹⁾	7704-34-9	109700	9300	30000	2000

¹⁾ For the ionchromatographic analysis method, the lowest value of the linear range is at least equal to the lowest maximally allowed leaching. (Chloride 6200 $\mu\text{g/l}$, Bromide 10 $\mu\text{g/l}$ and Sulfate 9300 $\mu\text{g/l}$)

²⁾ In respect of validation matrix II, an interim AG_{eis} of 500 $\mu\text{g/l}$ by interference of nitrate applies.

Method and quality assurance

	Eluates	
METHOD		
Sampling		
Preservation conditions	n/a	
Preservation period	n/a	
Sample pretreatment		
Sample size	n/a	
Preservation conditions	SIKB-protocol 3001	
Preservation period	SIKB-protocol 3001	
Task		
Related task	n/a	
Multiple	n/a	
FIRST-LINE CONTROL		
	Matrix I	Matrix II
	distilled water	(136 mg NaNO_3/l and 1100 mg Na-acetate/l)
Detection limit	< AG_{eis} (see "Analytes to be determined" above)	< AG_{eis} (see "Analytes to be determined" above)
Blank		
Control sample)	<AG	
Analyte(s)	all	
Recovery		
Chloride	80 - 110 %	80 - 110 %
Bromide	80 - 110 %	65 - 110 % ³⁾
Sulfate	80 - 110 %	80 - 110 %
Repeatability variation coefficient		
Chloride	< 6 %	< 6 %
Bromide	< 7,5 %	< 10 %
Sulfate	< 6 %	< 6 %



Continuation of performance sheet: bromide, chloride en sulfate

Intra-laboratory reproducibility variation coefficient

Chloride	< 8 %	< 8 %
Bromide	< 10 %	< 13 %
Sulfate	< 8 %	< 8 %

Additional quality assurance points

Proc.internal stand. n/a

Confirmation n/a

Specific points of attention n/a

- Ionchromatographic analysis methods have lower and upper linear limits. The validation matrices are different from the other parameters.
- Trennzahl between all analytes must be at least 0,5

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample within the measurement range of the task
Investigate analytes once a quarter on: all

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test AG



Performance sheet E.XVIII Determination of fluoride in eluates

Principle

The eluates are supplied in a non-preserved condition. Fluoride in the eluate is potentiometrically determined with a fluoride-selective electrode, whether in a flow-through system or not.

Conformity with the standard

NEN 6589:

The execution on the basis of this performance sheet follows the standard completely. Due to the fact that the standard has a different field of application (rain water), no conformity with the standard can be claimed.

NEN 6483

The execution on the basis of this performance sheet follows the standard completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates (µg/l) maximally allowed leaching	AG _{eis}		
			Non-moulded	Moulded	Moulded
Fluoride	7782-41-4	430	450	100	100

Method and quality assurance

	Eluates	
METHOD		
Sampling		
Preservation conditions	n/a	
Preservation period	n/a	
Sample pretreatment		
Sample size	n/a	
Preservation conditions	SIKB-protocol 3001	
Preservation period	SIKB-protocol 3001	
Task	NEN 6589 en NEN 6483	
Related task	n/a	
Multiple	n/a	
FIRST-LINE CONTROL	Matrix I distilled water	Matrix II (136 mg NaNO ₃ /l and 1100 mg Na-acetate/l)
Detection limit	<AG _{eis} (see "Analytes to be determined" above)	<AG _{eis} (see "Analytes to be determined" above)
Blank	<AG	
Control sample)		
Analyte(s)	all (E5.1.2.)	
Recovery		
Fluoride	80 - 110 %	80 - 110 %
Repeatability variation coefficient		
Fluoride	< 7,5 %	< 7,5 %
Intra-laboratory reproducibility variation coefficient		
Fluoride	< 10 %	< 10 %
Additional quality assurance points		
Proc.internal stand.	n/a	
Confirmation	n/a	
Specific points of attention		
-	Fluoride, chloride, sulfate and bromide have a different validation matrix from the heavy metals and cyanides.	
-	The calibration curve of CF-Fluoride is <u>non</u> -linear	



Continuation of performance sheet: fluoride

SECOND-LINE CONTROL

Eluates

Concentration range of analytes in the sample	within the measurement range of the task
Investigate analytes once a quarter on	fluoride

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test	AG
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Performance sheet E.XIX Determination of sodium en potassium in eluates

Principle

Potassium in the eluate is kept dissolved by acidising it with nitric acid to a pH 1-2. Subsequently the samples are measured with an AAS or AES flame, ICP-AES or ICP-MS.

Conformity with the standard

The execution on the basis of this performance sheet follows the standard as it is stated here under 'Method' completely. If the work is performed in accordance with the performance sheet, conformity with the standard may be claimed.

Analytes to be determined

name	CAS-number	eluates ($\mu\text{g/l}$)		AG _{eis}	
		maximally allowed leaching		Non-moulded	Moulded
Sodium	7440-23-5	Non-moulded	Moulded	1000	1000
Potassium	7440-09-7			1000	1000

Method and quality assurance

Eluates	
METHOD	
Sampling	
Preservation conditions	n/a
Preservation period	n/a
Sample pretreatment	
Sample size	n/a
Preservation conditions	SIKB-protocol 3001
Preservation period	SIKB-protocol 3001
Task	
	Standard
	NEN 6426, draft-NEN 6966
	NEN 6423, NEN 6424, draft-NEN 6965
	NEN 6442
	draft-NEN 6427
	technique
	ICP-AES
	AAS-flame
	flame-photometric
	ICP-MS
Related task	n/a
Multiple	n/a
FIRST-LINE CONTROL	
	Matrix I
	117 mg/l CaCl ₂
Detection limit	<AG _{eis} (see "Analytes to be determined" above)
	Matrix II
	2230 mg/l CaCl ₂
	<AG _{eis} (see "Analytes to be determined" above)
Blank	<AG
Control sample)	
Analyte(s)	all (E5.1.2.)
Recovery • • • Sodium and Potassium	• Sodium and Potassium • 80 - 110 % Sodium and Potassium
Repeatability variation coefficient	
Sodium and Potassium	< 7,5 %
Intra-laboratory reproducibility variation coefficient	
Sodium and Potassium	< 10 %
Additional quality assurance points	
Proc.internal stand.	n/a
Confirmation	For ICP-AES: result of second wave length meets criteria under repeatability conditions of E5.1.4.1 (results are not averaged).
Specific points of attention	
-	Sodium and potassium have a different validation matrix.
SECOND-LINE CONTROL	
Eluates	Concentration range of analytes in the sample
	Investigate analytes once a quarter on:
	within the measurement range of the task
	all



Continuation of performance sheet: sodium and potassium

THIRD-LINE CONTROL

Ring tests

Reporting limit ring test

AG



E 2 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.
NEN 6423:	1988	Water - Bepaling van het gehalte aan natrium met behulp van atomaire-absorptiespectrometrie (vlamtechniek).
NEN 6424	1988	Water - Bepaling van het gehalte aan kalium met behulp van atomaire-absorptiespectrometrie (vlamtechniek).
NPR 6425	1995	Atomaire-emissiespectrometrie met inductief gekoppeld plasma - Algemene richtlijnen.
Ontw. NEN 6427	1999	Water – Bepaling van 66 elementen met inductief gekoppeld plasma massaspectrometrie.
NEN 6429:	1994	Water en slibhoudend water - Bepaling van het gehalte aan lood met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6430:	1994	Water en slibhoudend water - Bepaling van het gehalte aan nikkel met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6432:	1993	Water en slibhoudend water - Bepaling van het gehalte aan arseen met atomaire-absorptiespectrometrie (hydridetechniek).
NEN 6433:	1993	Water en slibhoudend water - Bepaling van het gehalte aan antimoon met atomaire-absorptiespectrometrie (hydridetechniek).
NEN 6434:	1993	Water en slibhoudend water - Bepaling van het gehalte aan seleen met atomaire-absorptiespectrometrie (hydridetechniek).
NEN 6436	1997	Water en slibhoudend water - Bepaling van het gehalte aan barium met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6442	1979	Water - Vlamfotometrische bepaling van het gehalte aan natrium en kalium.
NEN 6443:	1977	Water en slibhoudend water - Bepaling van het gehalte aan zink met atomaire-absorptiespectrometrie (vlamtechniek).
NEN 6444:	1990	Water en slibhoudend water - Bepaling van het gehalte aan chroom met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6445:	1997	Water - Bepaling van het gehalte aan kwik na ontsluiting met broom met atomaire-absorptiespectrometrie (koude dampstechniek)
NEN 6448:	1981	Water en slibhoudend water - Bepaling van het gehalte aan



NEN 6451:	1980	chromium met atomaire-absorptiespectrometrie (vlamtechniek). Water en slibhoudend water - Bepaling van het gehalte aan koper met atomaire-absorptiespectrometrie (vlamtechniek).
NEN 6452:	1980	Water en slibhoudend water - Bepaling van het gehalte aan cadmium met atomaire-absorptiespectrometrie (vlamtechniek).
NEN 6453:	1980	Water en slibhoudend water - Bepaling van het gehalte aan lood met atomaire-absorptiespectrometrie (vlamtechniek).
NEN 6454:	1994	Water en slibhoudend water - Bepaling van het gehalte aan koper met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6456:	1981	Water en slibhoudend water - Bepaling van het gehalte aan nikkel met atomaire-absorptiespectrometrie (vlamtechniek).
NEN 6457:	1994	Water en slibhoudend water - Bepaling van het gehalte aan arseen met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6458:	1983	Water en slibhoudend water - Bepaling van het gehalte aan cadmium met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6463:	1997	Water en slibhoudend water - Bepaling van het gehalte aan vanadium met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6468:	1994	Water en slibhoudend water - Bepaling van het gehalte aan cobalt met atomaire-absorptiespectrometrie (grafietoventechniek).
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.
NEN 6603:	1988	Water en slib - Richtlijnen voor interne kwaliteitsbeheersing met controlekaarten bij chemische analyses.
NEN 6611:	1981	Water en slibhoudend water - Bepaling van het gehalte aan antimoon met atomaire-absorptiespectrometrie (grafietoventechniek).
NEN 6612:	1979	Water en slibhoudend water - Bepaling van het gehalte aan seleen met atomaire-absorptiespectrometrie (grafietoventechniek).
ISO 6879	1995	Air quality - Performance characteristics and related concepts for air quality measuring methods.
Ontw. NEN 6964	2001	Analyse van 28 geselecteerde elementen - Atomaire-absorptiespectrometrie (grafietoventechniek). Betreft de elementen Ag, Al, As, B, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Li, Mg, Mn, Mo, Ni, Pb, Rb, Sb, Se, Sn, Sr, Te, Ti, Tl, V, Zn.



Ontw. NEN 6965	2002	Analyse van 32 geselecteerde elementen - Atomaire-absorptiespectrometrie (vlamtechniek). Betreft de elementen Al, Sb, As, Ba, Be, Bi, B, Ca, Cd, Ce, Cs, Cr, Co, Cu, K, Li, Pb, Mg, Mn, Mo, Na, Ni, Rb, Se, Si, Sr, Sn, Ti, Fe, V, Ag en Zn.
Ontw. NEN 6966	2003	Milieu - Analyse van geselecteerde elementen - Atomaire-emissie-spectrometrie met inductief gekoppeld plasma.
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 (inductiefgekoppeld plasma).
NVN 7323:	1997	Uitloogkarakteristieken van vaste grond- en steenachtige bouwmaterialen en afvalstoffen. Bepaling van het gehalte van anorganische componenten. Bepaling van het gehalte van arseen, antimoon en seleen met atomaire-absorptiespectrometrie (hydridegeneratietechniek).
NEN 7324:	1997	Uitloogkarakteristieken van vaste grond- en steenachtige bouwmaterialen en afvalstoffen - Bepaling van het gehalte van anorganische componenten - Bepaling van het gehalte van kwik met atomaire-absorptiespectrometrie (koude-dampstechniek).
NEN 7777	2003	Milieu – Prestatiekenmerken van meetmethoden.
NEN 7778	2003	Milieu – Gelijkwaardigheid van meetmethoden.
ISO 8466-1:	1990	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	1994	Luchtkwaliteit - Toepassing en bepaling van de prestatiekenmerken van een automatisch meetsysteem..
NEN-EN-ISO 14403	2002	Water - Bepaling van het totale gehalte aan cyanide en het gehalte aan vrij cyanide met behulp van continue doorstroomanalyse.
NEN-EN-ISO 10304-2:	1996	Water - Bepaling van opgeloste anionen met vloeistofionchromatografie - Deel 2: Bepaling van bromide, chloride, nitraat, nitriet, ortofosfaat en sulfaat in afvalwater.



Appendices

Appendix E1 Principles for the determination of maximally allowed leaching concentrations (MAU)

1 Non-moulded materials (column test)

Within the scope of the Accreditation Programme, all fractions of the column test up to L/S 10 are mixed before they are analysed. The detection limits of the individual compounds are therefore not dependent of their presence in the past fraction L/S 10. For calculating the maximally allowed leaching (MAU), an applied height of five meter in the combined extracts up to and including L/S 10 has been assumed.

2 Moulded materials (diffusion test)

In general, the required quantification limits increase in test pieces, in which the volume increases relatively more than the surface. In the calculations, a standard proctor of a height of 15 cm, a diameter of 15 cm and a mass of 3500 grams has been assumed. The concentration drift on the basis of diffusion shows the lowest concentration in the first four steps of the diffusion test. In steps 5 and 6 the concentration is 2x as high, and in the last four steps 4x higher. With these data the concentration has been determined, by which, with the diffusion coefficient calculated, the immission standard is being met. With formula [3], the immission standard is translated into the calculated emission (E_{64}) on the basis of diffusion. The calculation is performed in accordance with NEN 7345, § 9.5. According to NEN 7345, the average of the 8 measured concentrations would be larger than 1,5 times the quantification limit. In order to calculate the lowest concentration it has therefore been divided by 1,5.

$$I = E_{64} * f_{temp} * f_v [3]$$

f_{temp} = temperature factor (0.7)

f_v = extrapolation factor (max 15)



3 Results

component	AG - standard ²				
		Non-moulded building materials applied height 5,0 m		Moulded building materials layer thickness 0,3 m	
		MAU	AG-eis ¹	MAU	AG-eis ¹
lead	0,5	100	30	41	10
cadmium	0,02	2,3	0,7	0,38	0,1
zinc	0,05	240	70	69	20
nickel	1	72	20	17	5
arsenic	0,2	83	20	14	5
chromium	1	41	10	48	10
copper	0,3	35	10	17	5
mercury	0,03	1,7	0,5	0,15	0,04
cyanide (total-free)	1 ³	0,25	1	0,48	1
cyanides (total-complex)	1 ³	1,3	1	2,4	1
chloride	90 ⁴	56400	10000	6200	1000
bromide	50 ⁴	270	80	10	3
fluoride	2 ⁵	430	100	450	100
sulfate	100 ⁴	109700	30000	9300	2000
molybdenum	1	18	5	4,8	1
barium	2	230	60	206	60
tin	0,3	9,3	2	10	3
cobalt	1	24	7	10	3
antimony	0,1	3	0,9	1,3	0,4
selenium	0,2	3,2	0,9	0,48	0,7
vanadium	4	110	30	80	20

MAU is the maximally allowed leaching concentration for category I;

AG-eis is the highest detection limit with which the MAU can be quantified with a precision better than 10%;

¹lowest detection limit, AG, for leaching of all categories. In the performance sheets, this has been rounded off to a practicable value;

²lowest detection limit, AG, according to one of the standards 7321, 7322, 7323 of 7324 in a relatively light matrix;

³detection limit NEN-EN-ISO 14403;

⁴quantification limit NEN-EN-ISO 10304-2;

⁵detection limit NEN 6589.

