Accreditation scheme

Laboratory analyses for soil, sediment and groundwater investigation

AS SIKB 3000

DRAFT TRANSLATION

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Foundation Infrastructure for the Quality Assurance of Soil Management
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Preface

This accreditation scheme has been adopted by the Soil Management Accreditation Board, in which all interested parties in the field of soil and building materials are represented. Where this Accreditation Scheme refers to "Board of Experts", the above Board is meant.
This Accreditation Scheme was drawn up under the guidance of a monitoring committee in which the interests of the parties involved were represented. This monitoring committee has reviewed the specifics in collaboration with the project office of the SIKB and the Dutch Accreditation Council (RvA).
The board also monitors the execution of the accreditation and revises this accreditation scheme, if necessary.

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Chapter 1 Introduction

1.1 Subject

The requirements contained within this accreditation scheme (AS) are applied by the accrediting institution for the assessment of laboratories at the application for and maintenance of an accreditation for this AS. This concerns analyses performed during the investigation into the environmental health of soil, groundwater and sediment.

The certificate of conformity to be issued by the accrediting institution is designated as an accreditation for AS SIKB 3000 in combination with the assessment of the competence of the laboratory for corresponding protocols to conduct specific analyses. The contents of AS SIKB 3000 are not in breach of the standards of NEN-EN-ISO/IEC 17025.

1.2 Scope of application

AS SIKB 3000 refers to the analyses of soil, groundwater and sediment samples and comprises the steps in the procedure from the transfer of samples to the laboratory, up to and including the certificate of analysis, including the aftercare and archiving (chapter 2). The requirements regarding validation and the accrediting institutions are set out in the respective chapters 3 and 4.

This AS is consistent with BRL SIKB 2000 (Fieldwork for environmental soil investigation) and with Accreditation Scheme AS SIKB 2000 (Fieldwork for environmental soil and sediment investigation).
The quality of steps in the procedure which are of influence, but which are not included in this AS, are assured in BRL SIKB 2000 and AS SIKB 2000. These steps in the procedure are:
- sampling;
- transportation and storage of samples for transfer to the laboratory.

Matrix subdivision AS SIKB 3000:
Within this AS, laboratories may be accredited either for only one of the matrices soil, groundwater or sediment separately, or for two random matrices out of the three, or else for all three matrices.
Protocol subdivision AS SIKB 3000:
Within this AS, laboratories may be accredited for a number of protocols. Figure 1.1 provides a schematic coherence of AS SIKB 3000 with the attached protocols and displays which subdivision applies for the analyses within the corresponding protocols. See § 1.5 for further specification of the accreditation.

Figure 1.1. Subdivision analyses within accreditable protocols for soil, groundwater and sediment.

AS SIKB 3000 Testing regime		
Soil	**Groundwater**	**Sediment**
Protocol 3010: Basic package • Clay/DS/OS • Elemental Analysis: Ba, Cd, Co, Cu, (non-volatile) Hg, Mo, Ni, Pb and Zn • Mineral oil • PAH • PCB • pH-$CaCl_2$	**Protocol 3110:** Basic package • Elemental Analysis: Ba, Cd, Co, Cu, (non-volatile) Hg, Mo, Ni, Pb and Zn • Mineral oil • PAH • pH • Conductivity	**Protocol 3210:** Basic package • DS/OS • Clay • Elemental Analysis Ba, Cd, Co, Cu, (non-volatile) Hg, Mo, Ni, Pb and Zn • Mineral oil • PAH • PCB
Protocol 3020: Additional I • OCP • Other OCP: delta-HCH and endosulfan sulphate • Tri- and tetrachlorobenzenes, penta- and hexachlorobenzene	**Protocol 3120:** Additional I • OCP/PCB • Tri- and tetrachlorobenzenes, penta- and hexachlorobenzene	**Protocol 3220:** Additional I • OCP, penta- and hexachlorobenzene • Other OCP: delta-HCH and endosulfan sulphate
Protocol 3030: Additional II • Volatile aromatic compounds, Volatile chlorinated hydrocarbons, MTBE and ETBE • Monochlorobenzenes dichlorobenzenes • Other aromatic solvents	**Protocol 3130:** Additional II • Volatile aromatic compounds, volatile chlorinated hydrocarbons, MTBE and ETBE • Monochlorobenzenes dichlorobenzenes	**Protocol 3230:** Additional II • Monochlorobenzenes dichlorobenzenes • Tri- and tetrachlorobenzenes
Protocol 3040: Additional III • Chloride • Cyanides	**Protocol 3140:** Additional III • Chloride, nitrate, orthophosphate and sulphate • Cyanides	**Protocol 3240:** Additional III • Chloride • Cyanides • pH-H_2O
Protocol 3050: Additional IV • Elemental Analysis: Sb, As, Cr, Sn and V • Elemental Analysis: Ag, Be, Te and Tl	**Protocol 3150:** Additional IV • Elemental Analysis: Sb, As, Cr, Sn and V • Elemental Analysis: Ag, Be, Te and Tl	**Protocol 3250:** Additional IV • Elemental Analysis: As, Cr, Sb, Sn and V
Protocol 3070: Additional V • Asbestos	**Protocol 3190:** Investigation protocol for groundwater	**Protocol 3260:** Additional V • Pentachlorophenol • Organotin compounds
Protocol 3090: Investigation protocol for soil		**Protocol 3270:** Additional VI • Asbestos
		Protocol 3290: Investigation protocol for sediment

1.3 Requirements regarding the execution

The requirements for the execution of the laboratory investigation are laid down in NEN 7777 and NEN 7778 and in performance sheets that form part of the corresponding protocols. With the exception of asbestos, all parameters in soil samples must be subjected to an obligatory pretreatment, in conformity with NEN-EN 16179. With the exception of asbestos, all parameters in sediment samples must be subjected to an obligatory pretreatment, pursuant to NEN 5719. Soil and sediment samples for the determination of asbestos shall undergo pretreatment in conformity with NEN 5707.
The preservation method and the preservability of soil, groundwater and sediment samples for the different parameters are laid down in NEN-EN-ISO 5667-3, NEN-EN-ISO 5667-15, NEN-ISO 18512 and the SIKB protocol 3001.
The performance sheets contain the requirements that the execution of the work must comply with. As such, the protocols referred to in figure 1.1 form part of this AS.

1.4 Normative references

Table 1.1 presents the standards containing provisions that are provisions of this AS. At the moment of publication of the AS in question, the versions referred to are in force.

Table 1.1. List of normative documents.

Standard	Title
NEN-EN-ISO/IEC 17025:2005 (incl. C1:2007)	General requirements for the competence of testing and calibration laboratories
NEN-EN 16179:2012	Sludge, Treated Bio-waste and Soil – Guidance for Sample Pretreatment
NEN 5719:1999	Soil – Pretreatment of Sediment Samples
NEN 5861:1999	Environment – Procedures for the Transfer of Samples
NEN 6603:2010	Environment and Food – Internal Quality Control by the use of Control Charts with Chemical and Microbiological Analyses
NVN 6419:2008	Protocol for the Preparation of Synthetic Laboratory samples for Organic Components in Ground and Surface Water
NEN 7777:2011/C1:2012	Environment and Food – Performance Characteristics of Measuring Methods
NEN 7778:2003	Environment – Equivalency of Measuring Methods
NEN-EN-ISO 5667-3:2012	Water Quality – Sampling – Part 3: Preservation and Handling of Water Samples
NEN-EN-ISO 5667-15:2009	Water Quality – Sampling – Part 15: Guidance on the Preservation and Handling of Sludge and Sediment Samples
NEN-ISO 18512:2007	Soil Quality – Guidance on Long and Short Term Storage of Soil Samples
SIKB-protocol 3001:2009	Preservation Methods and Preservation Term of Soil Samples

In principle, the most recent version applies. In case of replacement of the normative documents referred to and of the standards (of analysis) as referred to in the performance sheet by a new Dutch or international standard, the old normative document may be applied during a transition period of 12 months. After this transition period, the new document will become operative and the old document will lapse. Within 18 months after commencement of the transition period, the altered situation must be assessed by the accrediting institution. The accredited institution must provide for this and take the initiative relevant thereto.

A list containing definitions as these apply for this AS is set out in Appendix 2.

1.5 Accreditation for AS SIKB 3000

ISO/IEC 17025 is leading regarding all aspects (requirements of management and technical requirements) within the laboratory. If the underlying normative documents are not complete, additional provisions have been included in AS SIKB 3000 with regard to the technical implementation. The quality requirements per analysis are included in the protocols.

The laboratory can only be accredited for this AS if it also has a valid accreditation in conformity with NEN-EN-ISO/IEC 17025. This accreditation must be awarded by the Dutch Accreditation Council, or by an organization the Dutch Accreditation Council has concluded a Multi-Lateral Agreement MLA (EA/IAF) with. The laboratory must also hold a signed agreement with the SIKB, in any case containing arrangements for the contribution regarding maintenance of this accreditation scheme.

An institution may arrange to be accredited per performance sheet in accordance with AS SIKB 3000. In order to be acknowledged for AS3000, the accreditation must cover all compounds or elements referred to in the performance sheet in the specified protocols. Therefore, it is not possible to be accredited for one compound when multiple compounds are listed in the performance sheet. It is not obligatory for the sum parameter to qualify for a complete package. The accreditation for the performance sheet in question, in conformity with this AS, will be stated on the scope of accreditation in the accreditation certificate.

In order to be acknowledged for AS SIKB 3000, a laboratory must at least be accredited for the tests (see Appendix 2) which are included in protocol 3010 (for soil samples) and/or 3110 (for groundwater samples) and/or 3210 (for sediment samples).
A laboratory may choose from one or more of the following additional protocols to supplement this acknowledgment:

- 3020, 3030, 3040, 3050, 3090 (3010 obligatory for all) and 3070 for soil;
- 3120, 3130, 3140, 3150 and 3190 for groundwater (3110 obligatory);
- 3220, 3230, 3240, 3250, 3260, 3290 (3210 obligatory for all) and 3270 for sediment.

Remark:
The abovementioned obligation of acknowledgement for protocols 3010 and/or 3210 does not apply to the protocols 3070 (Analysis of asbestos in soil) and 3270 (Analysis of asbestos in sediment). For these protocols a laboratory may be acknowledged without the obligatory acknowledgement for the respective protocols 3010 and 3210.

In order to be eligible for an expansion of the accreditation with one or more of these additional protocols, the laboratory must at least be accredited for the protocols for which it seeks this expansion.
To obtain acknowledgement, the laboratory may outsource (by contract) no more than one of the tests per (randomly chosen) protocol to an institution accredited for that purpose, in conformity with AS SIKB 3000, subject to the condition that the laboratory itself carries out at least one test.

A laboratory may also be accredited for this AS if it only analyzes samples with a specific characteristic. The limitation in the scope of application must be stated in the scope of accreditation. The details as to where and when outsourcing will take place must be included in the procedures of the laboratory, in case of a deviation from the scope of application the laboratory is accredited for.

1.6 Rules and regulations for application of logo AS SIKB 3000

Logo 'Quality assurance for soil management SIKB'
The logo 'Quality assurance for soil management SIKB' has been developed to provide clarity to all parties involved with regard to the quality assurance of activities in soil management. The laboratory analyses for environmental soil investigation described in this accreditation scheme fall within the scope of this logo. This means that laboratories accredited and acknowledged for analyses described in this accreditation scheme may acquire the right to apply the logo. The rules and regulations that the application of this logo is subject to, are included in Appendix 1.

Accreditation mark from the Dutch Accreditation Council (RvA)
By acquiring an accreditation for activities described in this accreditation scheme a laboratory may acquire the right to apply an accreditation mark of the accrediting institution.

Chapter 2 Requirements regarding the procedure

Table 2.1 provides an overview of the procedural steps a sample undergoes for soil, groundwater and sediment samples respectively, from the moment the sample is taken until the archiving of the results with reference to the relevant standards. This chapter contains additional requirements, relative to NEN-EN-ISO/IEC 17025.

Table 2.1 Procedural flow sheet AS SIKB 3000 for soil, groundwater and sediment samples.

Sampling		
Soil	Groundwater	Sediment
NEN 5740 NEN 5725 NEN 5104/C1 NEN 5706 NEN 5742 NEN 5743 NEN 5766 NPR 5741 NEN 5707 VKB 2001	NEN 5740 NEN 5744/A1 NPR 5741 VKB 2001 VKB 2002	NEN 5720 NEN 5717 NEN 5104/C1 NEN 5706 NEN 5742 NEN 5743 NPR 5741 NTA 5727 VKB 2003
Preservation and preservability terms		
Soil	Groundwater	Sediment
NEN-ISO 18512 SIKB 3001	NEN-EN-ISO 5667-3 SIKB 3001	NEN-EN-ISO 5667-15 SIKB 3001
Sample transfer		
Soil	Groundwater	Sediment
NEN 5861		
Sample pretreatment		
Soil	Groundwater	Sediment
NEN-EN 16179	NEN 5744/A1	NEN 5719
Analysis		
Soil	Groundwater	Sediment
See fig. 3.1, chapter 3 and corresponding protocols:		
3010 - 3070	3110 - 3150	3210 - 3270
Reporting and Archiving		
Soil	Groundwater	Sediment
NEN-EN-ISO/IEC 17025 and AS3000, § 2.7		

QUALITY ASSURANCE NEN-EN-ISO/IEC 17025

2.1 Preservation and transfer

2.1.1 Preservation

Where sample preservation is concerned, the procedural requirements that apply for the commissioning authority regarding the transfer of samples as laid down in BRL SIKB 2000 and AS SIKB 2000 must be complied with.

The prescribed preservations, which, depending on the parameter, may consist of a specific type of packaging, the addition of a preservative and/or specific storage conditions, are listed per parameter in:
- NEN-EN-ISO 5667-3;
- NEN-EN-ISO 5667-15;
- NEN-ISO 18512;
- SIKB-Protocol 3001.

Time of commencement of the preservation term for mixed samples:
For a mixed sample prepared from samples taken on different calendar dates, the date upon which the oldest sample was taken will apply for the preservation term to commence. This date will be passed on to the laboratory as the sampling date.

Remark:
The day of sampling is indicated as day 0. This means that a preservation term of two days will lapse at the end of day 2. (For a sample taken on a Monday, the preservation term will lapse on Wednesday. A sample taken on Friday will have a preservation term up to and including Sunday).

2.1.2 Transfer and acceptance of samples and sample data

This AS solely refers to sub-procedures carried out after the sample has been accepted by the laboratory. Samples are transferred and accepted in conformity with NEN 5861.

2.2 Transportation and storage of samples

Transportation upon sampling
The execution of and requirements for transportation of samples by the sampler to the laboratory or to a central storage location of the laboratory are laid down in BRL 2000 and AS2000, including the corresponding protocols.

Transportation under responsibility of the laboratory
The laboratory is responsible for transportation of samples from the central storage location of the laboratory to the laboratory itself. During this transportation, samples must be stored in conformity with NEN-EN-ISO 5667-3 for groundwater samples and in conformity with NEN-EN-ISO 5667-15 for sediment samples. Soil samples must be kept at a temperature of between 2 and 8 °C during this transportation, in conformity with the standards for groundwater and sediment.

Samples containing asbestos do not require transportation and storage under cooled conditions. However, these samples do require to be double-packed in buckets or bags, which are clearly labelled "Caution, contains asbestos".

Sample storage in the laboratory
The storage of samples in the laboratory and in the central storage location of the laboratory is carried out in conformity with NEN-EN-ISO/IEC 17025 (§ 5.8).

2.3 Sample Pretreatment

Sample pretreatment is understood to mean the taking of a (sub-) sample representative of the sample obtained during sampling. Sample pretreatment takes place in the laboratory.

Remarks:
- When a sample is characterized as suspect asbestos-containing material, the sample may not be dried and pulverized. In that case, from the field-moist sample a sub-sample will be taken in duplicate for each analysis. Both sub-samples will be analyzed. The average of both results will then be reported as the result. If either one of the results is below the reporting limit, then in order to calculate the average for this result, the value of 0.7 * the reporting limit will be used.
  If the ratio between the two duplicate results exceeds a factor of 2.5, a notification of this will be recorded in the analysis report. In the report a notification will be recorded, indicating that the field-moist sample was used as a starting point of the analysis (see § 2.5.1).
- When a sample visibly contains asbestos, but has not been characterized as suspect asbestos-containing material, the commissioning authority will be notified of this. Subsequently, parties will consult on how to further handle the sample.

The sample-quantity to be used during extraction/digestion is laid down in the standards for analysis.

Soil samples

Soil samples are pretreated in conformity with NEN-EN 16179. Exceptions are:
- Soil samples with over 20% (m/m) foreign material content. These samples are pretreated as indicated in Appendix 4.
- Soil samples to determine asbestos. These samples are pretreated in conformity with NEN 5707.
- Soil samples to determine volatile components that are delivered in core samplers. Take a sub-sample from such samples to determine volatile compounds through no less than three insertions into the soil across the entire length of the core sampler. Remove the remaining sample material from the core sampler and take a sub-sample of this to determine the dry matter content.

Remark: determining the dry matter content is not necessary if the sample material comes from another package of the same sample.

Remarks:
- In conformity with the Dutch Soil Quality Decree definition, when over 20% (m/m) foreign material content is present in soil samples, strictly speaking these do not constitute soil samples, however in practice these samples are often presented as such to the laboratory. In such cases, a standard remark must be recorded in the analysis report, stating that the sample has over 20% foreign material content (see § 2.5.1).

The maximum number of sub-samples or core samples used for a mixed sample is (also) dependent on the investigation strategy applied and is set out in NEN 5740. The number of sub-samples or core samples used to compose a mixed sample must be stated in the analysis report. The sample quantity to be used during sample pretreatment and the extraction/digestion is laid down in NEN-EN 16179.

Groundwater samples

Groundwater samples are pretreated as described in NEN 5744 and in the relevant standard of analysis.
In addition to these standards, the following applies:

Groundwater samples for inorganic parameters, in conformity with NEN 5744, will be filtered and preserved in the field during sampling. Prior to the application of (part of) the sample in the laboratory, these samples need to be homogenized.

Groundwater samples for organic parameters, in conformity with NEN 5744, will be sampled with a low flow rate during sampling in the field, to prevent sludge from being collected along with the sample and subsequently, it will be preserved. Prior to sub-sampling, these samples may not be filtered, but they must be allowed to settle for no less than 8 hours under storage conditions in conformity with NEN-EN-ISO 5667-3 or SIKB protocol 3001. Sub-sampling for organic analyses must be carried out in such a way that there is as little whirling of the precipitated particles as possible (for example by decanting carefully or by using a pipette).

Remarks:
The 8 hour timeframe for precipitation may be replaced by centrifuging the sample.
Both for inorganic and organic analyses, it is allowed to insert sample bottles directly into the analyzing equipment, if these are equipped with provisions for sub-sampling, on the understanding that the above conditions are not compromised.

If there is a floating layer present in a sample, this should not be included in the sub-sampling. In such case, sub-sampling will be carried out with the aid of a glass pipette to be inserted through the floating layer, so that only the groundwater underneath is sub-sampled. The quantity of the material of the floating layer may be measured gravimetrically. The presence of the floating layer and possibly its mass will be recorded in the analysis report by way of a notification to that effect.

Sediment samples

Sediment samples are pretreated in conformity with NEN 5720 and NEN 5719. Exceptions are:

- Sediment samples with over 20% (m/m) foreign material content. These samples are pretreated as indicated in Appendix 4.
- Sediment samples for the determination of asbestos. These samples are pretreated in conformity with NEN 5707.

Remarks:

- In conformity with the Dutch Soil Quality Decree definition, when over 20% (m/m) foreign material content is present in material that was released from the bottom or banks of a body of surface water, strictly speaking these do not constitute dredging spoil samples, however in practice these samples are presented as such to the laboratory. In such cases, a standard remark must be recorded in the analysis report, stating that the sample has over 20% foreign material content (see § 2.5.1).

2.4 Analysis

The quality assurance and recording of analyses is done in conformity with NEN-EN-ISO/IEC 17025, where there are various options to choose from for validation (see chapter 3, fig.3.1). In all cases compliance with the requirements in the performance sheets of the methods of analysis must be verified. If during the procedure of analysis it appears that the preservation term may be or will be exceeded, then, in case arrangements to that effect have been made with the client, the assignment may be carried out in conformity with the analysis. In case a preservation term is exceeded, this must be recorded in the analysis report by way of a standard remark. (see § 2.5.1).

2.5 Requirements regarding reporting to the commissioning authority

2.5.1 Deviations upon transfer, acceptance and analysis of samples

The requirements for reporting are described in NEN-EN-ISO/IEC 17025 (§ 5.10). If a deviation from this standard is observed, next to the results of the investigation, a standard remark must be recorded in the analysis report. In this standard remark it must be specified which deviation is observed and that this may have consequences for the reliability of the reported result.

Example:
"Differences to the guidelines have been observed, which may have influenced the reliability of the marked results in this analysis report."

Below this sentence, the observed points are stated regarding:
- preservation;
- sample packaging;
- preservation term;
- sampling date;
- sample quantity.

Remark:
A standard remark is particularly important to make clear that the deviating treatment (for instance failure to preserve in the field) may result in a difference in respect of the original concentration (in the sample immediately after it was taken). A mere mention of the sample being delivered in an unpreserved condition will not be sufficient for this.
The remarks in this analysis report form an integral part of that report.

When it is necessary to use a higher reporting limit for the outcome of an analysis, a remark to that effect should indicate such in the analysis report. The remark must state the cause for the use of a higher reporting limit.

Remark:
The cause of a higher reporting limit must be indicated as clear as possible. Most often, the cause is a low dry matter level combined with the presence of a lot of naturally organic material, which interferes with the compounds to be investigated. A mere mention that there is a matrix interference, without an explanation what this means will not be sufficient for this.

For suspect asbestos-containing materials, the following standard remark will be included in the analysis report:

"The sample has been characterized as suspected of containing asbestos. For this reason, grinding the sample is not possible, but it has been analyzed in a field-moist condition in two-fold. The outcome is an average of the two duplicate results."

This standard remark must specify the relevant parameters.

2.5.2 Summation of concentrations for group parameters

Additional regulations apply for the summation of the observed concentrations for group parameters, which have been phrased in the Dutch Soil Quality Regulations.
Including the summation in the analysis report is optional, but may be requested by a commissioning authority.
If stating the summation of the observed concentrations in an analysis report is opted for, the application of additional regulations from the Soil Quality Regulations will be required here. Appendix 3 provides an interpretation with calculation examples of the additional regulations.

2.6 Requirements regarding aftercare

2.6.1 Complaints

Complaints are settled in conformity with NEN-EN-ISO/IEC 17025 (§ 4.8).

2.6.2 Storage of sample remnants and sample removal

Soil and sediment samples:
Following the end of the investigation, the remaining part of the delivered samples will be stored for at least a standard period (four weeks counting from the day of acceptance by the laboratory) under storage conditions as stated for the analysis in question in NEN-ISO 18512 or SIKB protocol 3001 for soil samples, and in NEN-EN-ISO 5667-15 or SIKB protocol 3001 for sediment samples, unless other arrangements have been made with the commissioning authority in question.
If the (agreed upon) storage period has lapsed, the remaining soil and sediment samples will be removed.
Groundwater samples:
Remainders of groundwater samples will be stored under storage conditions as stated for the analysis in question in NEN-EN-ISO 5667-3 or SIKB protocol 3001, unless other arrangements have been made with the commissioning authority.

Storage of remainders of samples is not advisable or not possible for a number of parameters. Such is the case when the entire sample is used up during analysis, when the storage period is longer than the preservation term as stated in NEN-EN-ISO 5667-3 or SIKB protocol 3001 or when the layer of air above the surface of the sample affects the value or the levels of the relevant compounds in the sample. In these cases, the remainders of samples may be removed after the analysis has been conducted.

2.7 Requirements regarding archiving

An additional requirement for managing registrations of NEN-EN-ISO/IEC 17025, § 4.13 is that the laboratory must keep the raw data of the investigation results of AS SIKB 3000 in their custody for no less than two years. The analysis reports must be kept for no less than five years. Electronic archiving is permitted.

Chapter 3 Validation of a test and requirements regarding the quality system

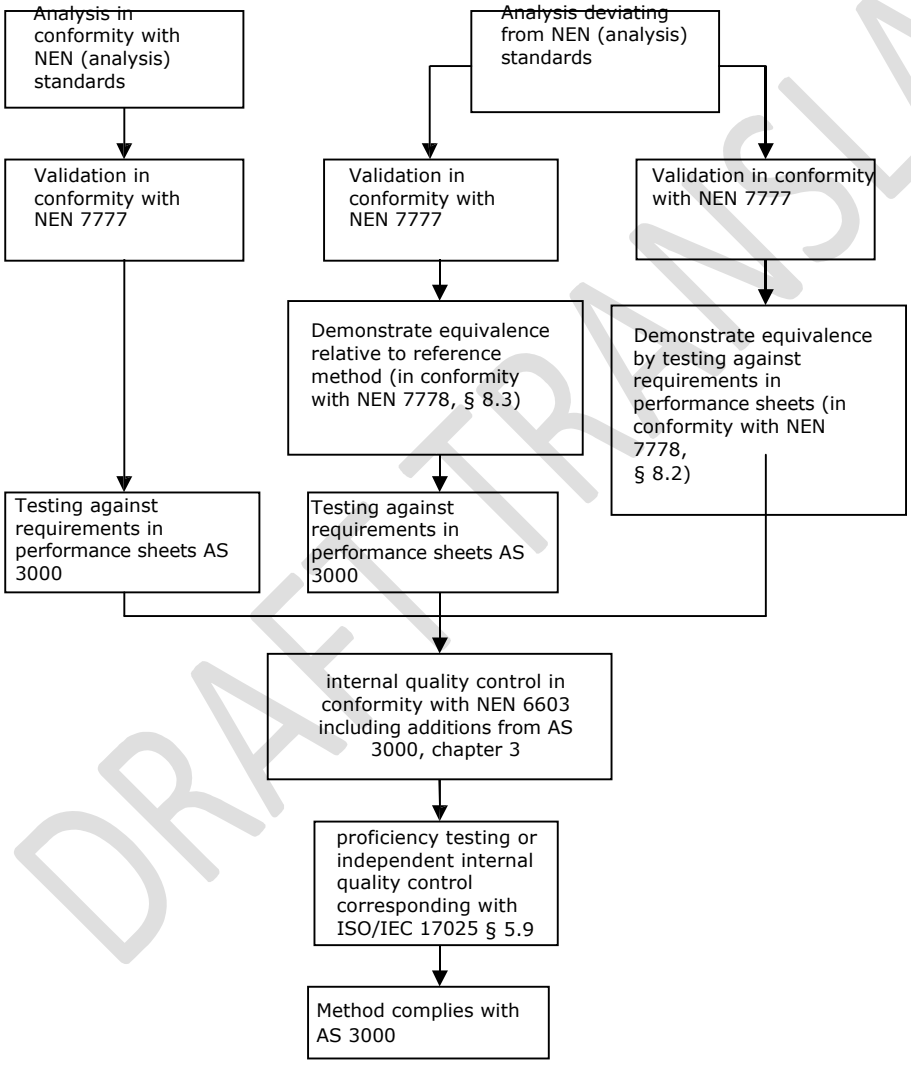
3.1 Introduction

This AS contains extra requirements set for validation investigation, equivalence investigation, internal and external quality control.

3.2 Validation investigation and equivalence investigation

Validation and equivalence investigations must be conducted in conformity with NEN 7777 and NEN 7778. The various possibilities are displayed in figure 3.1.

Figure 3.1 Various validation possibilities depending on the guiding principle of the analysis.



NEN 7778, 8.2: Equivalence relative to external requirements (based on estimated limiting values stipulated in the performance sheets of AS 3000).
NEN 7778, 8.3: Equivalence relative to a method of reference (referred to in the performance sheets of AS3000).
ISO/IEC 17025, 5.9: Quality assurance of the test and calibration results.

The validation of methods of analysis has been laid down in NEN 7777 and 7778. Depending on the analysis method to be validated, a validation programme must be drawn up in conformity with NEN 7777 and 7778.

3.2.1 Selecting the validation matrix

A validation investigation must be conducted in a matrix representative for the laboratory. The validation matrices to be used are set out in table 3.2. If practical samples are used for the validation, the basic material must be made homogenous, before sub-samples are compounded from the homogenous material. In order to determine a performance characteristic, the prescriptions for the analysis in question must be carried out in their entirety, including the applicable extraction or digestion. In principle, the sample pretreatment (minimization and/or homogenization for soil samples) is not included in the determination of the performance characteristics of a method of analysis. This is why for the validation of a method of analysis the preferred choice is the use of homogenous samples. In case of soil samples, the samples must be of a sufficiently small particle size to ensure the mass processed for the analysis is within the boundaries set in NEN-EN 16179.

Table 3.2 Validation matrices to determine performance characteristics.

Matrix	Validation matrices		
	Detection limit	**Other performance characteristics in the most difficult matrix**	**Exceptions**
Soil	• Most difficult matrix: soil with OS>10%[1)]	• Most difficult matrix: soil with OS>10%[1)]	• The determination of the performance characteristic recovery of cyanide (free) requires the use of clean sand and the most difficult matrix (two validation matrices) [4)]. • No most difficult matrix is defined for the parameter organic material since for validation a relevant level must be the guiding principle.
Groundwater	Synthetic groundwater matrix in conformity with NVN 6419 or a mixed sample of groundwater samples.	• For organic analyses: synthetic groundwater matrix in conformity with NVN 6419[2)]. For inorganic analyses: synthetic groundwater matrix in conformity with NVN 6419 or a mixed sample of groundwater samples[2)].	• In practice, the synthetic groundwater matrix in conformity with NVN 6419 has proven to be unsuitable for silver, nitrate and orthophosphate. Validation for these parameters is only possible with the aid of a mixed sample of groundwater.
Sediment	• Most difficult matrix: sediment with DS<40%, OS>10%[3)]	• Most difficult matrix: sediment with DS<40%, OS>10%[3)]	• The determination of the performance characteristic recovery of cyanide (free) requires the use of clean sand and the most difficult matrix (two validation matrices)[4)]. • No most difficult matrix is defined for the parameter organic material since for validation a relevant level must be the guiding principle. • For the parameter clay the dry matter level is not relevant, since the sample material is dried for analysis.

Remarks about the table:

[1)] Research of the data files of five environmental laboratories has shown that a 90-percentile value for organic material in soil samples amounts to 10%.

2) This synthetic groundwater matrix in conformity with NVN 6419 is intended for organic parameters; however, in practice it proved to be suitable for inorganic parameters as well. The exceptions to this are the parameters nitrate, orthophosphate and silver. For inorganic parameters a guiding principle of a mixed sample of groundwater samples may be used for validation. This also applies to organic parameters, where the determination of the detection limit is concerned. Subsequently, perform adduction on the desired concentration levels.

3) For sediment, pragmatic reasons led to the choice of an average organic matter level for the most difficult matrix. Research of the data files of some environmental laboratories showed that the 90-percentile value for organic material in sediment samples amounts to approximately 26 %. In practice, it is very difficult to find clean sediment samples with an organic matter level that complies with this 90-percentile value.
It is permitted to realize a dry matter level <40 % by adding water to the validation matrix. If after its addition, any of the water above is decanted during sample pretreatment, then the dry matter level of the remaining sample must stay at <40 %.

4) For cyanide (free) two validation matrices are applied for the performance characteristic recovery: clean sand and the most difficult matrix. In practice it has become apparent that recovery of the spiked cyanide (free) for the most difficult matrix is lower than the recovery of spiked cyanide (free) for clean sand.

Explanation for soil and sediment spiking:
During spiking the sample must be prepared in such a way that it is representative of a real sample. Test sample spiking is preferred here, with the sample being used in its entirety for the analysis. If the stability of the spiked material is sufficient, a waiting period of 48 hours is to be observed before the sample is processed (ageing). For the determination of volatile compounds (boiling point <300 °C at 1 atm) the stability is insufficient and following the spiking of the compounds, extraction may commence immediately. In the performance sheets an indication per parameter is given as to whether ageing must be applied for spiked samples. In some cases here, based on previous experience that this does not constitute a problem and for pragmatic practical reasons, it was opted to either or not perform ageing.

Explanation for groundwater spiking:
Spiking and ageing for groundwater samples must be carried out as described in NVN 6419.

To determine the bias of the soil and sediment in the framework of this AS, among other things, the SETOC 789/ISE 859-sample ("AS3000-control sample") may be used.

3.2.2 Requirements regarding performance characteristics

This paragraph provides a description of how to test the validation data.

Measurement range
The measurement range of the analysis must cover the entire domain of the detection limit (AG_{Rw}) up to and including 1.2 x the intervention value (indicated in the performance sheets). For sediment the measurement range most cover the domain of the target value up to 1.2 x the intervention value.
The determination of pH must be validated on two levels. Determining the electrical conductivity (for groundwater) must be validated across two decades. The determinations of dry matter, organic matter and clay must be validated in each of the sub-measurement ranges for which performance requirements have been indicated in the performance sheets.

Remarks:
- The validation on the highest decade for multi-component analyses may present practical problems. An example of this is the determination of volatile aromatic compounds and halogen compounds. Considering the large differences in intervention values for the various components, spiking on very different concentration levels would be necessary. Also, the level of the intervention value may be so high that spiking on that level would present practical problems. Also, available reference materials or standard solutions often confine the components in mutually equal concentrations and these are not always available in high concentrations.

When validation on the highest decade for multi-component analyses leads to practical problems that are difficult to solve, validation may be effected in a more restricted section of the measurement range, particularly in the section where dilution of sample extracts for the instrumental measurement is not required. This is subject to the condition that the sample pretreatment and extraction are performed in conformity with the reference method. The validation may then be carried out for concentrations in the sub-measurement range up to approximately 10 x the detection limit and in the sub-measurement range near the upper limit of the calibration function (in most cases this will comprise the linear domain of the measuring instrument). This must then be recorded in the validation report.

- For the determination of asbestos in soil and sediment samples, establishing the measurement range is not required.

Detection limit (AG_{Rw})

The detection limit of an analysis is determined in conformity with NEN 7777, under conditions of reproducibility. The reporting limit (RG_{Rw}) is based on the detection limit (AG_{Rw}), where the RG_{Rw} is the rounded up value of the AG_{Rw}.

Remark:
In general, the method blank of an analysis must comply with the requirement of that blank as referred to in the reference method in question. See the performance sheet in question in the protocols.

For the determination of asbestos in soil and sediment samples, establishing the detection limit is not required. For the determination of asbestos the determination limit will be set in conformity with NEN 5707.

Reporting limit (RG_{Rw})

As a supplement of NEN 7777 it applies that the requirements imposed for the reporting limit (RG_{Rw}) are related to the background values or target values in the performance sheets. These reporting limits (RG_{Rw}) pertain to analyses under reproducibility conditions where the calculation for the reporting limits is used as described under the detection limit.

Remark:
The limiting values for the reporting limits (RG_{geslim}) were established in mutual consultation with the entire laboratory sector.

Requirements:
- $RG_{Rw} \leq RG_{geslim}$ (see performance sheets);
- $RG_{Rw} \geq AG_{Rw}$.

For the determination of asbestos in soil and sediment samples the reporting limit equals the determination limit.

Intra-laboratory reproducibility coefficient of variation (vc_w)

The (intra-laboratory) reproducibility coefficient of variation of a method is determined in conformity with NEN 7777.

Requirement:
$vc_{Rw} \leq vc_{Rw,geslim}$ (see performance sheets)

For the determination of asbestos in soil and sediment samples, establishing the (intra-laboratory) reproducibility coefficient of variation is not required. Instead of this, the (pseudo) repeatability coefficient of variation will be established in conformity with NEN 5707 (see performance sheet for the determination of asbestos).

Recovery (*Tv*)
The recovery is an indication for the bias after spiking of the compound(s) in question to the matrix (standard addition).
Establishing the bias has preference over recovery, but is not always possible. In such cases where the bias cannot be established, the recovery will be determined on the basis of reproducibility.

Requirement:
$Tv_{geslim,min} \leq Tv \leq Tv_{geslim,max}$ (see performance sheets)

Remarks:
- See the explanation under 'Bias' for an additional interpretation of this requirement.
- In case of corrections in the method for an internal standard added prior to the extraction, the result of the validation sample must be tested against the requirement for bias.

Bias (*d*)
The bias of a method is established in conformity with NEN 7777, based on reproducibility. For the determination of the bias of soil and sediment in the framework of this AS, among other things, the SETOC 789/ISE 859-sample ("AS3000-control sample") or certified reference samples may be used.

Requirement:
$\|d\| \leq d_{geslim}$ (see performance sheets)

Explanation:
The requirements for recovery and bias apply for the average of the levels observed in the investigated sample material, not for individual measurement results. In this interpretation, the requirement of reproducibility ensures the fluctuation around the average remains within acceptable limits.

Model deviation
The model deviation (nonlinearity) of a method is established in conformity with NEN 7777.

3.2.3 Generic obligatory elements

Adding to the obligatory elements which are set out for each test in the performance sheets for soil, groundwater and sediment, the following obligatory elements will generically apply.

Method blank
In general, the method blank of a test must be lower than the reporting limit applied for that test.

The use of (procedural) internal standards
If one or more (procedural) internal standards are used in a reference method, these must be applied accordingly in the method used by the institution. The (procedural) internal standard(s) must be added at the moment - and applied in the manner - as described in the reference method.

Remarks:
- If multiple reference methods with different measurement techniques are mentioned for a test, then the conduct regarding the (procedural) internal standard(s) in the method used by the institution must be in conformity with the conduct in the reference method where within the same measurement technique is applied.
- Typical moments for the addition of an internal standard in a method are: prior to an extraction, a derivatization or an injection.

If no (procedural) internal standards are applied in a reference method, the institution may use internal standards to detect major errors in the test, if possible.

Conditions for use of internal standard(s):
- The internal standard must be added (to the sample or extract of the sample) at an early stage, so it is run through the entire test.
- The internal standard must be representative for the analyte(s) to be quantified, meaning labeled, deuterated compounds or a congener of the analytes to be quantified.
- An internal standard is applied when the test so allows.

A measurement value may only be corrected for a (procedural) internal standard if this is described in the reference method. A validation investigation conducted by the institution must determine which limiting value(s) the internal standard must conform to; this must be in reasonable proportion to the data as referred to in the performance sheet of the performance in question.

3.2.4 Reporting validation and equivalence investigation

The validation and equivalence investigation are concluded with a report. For the contents of the report, see NEN 7777 and NEN 7778.

3.3 Internal quality controls

The internal quality controls must comply with the requirements from NEN-EN-ISO/IEC 17025.
For a test, internal control is conducted for at least the components as indicated in the performance sheets. Independent internal quality control and proficiency tests are conducted for all components of a test. For actions to be carried out as a result of deviations from the various controls the cause, extent, solution and operational capacity must be recorded.

3.3.1 Internal quality control

The internal quality control of a test is conducted in conformity with NEN 6603.

Requirement (additional to the criteria stated in NEN 6603):
- $vc_{Rw} \leq vc_{Rw,geslim}$ (see performance sheets)

The internal quality control for the determination of asbestos in soil and sediment samples is conducted in a non-standard manner. See the relevant performance sheet for this. The results of this internal quality control must be registered.

Semi-annual performance investigation (additional to NEN 6603)
Every six months, for those analyses that a control chart has been effected for, a performance investigation must be conducted for all components of these analyses. The performance characteristics of the internal quality control sample are inspected here and compliance of the reproducibility with the requirements for the analysis as laid down in the performance sheet in question is assessed. For those components that are not involved in the internal quality control, the bias of recovery must be determined.
The average of the bias or recovery must comply with the requirements as stated in the performance sheets here. The requirement against which is tested must be regarded as an estimated limiting value as specified in NEN 7777.

If during the semi-annual performance investigation, it appears that the performance characteristics no longer comply with the requirements that were set, measurements must be taken to remedy the problem and to prevent incorrect results from being reported (see NEN-EN-ISO/IEC 17025 § 5.9.2).

Requirement:
- $Tv_{geslim,min} \leq Tv \leq Tv_{geslim,max}$ (see performance sheets)
  or:
  $|d| \leq d_{geslim}$ (see performance sheets)

3.3.2 Independent internal quality control

When not participating in proficiency tests, the laboratory must conduct a independent internal quality control. With the exception of the determination of asbestos, an analysis must be verified at least once a year, by way of a proficiency test or a independent internal quality control.

For the independent internal quality control, the following may be applied, in declining order of preference:
- reference material, representative for practical samples;
- material with consensus value, representative for practical samples;
- synthetic laboratory samples (for groundwater, in conformity with NVN 6419);
- (spiking) practical samples.

Requirements:
For reference material or samples with a consensus value[1)] (quantification in duplicate, under reproducibility conditions):
- $|d| \leq d_{geslim}$ (see performance sheets).

For synthetic laboratory samples (quantification in duplicate under reproducibility conditions):
- $Tv_{geslim,min} \leq Tv \leq Tv_{geslim,max}$ (see performance sheets)

For spiking practical samples (quantification in duplicate under reproducibility conditions, of sample with and sample without a spike):
- $Tv_{geslim,min} \leq Tv \leq Tv_{geslim,max}$ (see performance sheets)

For the requirement of the measurement in duplicate for all three options the following applies:
-
- |duplicate difference| $\leq 2s_{Rw,geslim}\sqrt{2}$
- Check in case of $2s_{Rw,geslim}\sqrt{2}$ < |duplicate difference| < $3s_{Rw,geslim}\sqrt{2}$ for one of the two measurements whether this is structural (→ action), or incidental (☐ most often no immediate action required) so as to be able to detect trends in sequential independent internal quality controls.
- In case of |duplicate difference| > $3s_{Rw,geslim}\sqrt{2}$: the cause must be discovered. If this cannot be found: repeat the independent internal quality control.

[1)] It is also possible to apply remnant samples from proficiency tests as samples with consensus values.

The duplicate-analyses require blind execution. This means that whoever carries out the analysis does not know the content of the samples, or does not know at all that these are control samples in the scope of a independent internal quality control.

Remark:

For the determination of asbestos, at least four times a year a independent internal quality control will take place. The recovery and (pseudo) repeatability (RSD) are tested here. The specifics of the independent internal quality control for the determination of asbestos in soil and groundwater are described in the relevant performance sheet.

Requirement (for the determination of asbestos in soil and sediment):

- $Tv_{geslim,min} < Tv < Tv_{geslim,max}$ (see performance sheet)
- $RSD < RSD_{geslim}$ (see performance sheet)

3.3.3 External quality control (proficiency tests)

The laboratory must regularly and successfully take part in proficiency tests for the accredited tests. When regular participation is not possible or if the frequency is less than once per year, additional independent internal quality control will be required. The own results must be checked against the results of the proficiency test. After reporting the proficiency test results where the test used is described, the Z-score is calculated per component. If the number of participants of the proficiency test is larger than or equals six, then the standard deviation, *s*, for testing the bias against the requirements in AS3000 is determined as follows:

$s_{Rw,geslim} > s_{ring} \rightarrow s = s_{Rw,geslim}$ (this may be the case when due to many measurements and robust statistics the s_{ring} has become very small and the spread as a result thereof is no longer realistic).

$s_{Rw,geslim} < s_{ring} \rightarrow s = s_{ring}$ (in this case, the s_{ring} for testing the bias is used).

If the number of participants is less than six, the proficiency test may only be used for testing the bias or recovery, if the real value of the sample material is known. If the real value is not known, then the proficiency test should be considered inappropriate and it may not be used for testing.

Only for those components with a concentration level higher than five times the determined *AG* will it be necessary to assess the Z-scores.

Requirement:
for both testing of the results relative to the group and relative to the bias:
- |Z-score| < 3;
- check in case of 2 < |Z-score| < 3 whether this is structural (→ action), or incidental (☐ most often no immediate action required) so as to be able to detect trends in sequential proficiency tests;
- no outliers;

Subsequent requirement:

- If no cause is observed during the investigation into the deviation(s), one of the following actions must be taken: conducting a independent internal quality control or, if the digest/extract generated is still present, to have the content determined by another laboratory with AS SIKB 3000 accreditation.

In case of a |Z-score| > 3 or repeated occurrences of |Z-score| > 2, further investigation must be conducted. A possible specification of this investigation is described in Appendix 5.

Remarks:
- When assessing results and determining as to whether timely action was undertaken by the laboratory, the reporting time and proficiency tests must be considered. It is possible that a proficiency test is reported when the results of the following proficiency tests have already been sent in. When testing results to detect trends it is possible that deviations can only be determined at the second following proficiency test, while the laboratory cannot be reproached for not undertaking timely action.

- For the determination of asbestos, proficiency tests from institutions accredited to that effect for groundwater and sediment are not available at the time of publication of this Accreditation Scheme. For the determination of asbestos it will be sufficient to conduct the proficiency test based on proficiency tests regarding the characterization of asbestos. The characterization must be carried out blind by anyone conducting the analysis. The requirement here is that the characterization is conducted properly by every conductor.

Chapter 4 Requirements regarding the accrediting institution

4.1 Accrediting institution

During the initial investigation or the test accreditation investigation it must be assessed whether the execution is in conformity with NEN-EN-ISO/IEC 17025, this AS SIKB 3000 and the protocols. The Dutch Accreditation Council must perform this assessment, or else an organization that the Dutch Accreditation Council has concluded a Multi-Lateral Agreement MLA (EA/IAF) or other equivalence agreement with.
The accrediting institution may issue accreditation certificates in conformity with this AS only after having concluded an agreement with the controller of this scheme, which is the Soil Management Accreditation Board. This agreement must explicitly refer to this AS.

The accrediting institution must observe the regulations in force for this accreditation. For the Dutch Accreditation Council, as far as accreditations for this AS SIKB 3000 are concerned, the Rules and Regulations for Accreditation will apply, supplemented by the provisions as set out in this chapter.

4.2 Accreditation investigation

During the initial, the interim (testing) and the reassessment investigation, also during the assessment of corrective measures, the accreditation institution will assess whether the execution is in conformity with NEN-EN-ISO/IEC 17025, this AS3000 and the corresponding protocols.

An accreditation certificate which has been issued has a validity of four years. In this period, this accreditation scheme AS SIKB 3000, including the corresponding protocols for which accreditation was issued must each year participate in the annual audit by the accrediting institution. As a rule, each separate test is to be assessed at least once every four years.

4.3 Communication between the accrediting institution and the Soil Management Accreditation Board

Communication in writing
The Soil Management Accreditation Board will inform the accrediting and accredited institutions of any alterations in the AS SIKB 3000 and its corresponding protocols as soon as possible.

Chapter 5 Literature

ISO 3534-1	2006	Statistics - Glossary and symbols - Part 1: General statistical terms and terms used in probability.
ISO 8466-2	2001	Water quality - Calibration and evaluation of analytical methods and performance characteristics - Part 2: Calibration strategy for non-linear second-order calibration functions.
ISO 5725-1 (incl. C1)	1994 1998	Accuracy (trueness and precision) of measurement methods and results. Part 1: General principles and definitions.
ISO 5725-2 (incl. C1)	1994 2002	Accuracy (trueness and precision) of measurement methods and results. Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.
NEN 5104 (incl. C1)	1989 1990	Geotechnics - Classification of unconsolidated soil samples.
NEN 5706	2003	Soil - Guidance on the description of sensory perceptions for environmental soil investigation.
NEN 5707 incl. C1	2003 2006	Soil - Investigation, sampling and analysis of asbestos in soil.
NEN 5717	2009	Soil – Sediment - Strategy for the preliminary study for exploratory and main survey.
NEN 5720	2009	Soil – Sediment – Strategy for exploratory survey – Investigation of the environmental quality of sediments
NEN 5725	2009	Soil quality - Strategy for the preliminary investigation prior to exploratory and main survey.
NTA 5727	2004	Soil - Sampling and analysis of asbestos in sediment and dredged sludge.
NEN 5740	2009	Soil quality – Strategy for exploratory survey - Investigation of the environmental quality of soil and soil lots.
NPR 5741	2009	Soil - Guidance for the selection and use of boring and sampling apparatus for soil, sediments and groundwater for the investigation of soil contamination.
NEN 5742	2001	Soil - Sampling of soil and sediments for the determination of metals, inorganic compounds, semi-volatile organic compounds and physico-chemical soil characteristics.
NEN 5743	1995	Soil - Sampling of soil and sediments for the determination of volatile compounds.

NEN5744 (incl. A1)	2011 2013	Soil quality - sampling of groundwater.
NEN 5766	2003	Soil - installations of wells for environmental research.
NVN 7312	1995	Leaching characteristics of solid earthy and stony building and waste materials - Sample pretreatment - Sample pretreatment for leaching tests and the analysis of inorganic components.
AS SIKB 2000	2009	Field work for environmental soil and sediment research.
Protocol 2001	2007	Installations of manual bores and wells, drawing bore plans, taking soil samples and levelling.
Protocol 2002	2007	Taking groundwater samples.
Protocol 2003	2008	Field work during environmental sediment investigation.
Protocol 2018	2007	Location-inspection and sampling of asbestos in soil.

Appendix 1 - Regulations for the use of the logo 'Quality Assurance of Soil Management SIKB'

The logo 'Quality Assurance for Soil Management SIKB', to be referred to hereinafter as "the logo", has been developed to provide clarity to all parties involved with regard to the quality assurance of activities in soil management, which include 'Laboratory Analyses for Environmental Soil Investigation'.

Management of the logo and supervision on its proper use for the Accreditation Scheme AS SIKB 3000 is in the hands of the Soil Management Accreditation Board, which operates under the Foundation Infrastructure of Soil Management (SIKB). The accreditation institutions authorized for that purpose will ensure proper use of the logo during their inspections at the accredited institutions.

Only laboratories that are accredited for the analyses as described in the Accreditation Scheme AS SIKB and have also fulfilled their annual contribution arising from this to SIKB, will be authorized to carry the logo. These laboratories acquire the right to carry the logo:

- On reports regarding laboratory analyses for environmental soil investigations, however only if the analyses for the investigations in question have been fully executed under the accreditation.
- On stationery, provided that the letter makes no mention of laboratory analyses for environmental soil investigations that were not or are not performed under the accreditation.
- In a general sense (including promotion, company presentations), if this documentation: also pertains to laboratory analyses for environmental soil investigations that were or are performed under the accreditation, and in no way whatsoever pertains to laboratory analyses for environmental soil research that were not or are not performed under the accreditation.

When a document is about multiple analyses of which one part was or is performed under the accreditation and one part was not or is not, then the logo may only be inserted in this document in such a way that it is perfectly clear which analyses have been conducted under the accreditation.

By "under the accreditation" is meant: "in conformity with the requirements as described in AS SIKB 3000, 'Laboratory Analyses for Environmental Soil Investigation', and the corresponding protocols". The version of these documents which is in force at the time of the performance of the analyses for the performing organization will apply here.

Upon its use, directly underneath the logo, which contains the text 'Quality Assurance for Soil Management SIKB', the relevant scope for the situation in question may be clearly displayed, for example by the notification 'AS SIKB 3000'.

Companies and institutions that violate the prescribed use of the logo may be sanctioned. A possible sanction may be the loss of the authorization to use the logo.

The logo is a registered trademark. Any misuse of the logo may be addressed under private law.

Appendix 2 – Definitions and interpretations of abbreviations

Dredged sludge
(Source: Dutch Soil Quality Decree)
Material released from the bottom or banks of a body of surface water consisting of mineral parts with a maximum grain size of 2 millimeters and organic matter in a ratio and with a structure as these are naturally found in the soil, also shells and pellets naturally present in the soil with a grain size of 2 to 63 millimeters.

Soil
(Source: NEN 5707)
Solid part of the earth with the liquid and gaseous elements and organisms present within.

Monitoring investigation
(Own definition from AS3000)
The investigation from the accrediting institution, which is performed after accreditation has been awarded, in order to determine that the accredited methods continuously comply with the requirements as set in the AS.

Estimated limiting value
(Source: NEN 7777)
Value of a performance characteristic, obtained from a former validation investigation or from estimations of a different kind, which aims to be a limiting value for the related performance characteristic.

Remark:
Estimations of performance characteristics obtained during investigations are directly (i.e. without statistical testing) compared with the relevant estimated limiting value. In case of limited exceeding (in case of an upper limit) or falling below (in case of a lower limit) the effectiveness of the measurement method will not immediately be at issue.

Soil
(Source: Dutch Soil Quality Decree)
Solid material consisting of mineral parts with a maximum grain size of 2 millimeters and organic matter in a ratio and with a structure as these are naturally found in the soil, also shells and pellets naturally present in the soil with a grain size of 2 to 63 millimeters.

Groundwater
(Source: Water Framework Directive)
All the water present below the soil surface in the zone of saturation and which is in direct contact with the soil or subsoil.

Remark*:*
For the definition of groundwater and its sampling, there is the issue concerning solid particles. Considering the fact that in the Target and Intervention Values a reference is made to dissolved components, in this Accreditation Scheme the choice was made to filter the groundwater for the quantification of inorganic components and to apply a sampling method for the quantification of organic components where the inclusion of solid particles is kept to a minimum as much as possible. These methods are described in NEN 5744.

Laboratory
(Own definition from AS3000)
The party responsible for the methods used to continuously comply with the requirements the accreditation is based on.

Commissioning authority
(Own definition from AS3000)
The company, the institution or the private person who orders the laboratory investigation to be carried out.

Performance requirements
(Own definition from AS3000)
Requirements specified in measurements or numbers, which are focused on certain (functional) properties of soil, groundwater or sediment, containing an achievable limiting value, which may unequivocally be calculated or measured.

Procedural requirements
(Own definition from AS3000)
Requirements specified in measurements or numbers, which are focused on the (identifiable) properties of the analytical methods applied in the laboratory and which contain an achievable limiting value, which may unequivocally be calculated or measured.

Reporting limit
(Own definition from AS3000)
Lowest concentration of the component in the sample which is reported to the commissioning authority.
The reporting limit to be used for each compound and/or element is determined on the basis of the detection limit, AG_{Rw}.

Admission test
(Own definition from AS3000)
The test of an accrediting institution to determine that all requirements as set out in the AS have been complied with.

Obligatory element
(Own definition from AS3000)
Actions and circumstances forming part of the method, which are the most critical and exert the most influence on the final result. Obligatory elements are always in conformity with the reference method and are obligatory for the equivalent methods. In the event that more than one reference method is given, for the specification of the obligatory element, the specifications from one of the reference methods must be followed. For an obligatory element, on the specified elements (for example the extraction) the (measurement) principle may not be deviated from and the elements must be carried out in conformity with the specified performance. This will not apply for other elements, not included as 'Obligatory Elements' (for example the measurement).

Test
(Own definition AS3000)
The quantification of the content of the compounds and/or elements referred to in one performance sheet.

Sediment
(There is no formal definition available for sediment. In the framework of this Accreditation scheme, the definition as stipulated in the Dutch Water Act is used)
Bottom or bank of a body of surface water.

Z-score
(Own definition AS3000)
The Z-score is calculated by dividing the difference between the value observed and the group average or reference value by the standard deviation belonging to the group average, reference value or duplicate sample. This measurement is used for:
- the assessment of the performance of the own laboratory in relation to the group average or reference value in third-line controls;
- the assessment of duplicate analyses of the independent internal quality controls.

Explanation of abbreviations: General abbreviations

AC Bodembeheer	Soil Management Accreditation Board.
AS	Accreditation Scheme. Throughout this document, where reference is made of AS, this AS is meant.
CCvD Bodembeheer	Central Board of Experts Soil Management.
SIKB	Foundation Infrastructure for the Quality Assurance of Soil Management.
NEN	Dutch Standard, issued by the Netherlands Standardization institute.
NEN-EN	European standard accepted as Dutch, issued in the Netherlands by the Netherlands Standardization institute.
NEN-EN-ISO	International standard accepted as Dutch and European standard, issued in the Netherlands by the Netherlands Standardization institute.
NTA	Dutch Technical Agreement, issued by the Netherlands Standardization institute.
NPR	Netherlands Code of Practice, issued by the Netherlands Standardization institute.
NVN	Netherlands Pre-Standard, issued by the Netherlands Standardization institute.
ISO	International standard, issued by the International Organization for Standardization.
AW	Background Value (soil and sediment).
SW	Target Value (groundwater).
IW	Intervention Value.

Explanation of abbreviations: Abbreviations of performance characteristics

c	concentration;
$\delta_{c,model,abslim}$	absolute limiting value for the model deviation;
d	bias;
d_{geslim}	estimated limiting value of the bias;
n	quantity;
AG	detection limit;
AG_{Rw}	detection limit based on intra-laboratory reproducibility;
BG	determination limit;
G_p	Result of a Grubbs' outlier test;
RG	reporting limit;
RG_{geslim}	estimated limiting value of the reporting limit;
RG_{Rw}	reporting limit based on intra-laboratory reproducibility;
RSD	*(pseudo)repeatability standard deviation (see NEN 5707, appendix D.1.2);*
s	standard deviation of the measurement quantity;
s_L	between-laboratory standard deviation (pure inter-laboratory standard deviation);
s_R	inter-laboratory reproducibility standard deviation;
s_{ring}	the standard deviation of a proficiency test in the assigned value;
s_{Rw}	(intra-laboratory)reproducibility standard deviation;
$s_{W,geslim}$	estimated limiting value of the (intra-laboratory) reproducibility standard deviation;
Tv	recovery;
Tv_{geslim}	estimated limiting value of the recovery;
vc_{Rw}	(intra-laboratory) reproducibility coefficient of variation;
$vc_{Rw,geslim}$	estimated limiting value of the (intra-laboratory) reproducibility coefficient of variation;
x_i	measurement result of a measurement;
$\bar{x}$	average measurement result.

Appendix 3 - Summation of concentrations for group parameters (additional regulations)

(This appendix provides an interpretation of the Soil Quality Regulations and is normative if summation of concentrations is reported)

There are additional regulations that apply regarding the summation of detected concentrations, which are laid down in the Dutch Soil Protection Act. The compounds that are included in a summation for soil, groundwater and sediment are specified in Appendix N of the Soil Quality Regulations. If summation is applied for soil, groundwater and sediment according to the additional regulations in the analysis report, then the summation must be performed as set out herein below.

If (part of) the detected concentrations are below the reporting limit used by the laboratory (RG_W or RG_r), then a total value will be given for these components in the analysis report, which equals the sum of all concentrations, where for the concentrations '< reporting limit' a value equaling 0.7 * this reporting limit will be used. In the analysis report a remark will be recorded referring to this total value, stating the application of the factor 0.7, in conformity with 'AS3000, Appendix 3'.

Increased reporting limits (additional regulations)
If one or more of the components require an increased reporting limit, for example as a result of a matrix failure during the measurement or a low percentage of dry matter, then a concentration of 0.7 x the increased reporting limit will be applied for the summation of these components. The cause of the increased reporting limit will be stated in the analysis report by way of a remark.

An example of a sum parameter is the quantification of the PAH-10-VROM level.
Table B3-1 contains further specification of examples for the calculation of the total value to be reported.

Table B3-1. Examples for the summation of concentrations for sum parameters based on the quantification of PAH-10-VROM in a soil sample with an organic matter percentage of 10 %.
The RG(RG_W or RG_r) for PAH-components (organic matter = 10 %) is 0.15 mg/kg DM for each PAH-component.

Concentrations of the ten PAH-components	Total value with factor 0.7 [a)]
10 x '<0.15'	1.05
3 components 0.30 7 components '<0.15'	1.64
1 component 0.42 9 components '<0.15'	1.37
2 components 0.43 3 components 0.51 5 components '<0.15'	2.92
10 components '<0.8' [b)]	5.6 [c)]
6 components '<0.15' 4 components '<0.5' [b)]	2.03 [c)]

Concentrations of the ten PAH-components	**Total value with factor 0.7** [a)]
3 components 0.42 6 components '<0.15' 1 component '<0.6' [b)]	2.31 [c)]
3 components 24.0 4 components 31.0 1 component 56.0 2 components '<10' [b)]	266 [c)]

a): In all cases, this total value will be accompanied by a remark regarding application of the factor 0.7.
b): Increased reporting limit as a result of any random cause.
c): This total value will be accompanied by a remark stating the cause of the increased reporting limit.

Appendix 4 – Pretreatment of soil samples with over 20% (m/m) foreign material content

(This appendix is normative)

For soil samples with over 20% (m/m) foreign material content, a pretreatment which deviates from NEN-EN 16179 will apply.
The minimum sample size that the pretreatment must be performed with is determined by the grain size in the soil sample, based on table B4-1.

Table B4-1. Sample pretreatment. Relation sample size and grain size (NVN 7312)

grain size D_{95} (mm)	minimum sample size (g)
< 0.125	5*
0.25	50
0.50	100
0.71	250
1	500
2	1,000
4	2,000
8	3,000
16	5,000
32	10,000
64	20,000

* deviating from NVN 7312

Remark:

1. Foreign material content are materials not included in the definition for soil (see Appendix 2). Examples of foreign materials are branches, glass, metal parts, textile, rubber, pieces of rubble and stones not being pebbles or shells < 63 mm.
2. The percentage of foreign material is determined visually.
3. The issues surrounding representativeness of the sampling method for determining volatile substances was investigated in ANVM project 108/109. As a result of occurring losses, the representativeness is considered insufficient for samples with a grain size over 4 mm. In those cases where there is a low concentration the sample pretreatment in itself may result in little or no detection of volatile substances. This is why for reasons of implementation, usually no volatile substances are determined in coarse-grained samples. However, this does not mean the analysis cannot be useful, for example when high concentrations are expected as a result of pollution. If volatile substances are detected in such case, it may be assumed that the actual concentrations will be higher.
   Direct sub-sampling to test sample for the quantification of volatile substances in shaped building materials is not possible without prior sample pretreatment (grain-size reduction) has been performed. Quantification of volatile substances is only advised when these substances are expected to be present in the sample in relatively high concentrations. The results must always be considered to be indicative.
4. For practical reasons, consistency with NEN-EN 16179, it was opted here to pre-reduce the samples to a grain-size of D_{95} <2 mm instead of a grain-size of D_{95} <4 mm.

If the grain-size D_{95} is larger than 2 mm, the particles that cannot be ground will be removed and the entire remainder of the sample will be pulverized under cryogenic conditions to <2 mm. Before pulverization, add a cooling agent to the sample, for instance liquid nitrogen or solid carbon dioxide. The temperature of the sample after pulverization must be below 5 °C.

Remark:

Particles that cannot be ground are particles that cannot be reduced applying the grinding techniques commonly used for soil. Examples are: metal parts (bolts, screws, wire, etc.), branches, textile parts, synthetic materials.

After pulverization, the tie in with NEN-EN 16179 is possible. Proceed the pretreatment with § 9.3 and § 9.4, or else § 9.5 from this standard.

In the analysis report there must be a reference to this Appendix 4 regarding the working method of the pretreatment.

Appendix 5 - Evaluation scheme for proficiency testing

(This appendix is informative)

The below working method for testing proficiency results is optional, however, it complies with the requirements of this AS.

After reporting the results of the proficiency test, the z-score for each component is calculated.
A result of a component will be significantly deviating if one of the following situations presents itself:

- The absolute value of the z-score for one or more detections is larger than 3 ($|z| > 3$).
- A proficiency test consisting of one or two samples from the same matrix, where the absolute value of the z-scores of one specific component:
  - for two samples within the last proficiency test is larger than 2 and at the same side of the average;
  - for more than two samples in the last *two proficiency* tests is larger than 2 and at the same side of the average;
- A proficiency test consisting of three samples of the same matrix, where the absolute value of the z-scores of one specific component:
  - for more than two samples in the last proficiency test is larger than 2 and at the same side of the average;
- for more than two samples in the last *two proficiency* tests is larger than 2 and at the same side of the average.
- A proficiency test consisting of four samples from the same matrix, where the absolute value of the z-scores of one specific component:
  - for more than two samples in the last proficiency test is larger than 2 and at the same side of the average;
  - for more than three samples in the last *two proficiency* tests is 2 and at the same side of the average.

In table B5.1, the marginal cases have been filled in for clarification purposes. At first, the last proficiency test will be tested. The maximum number of significant deviations here must be lower than what has been stated in the third column.
If the outcome of the assessment is sufficient and also the assessment of the next to last proficiency test sufficed, then subsequently, the last two proficiency tests will be tested together. The maximum number of significant deviations here must yet again be lower than what has been stated in the third column of table B5.1.

The sequence in the table is randomly chosen and is of no importance for testing. For the assessment, only the significant deviations are of importance and of which type (M or S) these are.

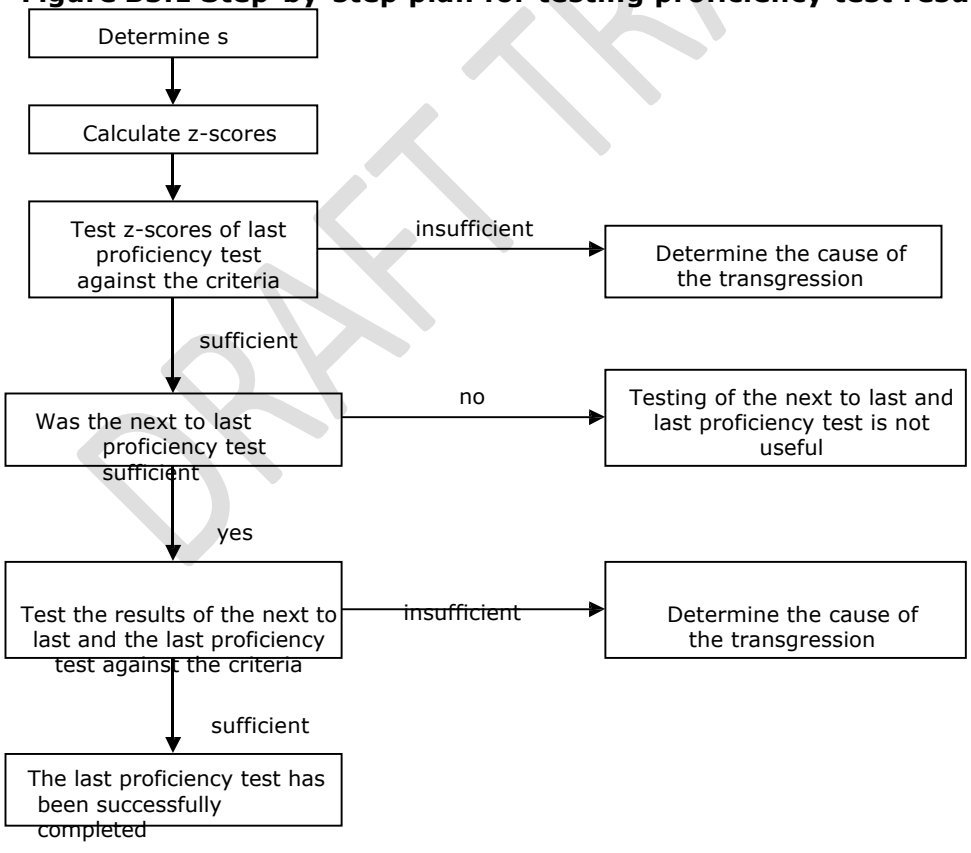
Table B5.1 Criteria for the assessment of proficiency tests[1]

Number of samples to assess	Proficiency test(s)	
	Sufficient[2]	**Insufficient[3]**
1	M	S
2	G,M	M,M
		G,S
3	G,M,M	M,M,M
		G,G,S
4	G,G,M,M	G,M,M,M
		G,G,G,S
5	G,G,G,M,M	G,G,M,M,M
		G,G,G,G,S
6	G,G,G,G,M,M	G,G,G,M,M,M
		G,G,G,G,G,S
7	G,G,G,G,M,M,M	G,G,G,M,M,M,M
		G,G,G,G,G,G,S
8	G,G,G,G,G,M,M,M	G,G,G,G,M,M,M,M
		G,G,G,G,G,G,G,S

[1]) **G** = |z| < 2*standard deviation (level is in the 95% reliability range), **M** = 2*standard deviation < |z| < 3*standard deviation (level is outside the 95% reliability range) and **S** = |z| > 3*standard deviation (a transgression, determine cause);
[2]) z-scores per parameter permitted within one proficiency test;
[3]) z-scores per parameter for which the cause of the deviation must be determined.

Figure B5.1 provides the plan of the testing procedure for clarification purposes.

Figure B5.1 Step-by-step plan for testing proficiency test results



Example:

In the proficiency test SETOC 2006.1, consisting of four samples, the following scores were reached for cadmium: G, G, M, M. According to the table, this is permitted. Subsequently, the results for cadmium of the preceding proficiency test will be involved in the evaluation.
In the proficiency test SETOC 2005.4 it appeared that the cadmium level of one of the samples was undetectable. For the three other samples, the following scores were reached: G, G, M.
The combined results of SETOC 2005.4 and 2006.1 lead to: G, G, G, G, M, M, M.
The number of samples to assess is 7 and the combined score that was reached is permitted.

Supposing the following score was reached in SETOC 2005.4: G, M, M. This score is permitted. An assessment of the combined SETOC 2005.4 and 2006.1 will now lead to four average scores, which is NOT permitted. This requires action to be taken.