

**Accreditation Scheme
Laboratory Analyses for
Environmental Soil Investigation**

AS SIKB 3000



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Preface

This Accreditation Scheme has been determined by the Accreditation Council for Soil Management, in which interested parties in the sector of soil and building materials are represented. Whenever in this Accreditation Scheme "Council of Experts" is mentioned, the above mentioned Council is referred to.

The drawing up of this Accreditation Scheme has been supervised by a committee that acting in the interest of the parties involved. In collaboration with the production bureau of SIKB and the (Dutch) Council for Accreditation (RvA), this supervising committee has tested the outcome.

The Council will also supervise the execution of the accreditation and will alter this accreditation scheme if need be.

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Helpdesk / Users Manual

Any questions regarding content and application can be directed at your accrediting institution or at the SIKB. Regarding points of difference, see the Chapter 'Complaints' in this document.

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1 Introduction

1.1 Objective

The requirements included in this Accreditation Scheme are used by the accrediting institution for assessing laboratories at the application for or the retaining of an accreditation for this AS. Analytical activities are involved that are performed during an orientating investigating, an informative investigation, a further investigation, a reorganising investigation and other comparable investigations of soil. The conception of this AS is modular, so that at a later stage other matrices can also be included without the need for structural changes.

The certificate of conformity issued by the accrediting institution is designated as an accreditation for AS SIKB 3000 in combination with the competence assessment of the laboratory to perform specific tasks. The contents of AS SIKB 3000 are not inconsistent with the criteria of NEN-EN-ISO/IEC 17025.

1.2 Application

This AS is linked to various other (normative) documents. AS SIKB 3000 is linked to BRL SIKB 2000 (Fieldwork for environmental soil investigation), for which the samples must be transported and stored according to the directives laid down in VKB Protocol 2001 (Placing handmatic Plaatsen van handboringen en peilbuizen, maken van boorbeschrijvingen, taking soil samples and levels).

The procedure described in this AS refers to the analysis of soil samples. The following stages in the process can be distinguished:

Stages in the process that do exert an influence, but fall outside this AS (the quality is assured through BRL SIKB 2000 and the preservation during storage and transportation in VKB Protocol 2001):

- sampling
- the transportation and storage of samples for the transfer to the laboratory

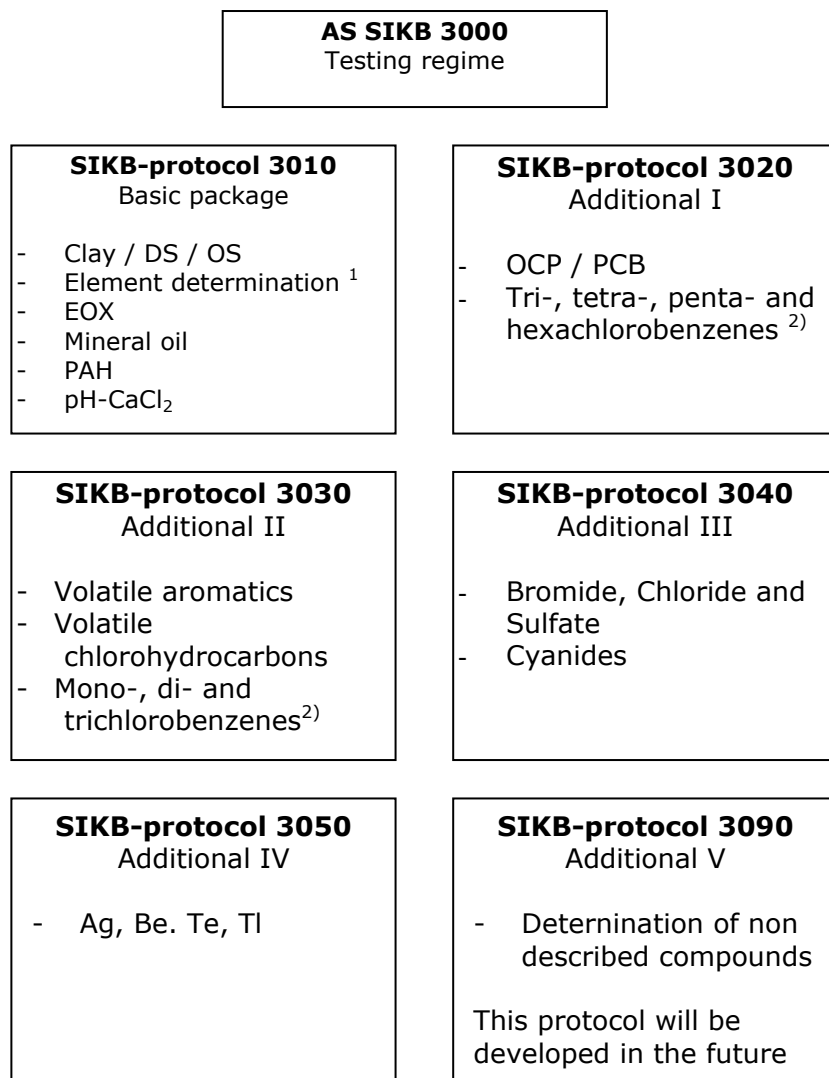
Stages in the process that are part of this AS:

- the transfer to the laboratory
- transportation to the laboratory under the responsibility of the laboratory
- preservation check
- term of delivery
- sample pretreatment
- preliminary operations prior to the measurements
- the analysis
- quality management
- the analysis certificate
- validation investigation
- the after-care
- requirements regarding the accrediting institution

Protocol subdivision AS SIKB 3000

Within this AS, laboratories can be accredited for a number of protocols. In *figure 1.1* the subdivision, which applies to the analyses within the relevant protocols are schematically rendered. The laboratory has to be accredited first for the basic protocol (SIKB protocol 3010), thereafter, one or more of the additional protocols can be added to this. The requirements of the procedure are described in AS SIKB 3000 and SIKB Protocol 3001. Besides this, the performance requirements have been laid down in the relevant (analysis) protocols. It is the rule in this respect that the AS and all protocols in question, for which a company has been or wants to be accredited, fall under the testing regime. The build-up and the relation of AS SIKB 3000 to the attached protocols have been rendered in *figure 1.1*.

Figure 1.1 Subdivision of analyses within the protocols suitable for accreditation



¹) The following metals come under protocol 3010: As, Cd, Cr, Cu, (non-volatile) Hg, Ni, Pb, Zn, Ba, Co, Mo, Sn, Sb, Se and V.

²⁾ Trichlorobenzenes may be determined according to Protocol 3030 (volatile compounds) or according to Protocol 3020 (OCP and PCB), if the applied method meets the requirements laid down in the performance sheet.

1.3 Requirements regarding Execution

The requirements drawn up for the execution of the laboratory work were laid down in the performance sheets that form part of the related protocols. For all parameters a pretreatment in conformity with Draft-NEN 5709 is an obligatory action. For the various parameters, the preservation method and the requirements regarding the keeping qualities of the samples have been laid down in SIKB Protocol 3001.

The requirements which the execution of the work must comply with, are listed in the performance sheet. For this reason, the documents mentioned in the following overview form part of this AS. An institution may be accredited in conformity with AS SIKB 3000 for each protocol separately. The accreditation includes all compounds or elements mentioned in the performance sheet in the defined protocols (therefore it is not possible to be for instance accredited for one compound or one performance sheet, when more compounds and/or performance sheets are mentioned in the protocol). The accreditation in accordance with this AS for the protocol in question is mentioned in the appendix to the accreditation certificate.

In order to be accredited for AS SIKB 3000 a laboratory must:

- have command of at least those performances that are included in protocol 3010;
- pro protocol, one of the performances may (by contract) be put out to another institution accredited for it in conformity with AS SIKB 3000;
- apart from this, at least one performance pro protocol must be performed by the company itself;

Each laboratory which is accredited for this AS, should command the methods as described in SIKB Protocol 3010. This has to be demonstrated by means of a validation investigation, the performance characteristics of which must meet the requirements drawn up in the performance sheets in SIKB Protocol 3010. In respect of the remaining protocols (SIKB Protocol 3020, SIKB Protocol 3030, SIKB Protocol 3040, SIKB Protocol 3050 and SIKB Protocol 3090), a laboratory may extend the accreditation pro protocol.

Basic protocols for accreditation

Protocol 3001: Method of preservation and preservation period of environmental samples

Protocol 3010: Basic package, the determination of clay, DS, OS, As, Ba, Cd, Co, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Sn, Se, V, Zn, EOX, mineral oil, PAH and pH-CaCl₂.

Additional protocols

Protocol 3020: Additional I, the determination of OCP, PCB and chlorobenzenes (tri- (optional), tetra-, penta-, and hexachlorobenzenes).

Protocol 3030: Additional II, the determination of benzene, toluene, ethylbenzene, (o/m/p) xylene, volatile halogenated hydrocarbons and chlorobenzenes (mono-, di- and trichlorobenzenes (optional)).

Protocol 3040: Additional III, the determination of bromide, chloride, sulphate and cyanide.

Protocol 3050: Additional IV, the determination of Ag, Be, Te and Tl

Protocol 3090: Additional V, the determination of chemicals in soil (to be specified hereafter) that have not been described in other protocols

1.4 Normative References

The following standards contain regulations that, if referred to, are also regulations of this AS. At the date of publication of the AS at hand, the above mentioned versions were in force. However, all normative documents can be revised; parties that enter into agreements on the basis of this AS should check the possibility to apply the most recent version of the normative documents mentioned below.

NEN-EN-ISO/IEC 17025	2000	General requirements regarding the competence of testing- and calibration laboratories
Protocol 3001	2003	Method of preservation and preservation period of environmental samples
Draft-NEN 5709	2004	Soil- Sample preparation for the determination of organic and inorganic parameters in soil
NEN 5740	1999	Soil - Investigation strategy for exploratory surveys; Investigation of the environmental quality of soil and soil lots
NEN 7777	2003	Environment - Performance characteristics of measurement methods
NEN 7778	2003	Environment - Equivalency of measurement methods
NPR 6603	1988	Water and sludge - Guideline for internal quality control by the use of control charts with chemical analyses

In principle, the most recent version applies. In case any alterations have been made in afore mentioned normative documents and the (analysis) standards mentioned in the performance sheet to a new Dutch standard, the old normative document may be applied during a transition period of 6 months. After this transition period the new normative document becomes operative and the old one will elapse. If a standard is withdrawn by the issueing of an international standard, the Dutch standard will remain in force.

1.5 Requirements regarding Determination methods

In this accreditation scheme requirements and determination methods are laid down. In the subsequent paragraphs these will be elaborated further. NEN-EN-ISO/IEC 17025 is authoritative regarding the requirements of the system within the laboratory. AS SIKB 3000 is authoritative regarding the technical details with respect to the underlying (normative) documents.

1.6 Acceptance of Investigation Reports delivered by the Laboratory

The laboratory may be accredited for this AS only, when it also possesses a valid accreditation according to NEN-EN-ISO/IEC 17025. This accreditation must have been provided either by the (Dutch) Council for Accreditation or by an organisation, with which the Council for Accreditation has entered into a Multi Lateral Agreement MLA (EA/IAF).

1.7 Rules and Regulations regarding the Application of Accreditation Mark AS SIKB 3000

Quality mark 'Quality assurance for soil management SIKB'

The quality mark 'Quality assurance for soil management SIKB' has been developed in order to provide clarity for all parties concerned about the quality assurance of activities regarding soil management. The laboratory analyses for environmental soil investigation described in this accreditation scheme fall within the scope of this quality mark. This means that laboratories that have been accredited for the analyses described in this accreditation scheme may acquire the right to apply the quality mark. The rules and regulations in connection with the application of this quality mark are included in attachment 2.

Accreditation marks of the (Dutch) Council for Accreditation

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. The rules and regulations connected with the application of the accreditation mark of the (Dutch) Council for Accreditation are included in the document 'Rules and regulations for the Use of Accreditation marks' (RvA-R3). This document can be downloaded from the website of the (Dutch) Council for Accreditation (RvA), www.rva.nl.

1.8 Definitions

1.8.1 General definitions

Below, please find a list of conceptions and their definitions used in this accreditation scheme:

Remark:

The most relevant definitions have been already included in NEN 7777 and 7778. Only the most common definitions are listed here.

Laboratory

The party that is responsible that the methods applied continuously meet the requirements, on which the accreditation is based.

Client

The company, institution or private person that commissions the laboratory investigation.

Audit

The investigation of an accrediting institution performed after that institution has been granted an accreditation to determine whether the accredited methods continuously meet the requirements drawn up in the AS.

Soil

Solid part of the earth with the liquid and gaseous elements and organisms found in it. (NEN 5707).

Requirements of the Performance

Specific requirements expressed in measures and numbers that are especially applicable to certain (functional) characteristics of soil and contain a limiting value aimed at, that can unambiguously be calculated or measured.

Requirements of the Procedure

Specific requirements expressed in measures or numbers that are especially applicable to the (identifiable) characteristics of the analytical methods used in the laboratory and contain a limiting value aimed at, that can unambiguously be calculated or measured.

Investigation for Admission

The investigation of an accrediting institution in order to determine whether all the requirements laid down in the AS are met.

1.8.2 Definitions: equivalence of measurement methods

Points of special interest

Performances or circumstances that are critical for the reference method and that can influence the final result. It is conceivable, however, that equivalent methods exist, for which these performances and circumstances do not play a role. The points of special interest do therefore not form an obligatory part of an equivalent method.

Equivalent (measurement) method

Measurement method that meets the requirements drawn up from the point of view of effectiveness (fitness of purpose) for the intended use of the reference method.

Estimated limiting value

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

Remark:

1. Assessments of performance characteristics obtained by investigation are directly (i.e. without statistical testing) compared to the estimated limiting value.
 - In case of a slight exceeding (in case of an upper limit) or understepping (in case of a lower limit) of the value, the fitness of purpose of the measurement method is not directly at issue.

Reference (measurement) method

A measurement method, the suitability of which from the point of view of effectiveness (fitness of purpose) has been accepted as suitable for the intended application.

Supporting performance

Measurement method forming part of another measurement method, which for this reason makes less demands to the quality assurance.

Application

The population samples in which valid measurements can be made, either with the reference method or an equivalent method.

Obligatory parts of the investigation

Obligatory parts are those performances and circumstances that are most decisive for and have the greatest influence on the final result. These parts are also obligatory for the reference method and for equivalent methods.

1.8.3 Definitions: Performance Characteristics for Measurement Methods

Detection limit

Lowest concentration of the component in the sample, the presence of which can be determined with a reliability of 99%.

(Intralaboratory) repeatability

Degree of conformity between the results of successive measurements of the same measurand, conducted under the same circumstances [NPR 2814].

Reporting limit

Lowest concentration of the component in the sample reported to the client. The reporting limit applied may never be lower than the detection limit determined for that analysis.

(Intralaboratory) reproducibility

Degree of conformity between the measurement results of the same measurand, obtained under different measurement conditions. [NPR 2814].

Retrieval

Fraction of the measurement component retrieved during analysis, after the addition of a known amount of the measurement component to the sample under defined circumstances.

1.8.4 General abbreviations

AS Accreditation Scheme. Whenever in this document AS is mentioned, this AS is referred to;

CCvD Centraal College van Deskundigen (Dutch) Central Council of Experts)

SIKB Stichting Infrastructuur Kwaliteitsborging Bodemonderzoek (Dutch) Foundation Infrastructure for Quality Assurance of Soil Investigation

NEN Dutch Standard, published by the Dutch Institute for Standardization

NPR Dutch Directions for Practice, published by the Dutch Institute for Standardization

NVN Dutch Preliminary Standard, published by the Dutch Institute for Standardization

ISO International Standard, published by the "International Organisation for Standardization"

1.8.5 Abbreviations of Performance Characteristics

AG Detection Limit (DL)

AG_{eis} Required detection limit

AG_r Detection limit under repeatability conditions

n Number of repetitions

S_R Interlaboratory reproducibility standard deviation

T_V Terugvinding (Retrieval)

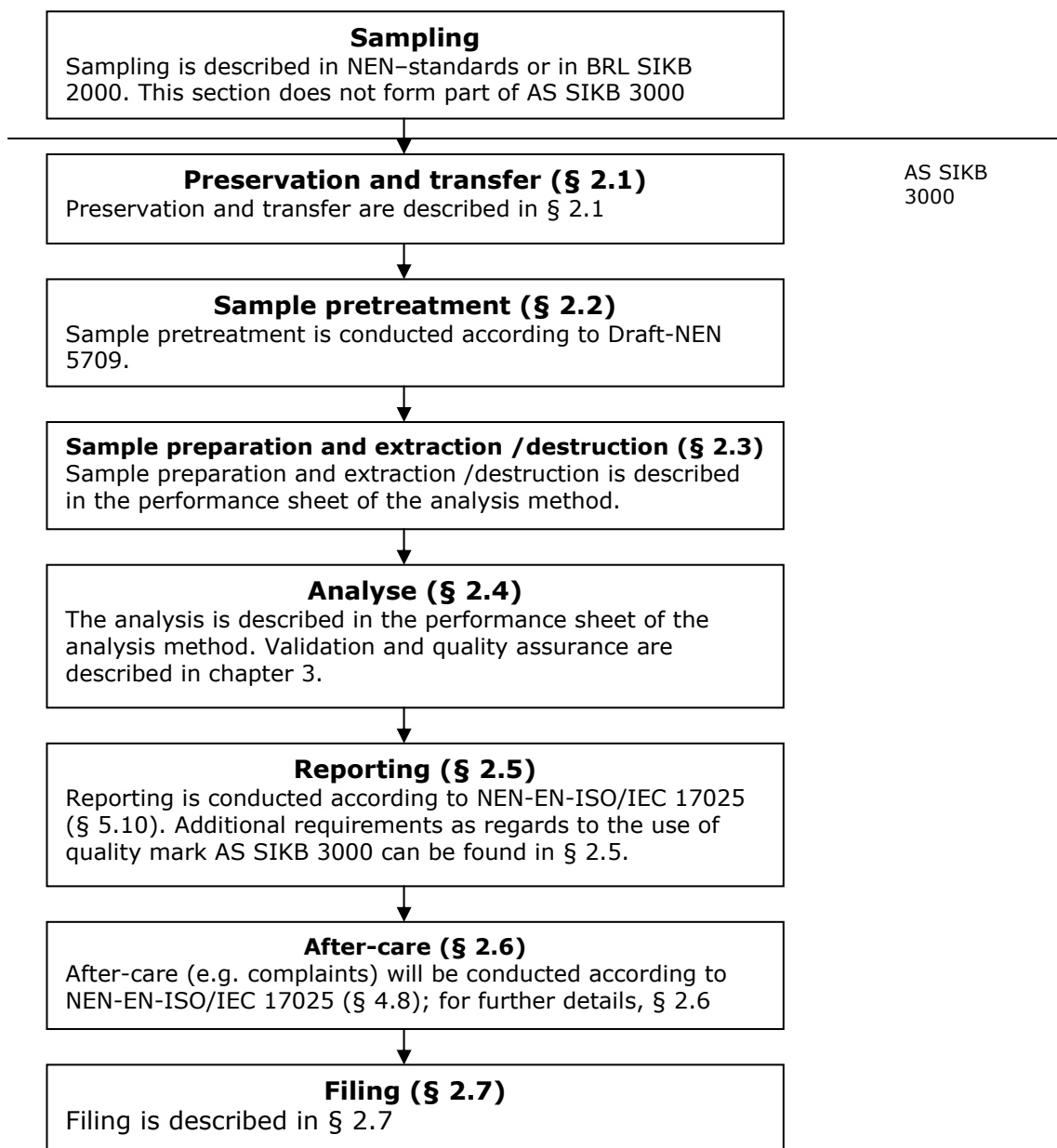
S_W (Intralaboratory) reproducibility standard deviation

VC_w (Intralaboratory) reproducibility variation coefficient

2 Requirements of the Procedure

Figure 2.1 presents an outline of the stages of the procedure a sample passes through from sampling until and including the filing of the results. Links have been included between the different stages and the structure of this AS by means of a reference to the relevant paragraph in which the additional information can be found. In a number of cases, in the paragraphs referred to additional requirements have been formulated in respect of NEN-EN-ISO/IEC 17025.

Figur 2.1 Scheme of the successive procedures according to AS SIKB 3000



For further information, please refer to the explanatory Appendix to Chapter 2 (Appendix 1). The information in this appendix is for illustrative purposes only and is therefore not normative.

2.1 Preservation and Transfer

In general, the responsibility of the laboratory takes effect after the laboratory has accepted the sample (see § 2.1.3.1). For the transfer of samples, the requirements of the procedure in respect of sample preservation, laid down in SIKB protocol 2010 or other (normative) documents applicable to the client, must be met by the client. The client is responsible for a correct preservation. The client must record on the sample transfer forms, if the samples have been preserved. The executing laboratory is not obliged to determine in retrospect, whether the preservation has actually been carried out.

The requirements made in this AS to the preservation of samples, only refer to the stages of the procedure being performed after the transfer of the samples. The transfer of the samples can take place in the laboratory, but also in an (interim) storage at the laboratory or during a courier service of the laboratory in question.

2.1.1 Preservation

The laboratory investigation aims at determining the contents during sampling. Care has to be taken that the concentrations in the sample remain constant. For this reason, after the sampling a preservation technique is being applied, which aims at confining changes in the sample in respect of the original contents to an acceptable minimum. Preservation will only be sufficiently effective during a certain period of time. This period of time is the so-called preservation period. Within this period, samples have to be treated in such a way, that the concentration has been secured by (for instance) an extraction or by measuring.

The prescribed preservation methods that - depending on the parameter - may consist in packaging, method of preservation, circumstances of storage, are mentioned for each parameter in SIKB Protocol 3001 "Method of Preservation and Preservation Periods of Environmental Samples". The current version is available on www.sikb.nl; for the laboratory performing the task, this version is binding.

2.1.2 Preservation Periods

2.1.2.1 Backgrounds of the Preservation Period

When a sample has been properly preserved according to the directions, the preservation period is of special important for achieving a result that meets the expected measurement uncertainty.

For projects, the (sub)samples of which are delivered over a longer period of time, while the assignment is only given afterwards, in concert with the client, the laboratory may choose to secure the contents by, for instance, extracting. In this way, it can be guaranteed that the preservation periods are not exceeded and consequently the quality of the analyses can be assured.

With respect to the acceptance the samples, the following main conditions should be observed first of all:

1. The assignment must be complete before it can be executed.
2. The samples should meet the technical specifications regarding packaging and preservation.

If it appears that during the acceptance by the laboratory or during entry into the laboratory the sample has not been properly preserved, this must be recorded in the analysis report. A sample that has not been preserved correctly, will always be processed

all the same. Even when the right preservation is lacking, an analysis will be executed according to AS SIKB 3000 and reported on.

In the analysis report the following remark must be included:

"Deviations from the directions have been established that may have influenced the reliability of the marked results in this analysis report."

Following this "heading", the established deviations will be mentioned, e.g.:

The sample has been delivered for the analysis in question in an unsuitable receptacle.

Criterion:

If it appears from the transference form or from the (standard) receptacle used that the sample has not been preserved properly, this should be recorded in the analysis report. In the analysis report it should be indicated which test the recorded remark refers to.

If the two main conditions are met, the preservation periods given in protocol 3001 are in force and the sample can be processed. When the samples are delivered in time (for instance on the day of sampling or the first day after), the laboratory must make sure that the contents are safeguarded within the given preservation periods.

2.1.3 Acceptance of Samples and Sample Data, Transport and Storage of Samples

2.1.3.1 Transfer and Acceptance of Samples and Sample Data

This AS refers to subprocedures that are carried out after the sample has been accepted by the laboratory. In a number of cases, the moment of accepting the sample may be before the transport and / or the (interim) storage of the sample. Therefore, the requirements of the procedure that refer to the transport and the interim storage are also described in this AS.

The transfer of samples from the client to the laboratory is recorded in the form for the transfer of samples or in an instruction form. During the transfer, the presence of the samples (number of pots / bottles) is determined. In case any samples (or pots / bottles) are missing compared to the number indicated by the sampler, this will be recorded in the form. The individual samples must be provided with a unique code (e.g. a barcode; project- and sample description).

At least the following data should be provided:

- Name of client
- Date of sampling
- If applicable, an indication of a health hazard (e.g. warning stickers for samples suspected to contain asbestos).

If the client has not specifically stated otherwise, the laboratory will assume that the preservation of the sample has been executed according to the requirements of this accreditation scheme.

If according to the statement of the client (of the analysis) the sample has not been preserved, the sample will be processed. The laboratory will not contact the client prior to this. If the client requests the preservation, the laboratory will as yet carry it out.

Remark:

The laboratory is under no responsibility to check whether the sampling has actually been executed according to the requirements. The responsibility for this rests with the client.

Remark:

If no information is given by the client regarding the state of preservation of the samples, but the receptacles used were supplied by the laboratory and were in addition supplied with a preservative, it may be assumed that the samples delivered have been

preserved. Nevertheless, it has to be checked, whether the expiry date of the preservative has not elapsed.

Acceptance of an order refers to the part of the procedure when the laboratory has received all samples as well as complete order (inclusive of sample data) from the client (of the analysis) on the location of the laboratory. Only after the order has been accepted, the laboratory can start its activities of preparation and analysis for securing the contents.

Remark:

When the (analysis) client hands over the samples to a courier or to a location that is managed by the laboratory, from that moment onwards the laboratory is responsible for the transportation- and storage conditions.

The laboratory will not verify the date of the sampling, neither will it let its actions depend on the date of the sampling. If the date of sampling is not known, the standard remark of § 2.1.2 will be included, with the addition, that the remark follows from the fact that the sampling date is unknown.

After the reporting, this may lead to requests of the (analysis) client to introduce a sampling date as yet, and to generate another a report (without the standard remark). The laboratory will only perform this, after the (analysis) client has sent in a written confirmation regarding this request. The fact that it concerns a corrected report will be made clear in the report by means of a remark.

After acceptance of the samples the client must be in the possession of a receipt.

2.1.3.2 Interim Storage of Samples

With interim storage of samples the short-term storage prior to the transportation to the laboratory is referred to. This may either be in the location, in which the samples have been taken, or in a central place, where samples are collected. An agreement should be made as to which party is responsible for the interim storage.

- Transport the samples to the laboratory that will execute the analysis as soon as possible after sampling, in any case within the time periods mentioned in Protocol 3001 regarding the number of days within which the laboratory must process the samples.
- Store all soil samples that have not been taken to the laboratory on the day of sampling in a cooling room, refrigerator or any other means of cooling, with a storing temperature between 1 and 5° Celsius. During storage of the samples, the temperature of the storage room must be between 1 and 5 °C for 95% of the check-ups.

Criterion:

If the responsibility of the interim storage of samples rests with the laboratory, it must to be ascertained that for 95% of the check-ups the temperature of the store room during storage of the samples will be between 1 - 5 °C. At least once every working day, the temperature should be taken at the entrance of the cooling room with a calibrated thermometer in a sufficient amount of water. If this is not complied with, it must be recorded in the analysis report.

2.1.3.3 Transportation of the Samples

With transportation of the samples reference is made to both the transportation from the sampling location to a central storing place and to the laboratory. Samples must be transported as described in VKB Protocol 2001.

Care should be taken that the samples do not freeze and that they warm as little as possible to prevent volatilization and decomposition. To achieve this, during the transport

provisions should be made, e.g. by using a coolbox with cooling elements or an active cooling system. In view of the great variation in circumstances no requirements are made in respect of the temperature range during the transportation as such.

2.1.3.4 Storage of Samples in the Laboratory

During storage in the laboratory, the samples should be cold-stored at a temperature of 1 to 5 °C. It has to be ascertained that this requirement is met. Even if preservation techniques are applied, it is advisable to secure the samples as soon as possible.

Criterion:

The temperature of the cold-storage room of the laboratory should be between 1 and 5 °C during 95% of the time. This should be ascertained by taking the temperature at least twice a day with a calibrated thermometer in an ample amount of water at the entrance of the cold-storage room. If this is not complied with, it should be recorded in the analysis report.

2.1.3.5 Necessary Performances on-site and Aim of the Investigation

The client is responsible for the delivery of all relevant information in respect of the execution of the investigation, in order that for the laboratory the purpose of the investigation is sufficiently clear. This is important in view of the choices the laboratory has to make regarding the right method of pretreatment for the parameters to be determined.

2.2 Sample Pretreatment

Sample pretreatment is performed in the laboratory. By sample preparation reference is made to:

The taking of a representative (sub)sample from the sample acquired during sampling.

The main procedures with respect of sample pretreatment are:

- Preliminary Reducing
- Sub-sampling (quartering, random sampling, etc.)
- Homogenizing
- Reducing
- Decanting (if applicable)

All samples investigated according to this AS undergo a sample pretreatment procedure according to Draft-NEN 5709.

The number of sub- or random samples used for the compound sample, influences the representativity of the (analysis) sample. The maximum number of subsamples to a compound sample depends (also) on the investigation strategy employed (for further information, please refer to NEN 5740 and Draft-NEN 5709). If the assignment of the (analysis) client does not comply with the maximum number of samples composing the compound sample, the laboratory will execute performances according to the assignment. If the number of subsamples has been exceeded, this must be recorded in the analysis report.

Criterion:

As the investigation strategy (NEN 5740) is often not known to the laboratory, the laboratory should test the assignment only in respect of the criteria of Draft-NEN 5709. This means that a composite sample may consist of 10 subsamples at the most, and at least 3 test samples should be taken from each subsample over the complete scale of the

sample. If latter requirement in respect of the number of composite samples has not been complied with, this must be recorded in the analysis report.

The sample pretreatment, as executed in the laboratory, is described in Draft-NEN 5709 and in the protocols containing the performance sheets of the related analysis methods.

2.3 Sample Preparation / Extraction / Destruction

Sample preparation is:

The working up of the subsample derived from the sample pretreatment for the execution of the analyse.

The amount of the sample input during the sample preparation and the extraction / destruction is described in Draft-NEN 5709 as well as in some cases in the analysis standard. Draft-NEN 5709 mentions the minimum amount of the sample to be used. This only depends on the size of the particles of the sample in question. This prescribed minimal amount to be used according to Draft-NEN 5709 is the guideline and should therefore be preferred to the amount formulated in the analysis standard.

2.4 Analysis

The analysis is executed and recorded in accordance with NEN-EN-ISO/IEC 17025. Additional requirements regarding the validation, execution and additional quality assurance of the analysis are described in Chapter 3 and in the performance sheets of the related analysis methods. If during the analysis process it turns out that a preservation period may be or will be exceeded, the laboratory will execute the activities in accordance with the assignment. In case a preservation period has been exceeded, this will be recorded in the report by means of a standard remark (see § 2.1.2).

2.5 Requirements regarding Reporting to the Client

The requirements to be met regarding reporting are described in NEN-EN-ISO/IEC 17025 (§ 5.10). If a deviation of this standard is established, a standard remark will be recorded in the report. Except for the investigation results, the laboratory will report the following data:

With respect to the preservation:

- If applicable, the unpreserved delivery of the samples and a consequent preservation performance in the laboratory.
- If applicable, the unsuitably preserved delivery of the samples.
- If applicable, that it is unknown, whether the sample was preserved at the time of delivery.

With respect to the packaging:

- If applicable, the delivery of the samples in an incorrect receptacle.

With respect to the preservation period:

- If applicable, the exceeding of the preservation period.

With respect to the sampling date:

- If applicable, that the date of sampling is unknown.

If one of the above mentioned deviations applies, it will be recorded in the report in connection with the standard remark (see § 2.1.2). If after the reporting of the investigation additional data become available to the laboratory (such as the sampling date or the information that the sample was preserved at delivery) and if they lead to the issuing of a new analysis report, it will be noted in this report that it concerns a corrected version.

Remark:

The remarks in the report form an integral part of that report.

Requirements for the application of the accreditation mark AS SIKB 3000 are described in Attachment 2.

The minimum reporting limit laboratories are allowed to apply is determined on the basis of the DL (Dutch AG) the laboratory has determined for the performance in question as well the target value. The minimum reporting limit is:

- three times, if three times the DL is smaller than the target value
- the target value, if the DL is smaller than or equivalent to the target value and if three times the DL is larger than or equivalent to the target value
- the DL, if the DL is larger than the target value.

If for a group of parameters the measured concentrations must be added up and (part of) the measured concentrations fall beneath the reporting limit used by the laboratory, for the total sum a value of $0.7 * \text{the reporting limit}$ is applied for those components. If the concentrations of all components fall beneath the reporting limit, in front of the sum, a sign indicating 'smaller than' will also be inserted. An example of this is the determination of the PAK 10 VROM content: if the reporting limit for all components is 1 [mg/kgDS] and the concentrations of all components fall beneath the reporting limit, the PAK 10 VROM content will be <7 [mg/kgDS]; if 1.5 [mg/kgDS] has been determined for the concentrations of one of the components and the rest of the concentrations fall beneath the reporting limits, the PAK 10 VROM content will be 7.8 [mg/kgDS] ($9 \times 0.7 + 1.5$).

2.6 Requirements regarding the After-Care

2.6.1 Complaints

Any complaints will be settled in accordance with NEN-EN-ISO/IEC 17025 (§ 4.8). With respect of this, a complaint-file should be set up, in which the relevant information is recorded. A responsible person must be designated for this, who is in charge of supervising the settlement of the complaints. The person or institution that lodges the complaint, should be informed in writing about the findings and the conclusions. The laboratory should also check if complaints are of a structural nature and take the necessary measures to solve the problems.

2.6.2 Sample Storage and - Removal

After the investigation has ended, the remaining part of the delivered samples will be stored for a standard period under storage conditions (in darkness and at a temperature between 1 and 5 °C), except in case other agreements have been made with the client. If the (agreed) storage period has expired, the samples will be transported to a recycling

firm. This firm will destroy or recycle the abducted samples in a manner, which is controllable and shows a sense of responsibility towards environmental hygiene.

2.7 Requirements regarding Filing

The additional requirement regarding the management of registrations of NEN-EN-ISO/IEC 17025 § 5.4.7 and § 5.10 is, that the laboratory must keep the raw data of the investigation results of AS SIKB 3000 for 2 years. The analysis reports must be kept for at least 3 years.

3 Validation of a Performance and Requirements regarding the internal Quality System

3.1 Introduction

This chapter describes the method in which the validation investigation and the equivalence investigation must be performed. Reference will be made as much as possible to the standards that have been drawn up in respect of this: the NEN 7777 (Environment - Performance characteristics of measurement methods) and the NEN 7778 (Environment - Equivalency of measurement methods). These standards often present various possibilities. In this chapter, the possible choices having been made in the context of this AS are described.

§ 3.2 till and including 3.5 deal with the validation investigation related to the choice of the performance characteristics (§ 3.2), the validation matrix (§ 3.3) and the number of decades within which the validation has to be performed. (§ 3.4). § 3.5 describes the procedure for each performance characteristic and its quantification.

§ 3.6 describes the equivalence investigation of a non-standardized performance. The equivalence investigation is not an obligatory part of the validation investigation. It only needs to be executed, if a standard method was prescribed as a reference method, yet the laboratory wishes to deviate from the standard method in respect of some important details. The equivalence investigation is additional to the validation investigation.

§ 3.8 describes the usefulness of the performance characteristics of the FeNeLab interlaboratory investigation for the validation investigation and the equivalence investigation.

The important information that can be obtained about the group performances of the participants will also be indicated.

The last paragraph of this chapter deals with the internal quality audits by means of control charts. In addition, requirements will be formulated regarding the frequency of blind sample testing and proficiency testing, which depends on the number of analyses performed yearly. Another important component of the internal quality audit is the periodical test, whether the analysis method of the previous term shows performance characteristics equivalent to those determined at the validation.

3.2 Selecting the Performance Characteristics

With the introduction or alteration of a specific performance, an intralaboratory validation investigation has to be executed. The following performance characteristics can be involved in the validation investigation:

- detection limit;
- measurement range;
- deviation from the standard (alinenarity)
- accuracy/retrieval;
- intralaboratory reproducibility.
- selectivity
- robustness

Concerning a standard method, the laboratory has to prove its ability to execute the method according to the requirements of the standard. The following performance characteristics must be determined:

To perform	Performance characteristics
Always	Measurement range, accuracy, reproducibility
If relevant	Detection limit, deviation from model (alinenarity)

If an analysis is not performed according to a standard method, the following performance characteristics must be determined:

To perform	Performance characteristics
Always	Measurement range, accuracy, reproducibility, selectivity, robustness
If relevant	Detection limit, deviation from model (alinenarity)

These performance characteristics don't always have to be carried out in separate investigations. Very often (historical) information is available in another way, on the basis of which a sufficiently correct estimation of the relevant performance characteristics can be made. Some examples of this are:

- Determining the intralaboratory reproducibility from control charts of the control samples that meet the set requirements.
- Determining the intralaboratory reproducibility and accuracy from the results of interlaboratory investigations with reference materials with a consensus value (FeNeLab- interlaboratory investigation), in which the laboratory has to participate regularly and continuously.
- Determining the robustness or selectivity from the reproducibility standard deviation, for example from control charts of control samples that meet the set requirements.

In order to determine the performance characteristic, the analysis instructions must be followed completely, inclusive of the applicable extraction or digestion. If this is not applicable, it will be indicated in the performance sheet explicitly, for which part of the performance the performance characteristics are valid.

The sample preparation (reducing and/or homogenizing) falls in principle outside the scope of the determination of the performance characteristics of an analysis method. Preferably, homogeneous samples are selected for the validation of an analysis method. These samples must have a sufficiently small particle size, in order that the mass to be processed for the analysis, falls within the limits set by Draft-NEN 5709. If on-site samples are used for validation, care has to be taken that the working material will be made homogeneous first, and that the subsamples will be composed from the homogeneous material afterwards.

During validation, four situations can be discerned:

- Parameters that determine the method: No deviation from the (measurement) principle is allowed and the method has to be executed in conformity with the defined performance.
- Parts of the test determine the method: For the specific parts (e.g. extraxtion) no deviation from the (measurement) principle is allowed and these parts of the test have to be executed in conformity with the definined performance. This does not apply for the other parts of the test (e.g. the measuring).
- Attention points: Parts of the investigation that are obligatory for the reference method, but possibly not obligatory for any other method. An example of this is the maximum amount of matter that is allowed to be present during the destruction for the determination of metal.

- Method without limitations: The measurement principle and the method of execution may be chosen freely.

In each of the above four situations it must be shown in the validation investigation that the performance characteristics meet the requirements set in the performance sheet.

If the method is altered, a (limited) re-validation will be necessary, focussing on those performance characteristics that may have been influenced by the alteration.

3.3 Selecting the Validation Matrix

For the selection of the validation matrix a distinction is made between the validation matrix for the detection limit and the validation matrix for the rest of the performance characteristics. For the determination of the detection limit (clean) sand must be used as validation matrix. This was chosen, because in the Dutch Act on Soil Remediation, the defined target values for this matrix are also the lowest. For this reason it is important to know, if this can be measured with the extraction and analysis method applied.

For all performance characteristics other than the detection limit, according to NEN 7777 a validation in the most difficult matrix suffices. Regarding soil, a distinction is made in:

- poor in organic matter, i.e. soil with a content of organic matter of < 10%.
- rich in organic matter, i.e. soil with a content of organic matter of > 10%.

Remark:

During an investigation of the data files of 5 laboratories for environmental research, it turned out that the 90%percentile value for organic matter is 10%.

The table below indicates for each parameter what the matrix-related critical phases are in respect of the pretreatment (digestion-/extraction output) and/or the measurement (matrix effects like signal repression and/or blank-interference). On the basis of these considerations, the table indicates, in which cases a most difficult matrix may be defined:

Table 3.1 Most difficult matrix for the analyses performed

Parameter	Critical steps		Most difficult matrix	
	Digestion/ extraction	Measurement	Poor in organic matter	Rich in organic matter
pH	---	---	X ¹⁾	
Dry matter	X	---		X
Clay	X	---		X
Organic matter	---	---	X ²⁾	
Metals (7)	X	---		X
Mercury	X	---		X
anions (Cl ⁻ , F ⁻ , Br ⁻ , SO ₄ ²⁻	---	---	X ¹⁾	
cyanides	---	---	X ¹⁾	
Aromatics and volatile CHC and volatile chlorobenzene s	X	---		X
PAH (10 VROM)	X	---		X
EOX	X	---		X
Mineral oil	X	---		X
OCP and PCB and non- volatile chlorobenzene s	X	---		X

¹⁾: No matrix can be mentioned here that, compared to the other matrices, presents extra problems during the determination. For this reason a matrix poor in organic matter may be used as a most difficult matrix (optionally it may also be one of the other matrices),

²⁾: For organic matter no 'most difficult matrix' is defined, because during validation, the analysis must be performed on an appropriate level.

3.4 Selecting the Number of Decades during which Validation should be performed

According to NEN 7777 the determination of the reproducibility standard deviation must be performed in two submeasurement ranges, unless the field of application is one decade the most; in this case one submeasurement range will be sufficient. The submeasurement ranges to be selected are the lowest and highest decade of the measurement range. In principle, the same goes for accuracy; only if there is no suitable reference material available in the required submeasurement range, this may be a reason to validate on less or on different levels.

The range of the method must extend to at least the target value till 1,2 x the intervention value. On this basis, the table below gives an indication of the required number of decades the method must at least include.

Table 3.2 Number of Decades for each Performance

Parameter	Target value (mg/kg ds)	Intervention value (mg/kg ds)	Ratio intervention value/ target value	Minimal number of decades ¹⁾
pH	n.a.	n.a.	n.a.	2 ²⁾
Dry matter	n.a.	n.a.	n.a.	2
Clay	n.a.	n.a.	n.a.	2
Organic matter	n.a.	n.a.	n.a.	3
Metals (7)	between 0,8 and 140	between 12 and 720	between 1,9 and 15	2
Mercury	0,3	10	33	2
Cyanides (free / complex pH>5)	1 / 5	20 / 50	20 / 10	2
Aromatics and CHC	between 0,01 and 0,1 between 0,002 and 0,4	between 1 and 130 between 1 and 60	between 100 and 13000 between 2,5 and 2000	3
PAH (sum 10 VROM)	1	40	40	3
EOX	0,3	n.a.	n.a.	3
Mineral oil	50	5000	100	3

¹⁾ The number of decades has been calculated from the ratio $1,2^*$ intervention value in respect of the AG_{eis} ,

²⁾ The determination of the pH must be validated on at least 2 levels.

The table shows that, with the exception of the determination of the pH, the minimum number of decades is in all instances at least two. According to NEN 7777, this means that validation of the reproducibility must be performed on two levels, viz. the lowest and the highest decade. For the determination of the pH, validation must be performed on two levels.

The determination of accuracy will in many cases be performed on the basis of material stemming from the FeNeLab interlaboratory investigation. As this material is not available in different ranges, validation may in this case be performed on one level, on the provision that the concentration(s) for the determination(s) in question lies or ly within the required decades. (see Table 3.2). Appendix 3 shows the consensus values of the FeNeLab sample.

3.5 Testing Procedure and Quantification of Performance Characteristics

During the testing of the performance characteristics in comparison to the given requirements in the performance sheet, the starting point for the given performance characteristics is an estimated limiting value as defined in NEN 7777 and NEN 7778.

3.5.1 Detection limit (DL, Dutch: AG)

The detection limit (DL) of a component is determined according to NEN 7777. As the external requirements imposed under repeatability conditions must be determined, within this AS a detection limit is also operative, which under repeatability conditions is determined $DL_r (=3*S_r)$. This is a deviation from what has been determined in NEN 7777. For determining the detection limit a (clean) sand sample is taken to start from, unto which a known amount of the material is spiked. For the determination of the detection limit, the performance is octuple at the least. With this procedure, the samples must pass through the whole proces (from sample pretreatment (if applicable) until and including the analysis).

Explanation:

Spiked samples must be prepared in such a way that they are representative for an on-site sample. If the stability and the volatility of the analyte are sufficient, 48 hours should pass before the sample is being processed. For the determination of volatile compounds (boiling-point <300°C at 1 atmosphere) the stability is insufficient, and after the spiking of the compounds the extraction can be started. The material will therefore not become out-of-date. As regards to the determination of the PAK VROM content out-datedness does not play a role during the process, as one of the compounds (naphtalene) comes under volatile compounds. Determine the concentration of the analysis sample eightfold with - and in singular number without - addition. The concentration of the added analytes must be so high, that the measurement uncertainty at this level of concentration is small in respect of the measurement uncertainty of the analysis method aimed at. When selecting the "blank" sample material to which the analytes will be added, the most difficult matrix for the performance in question must be taken into account (see Table 3.1).

Criterion:

The detection limit must meet the requirement mentioned in the performance sheet.

Explanation:

The requirement of the detection limit is in principle 0,33 times the target value, except when this requirement cannot be met with the described analysis method (see the performance sheets in the related protocols).

3.5.2 Determining the Measurement Range of the Performance

The determination of the measurement range is executed according to NEN 7777. The measurement range is the domain between the detection limit and the highest value (minimal intervention value) of the measurement quantity that can be determined according to the instructions, whereby the deviation from the method should fall within certain limits. If the upper limit of the linear range is known, the measurement range can be easily found.

Explanation:

The measurement range is defined for the whole analysis method. If dilution of the sample forms an explicit part of the determined measurement method, during the determination of the measurement range the applied dilution should be taken into account. However, it should not be taken for granted that by diluting extracts or destruat the upper limit of the measurement range can be enlarged indefinitely without this being shown.

3.5.3 Testing of the Standard Deviation (alinerity)

The standard deviation (alinerity) of a method is determined in accordance with NEN 7777.

The testing of the standard deviation concerns the chosen calibration function of the analysis-instrument used and will be executed with the aid of calibration standards and testing standards. As only that part of the linearity is tested which is related to the instruments, these standards are not applied to the whole method.

The testing of the usefulness of the chosen calibration function for the whole measurement range is described in NEN 7777. The testing must be executed on the basis of absolute limiting values.

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

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

3.5.4 Determining Intralaboratory Accuracy

The intralaboratory accuracy of a method is determined in accordance with NEN 7777. The intralaboratory accuracy of the performance can be determined – in decreasing degree of preference – in the following ways:

- by regular, continuous participation in interlaboratory investigations with reference materials with a consensus value (FeNeLab-interlaboratory investigation)
- by representative certified reference material
- by a representative sample with a conventional true value
- "spiked samples" (retrieval as an estimator of the accuracy)

The participation in a regular, continuous interlaboratory investigation is preferred to certified reference material, because in course of time this leads to a better insight into the accuracy with respect to other laboratories.

A representative sample with a conventional value can derive from a proficiency test, which is being taken part in.

Criterion:

The retrieval must meet the requirement mentioned in the performance sheet.

3.5.5 Determining the Intralaboratory Reproducibility Standard Deviation

For the determination of the (intralaboratory) reproducibility standard deviation, according to NEN 7777 the following procedures may be used:

- regular, continuous participation in interlaboratory investigations with reference materials having a consensus value (FeNeLab-interlaboratory investigation)
- duplo analyses of different laboratory samples on different days
- repeated analyses of the same sample on different days
- repeated analysis of spiked laboratory samples

Criterion:

The intralaboratory reproducibility standard deviation must be lower than the requirement given in the performance sheet.

3.5.6 Determining the Selectivity

Selectivity must be determined in accordance with NEN 7777, if the deviation from the standard occurs in those points in the process, where the selectivity could have been diminished. In those cases, with the aid of on-site samples, it needs to be determined whether the selectivity of the performance is at least equivalent to the standardized performance. The selectivity only has to be determined in those cases, in which the influence is insufficiently covered by another performance characteristic, such as accuracy.

3.5.7 Determining the Robustness

Robustness quantifies the change in measurement result by deviations in the performance and the measurement circumstances, as well as in the nature and composition of materials, as they can be found in practice.

The intralaboratory reproducibility is a good indicator of robustness. As information about the reproducibility is often abundantly available, a separate determination of the robustness won't usually be necessary.

3.6 Equivalence Investigation of a non-standardized Performance

3.6.1 Execution of the Equivalence Investigation

The equivalence investigation is not an obligatory part of the validation investigation. It should be executed only, if a standard method has been prescribed as reference method and if the laboratory wishes to deviate from the standard method in important parts of the investigation. The equivalence investigation is additional to the validation investigation.

The equivalence investigation is executed in accordance with NEN 7778. The equivalence investigation includes:

- intralaboratory reproducibility as a characteristic of precision
- accuracy as a characteristic of systematic deviation
- detection limit, only if applicable

These performance characteristics are determined during the validation investigation.

The equivalence investigation can be considerably shortened, if the testing needs to be performed only for one value, as is the case with intralaboratory reproducibility and the detection limit.

3.6.2 Testing the Equivalence

The equivalence must be shown for each performance characteristic. The test is to be executed in view of the requirement mentioned in the performance sheet according to the method given in NEN 7778 for an estimated limiting value.

The performance characteristic have to comply with the estimated limiting value. If this is only slightly exceeded, the effectiveness is, however, not at stake. The numerical value of a performance characteristic will directly be compared to the limiting value. At the lower limit it must be higher, at the upper limit it must be lower than the limiting value.

3.7 Reporting the Operations

The validation investigation is concluded with a validation report. The conclusion of the investigation should be relevant in relation to the requirements of this AS and must be recorded in the performance sheet, in which the result of the validation and the eis are also included.

Complete documentation of every validation investigation performed must be available during the accreditation survey.

3.8 Usefulness of Performance Characteristics from the FeNeLab Interlaboratory investigation for the Validatie investigation and the Equivalence investigation

Since 1999, the majority of laboratories for environmental research have participated in a continuous interlaboratory investigation. Daily or weekly, the same samples are analysed and the results centrally processed and reported. In course of time, an enormous amount of data of analysis results have been collected from the participating laboratories as a group, from each laboratory individually, as well as in respect of the groups results.

The following performance characteristics from this data file are relevant for the validation investigation in the context of this AS:

- Intralaboratory reproducibility standard deviation (S_W),
- Interlaboratory reproducibility standard deviation (S_R),
- accuracy, rendered by the deviation from the intralaboratory average in respect of the "overall" average (consensus value)

In the above mentioned interlaboratory investigation homogenized samples are used. Any effects caused by sample inhomogeneity are not taken into consideration. As the sample pretreatment for the regular samples is executed in conformity with Draft-NEN 5709, it may be assumed that the inhomogeneity in the pretreated subsample exerts a slight influence on the overall measurement uncertainty.

The validation data from the interlaboratory investigation of the FeNeLab sample can therefore be used in the validation investigation in the context of this AS. Information about effects related to the concentration can be acquired from a sufficiently diverse supply of reference samples. In this case, additional validation is not necessary. In order to be able to use these data for the in-house validation investigation or equivalence investigation, it is a prerequisite for the laboratory in question to participate in the FeNeLab interlaboratory investigation on a regular basis (daily or weekly) and to report the results to the organising institution. The concentrations and the related performance characteristics that have been reported in the 3rd quarter of 2003, are listed in Attachment 3.

3.8.1 Usefulness of the Performance Characteristics for Intralaboratory Validation

The S_W and the consensus value that have been determined during the interlaboratory investigation can be tested with the estimated limiting values as mentioned in the performance sheet. The remaining performance characteristics to be determined (a.o. detection limit) are of course still to be determined.

3.8.2 Usefulness of the determined Performance Characteristics for Interlaboratory Validation

The S_R and the accuracy determined during interlaboratory investigation are suitable to be tested in respect of the estimated limiting values as mentioned in the performance sheet. The S_R is not an obligatory part of the validation investigation. The S_R can, however, provide important information about the group performances of the participants in the FeNeLab interlaboratory investigation.

3.8.3 Usefulness of the determined Performance Characteristics for Equivalence Investigations

During the execution of the equivalence investigation in accordance with NEN 7778, § 9.2.7 and § 9.2.8, the (S_W) and the accuracy determined in the FeNeLab interlaboratory investigation can be used for testing in comparison to the imposed performance characteristics, in particular to the respective estimated limiting values as given in the performance sheet of the performance in question.

The detection limit must be determined separately in accordance with NEN 7777.

3.9 Internal Quality Audits

Internal quality audits should meet the requirements described in NEN-EN-ISO/IEC17025. This chapter presents specific subjects in connection with the purpose of this AS.

The internal quality audits are:

1. Control charts for monitoring the performance
2. Blind or double blind sample testing
3. Proficiency testing
4. Semiannual performance investigation

3.9.1 Control Charts for Monitoring the Performance

Monitoring of a performance by a control chart must be performed according to NPR 6603. As several points mentioned in this standard will need clarification, in this paragraph a number of issues will be explained and additional points of action will be described.

- ✓ When starting a control chart, for at least the first 10 observations an outlier test is to be executed, if the observed performance characteristics don't meet the requirements. To identify the outliers, Grubbs-test will be performed twice at the most. When the outliers have been removed, both the average and the standard deviation will be calculated again and the outlier test will be repeated. This whole procedure will be repeated until no outliers will be found any more. To be able to use the remaining observation data for initiating a control chart, at least 8 observations should remain.

Grubbs-test:

$$G_p = \frac{|x_p - \bar{x}|}{s}$$

in which x_p individual observation tested
 s standard deviation

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

with $n=10$ $G_p = 2,482$
 $n=9$ $G_p = 2,387$
 $n=8$ $G_p = 2,274$

- ✓ If uncontrolled quality is found as a result of 11 observations, all above or all below average, the cause of the shift should be immediately examined. The contents belonging to the eleventh observation can still be reported. If the problem cannot be solved (the cause is unknown or is impossible to remove), the retrieval must be determined again and tested in comparison with the criteria stated in the performance sheets. The control chart will be immediately closed and the charts joined. The new chart will start with $n=0$, $\sum x=0$ and $\sum x_i^2=0$. When a new control chart is closed, the (cumulative) standard deviation which was determined in the previous periods, is still being used. During the determination of the relevant performance characteristics, samples may still be analysed and reported. To prevent quality from becoming uncontrolled, the results of the control chart for performance monitoring have to comply with the characteristics of the last control chart.
- ✓ When calculating the average and the standard deviation of a full control chart, uncontrolled results caused by the exceeding of the 3s-limits are not incorporated.

- ✓ With the closure of a control chart, the average and the standard deviation are tested with respect to the historical data of previous charts and the requirements set in the performance sheets regarding accuracy (retrieval) and intralaboratory reproducibility.
- ✓ NRP 6603 describes, how the test with respect to the historical data of the previous charts is to be executed for the standard deviation, but not for the average. The average must be determined with the t-test:

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

X_1 and X_2 are the average values of the previous control chart(s) and the present control chart respectively.

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

s_1 and s_2 are the standard deviations from the previous control chart(s) and the present control chart respectively.

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

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

- ✓ If the average and/or the standard deviation may not be joined together, the cause of it should be investigated immediately. If the quality of the analysis has improved (retrieval closer to 100% or a smaller standard deviation) the cause of this does not have to be examined further. If the standard deviation value has increased or the retrieval deteriorated, but the requirements set in the performance sheet are nevertheless being met, the cause does not need to be examined and the charts may be joined. In latter case, a recalculation of an average or standard deviation may be considered.

If no improvement of quality is observed, or if the retrieval of the standard deviation does not meet the requirements of the performance sheet, the cause of this is to be investigated. If even so the problem cannot be solved (the cause is unknown or cannot be removed), the relevant performance characteristics are to be re-determined and tested again to the criteria stated in the performance sheets. In case of a deviation from the average, the accuracy/retrieval is to be re-determined and in case of a deviation from the standard deviation the intralaboratory reproducibility standard deviation.

The control chart is closed and the charts are joined. The new chart starts with $n=0$, $\sum x=0$ and $\sum x_i^2=0$. During the determination of the relevant performance characteristics, samples may still be analysed and reported. To prevent quality from becoming uncontrolled, the results of the control charts for performance monitoring have to comply with the characteristics of the last control chart.

After it has been concluded that no cause can be detected, the performance characteristics of the new control chart are to be determined within 10 measuring days. As an additional requirement these 10 measuring days have to fall within a period of 3 months.

- ✓ In order to make sure that the last data will still exert sufficient influence on the values when the control charts are joined, during the joining the preceding four charts (approximately 120 observations) will be maximally made use of. With the start-up of 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.

- ✓ In a control chart, fixed warning- and alarm limits may be applied, if the standard deviation in the analysis is smaller than or equal to the intralaboratory reproducibility standard deviation mentioned in the performance sheet. For the standard deviation, maximally the intralaboratory reproducibility standard deviation, as given in the performance sheet of the related parameter, may be used. During the closing of the control chart, the average and the standard deviation are tested in respect of the criteria mentioned in the performance sheet.
- ✓ If several control chart samples are being processed for an analysis each day, according to a predetermined pattern only one control sample has to be registered in the Sheward chart. The control chart samples that are not registered in the Sheward chart, still have to be tested with the aid of the current Sheward chart.

Apart from this audit, the laboratory has to make sure that the degree of sensitivity of an analysis does not drop beneath a critical borderline-value, so that the required detection limits can always be met. This can be guaranteed, for example, by setting requirements to the slope of the calibration curve.

The laboratory should also periodically, by means of a blank analysis, test the (cross)contamination of the applied chemicals and the methods of pretreatment and analysis. For the applied chemicals, a blank control is to be executed, when a new batch is introduced. Regarding the pretreatment- and analysis methods, for each series at least one blank sample test is to be executed. The blank sample must in this case pass through the entire process (from sample pretreatment till and including the analysis).

For most components, in the related performance sheet a requirement is given for the maximum concentration of the required detection limit analyte in the blank sample. Generally speaking, latter value must be lower than the detection limit ($AG_{r,eis}$) required in the performance sheet of the analyte in question. If the blank level exceeds the detection limit, the laboratory has to prove that the blank level is under control, by executing a multiple blank analysis for each measurement series. The standard deviation, calculated on the basis of the multiple blank determination, should in this case come beneath the detection limit given in the performance sheet. If a extremely increased blank level is observed without an apparent cause, the whole series has to be started again. If the average content in the blank determination has increased with more than twice the $AG_{r,eis}$ in respect of what has been found during start-up or periodical validation, the laboratory should to determine whether the set detection limit can still be met.

If the above mentioned requirement is met, the contents in the samples that have been measured in the same series, are corrected for the average value of the related blank samples.

3.9.2 Blind Sample Testing

The laboratory has to perform blind sample testing, if it does not participate or if it insufficiently participates in proficiency testing (see § 3.9.3).

Depending on the number of analyses executed by the laboratory, an analysis must be audited at least 1 to 4 times a year by proficiency testing and/or blind sample testing. The type of soil, with which the audit is performed, should correspond to the on-site samples analysed. The number of samples is based on the number of samples that was tested for the analysis in question during the previous calendar year.

0	–	5000 per annum	1 tests
5000	–	10.000 per annum	2 tests
10.000	–	20.000 per annum	3 tests
> 20.000		per annum	4 tests

The following materials (with a decreasing degree of preference) may be applied for blind testing audits:

- Reference material or material with consensus value (e.g. material from the Fenelab interlaboratory investigation);
- (Additions to) on-site samples;
- Synthetic laboratory samples.

For blind sample testing, the above described sample must be analysed in duplicate. If an addition has been applied to an on-site sample, the original on-site sample must also be analysed. The contents measured in duplicate must be tested in respect of the requirements given in the performance sheet. The difference in the duplicates should be smaller than 4 times the given $S_{W,eis}$ if the content is higher than 5 times the determined AG.

The analysis must be a blind or a double blind test. This means that the person performing the analysis doesn't know the content of the sample or even doesn't know at all that the sample at hand is participating in blind sample test.

The accuracy (retrieval) is to be tested in respect of the requirements given in the performance sheet. For the test, it has to be taken as a starting-point that the requirement imposed is an estimated limiting value.

3.9.3 Proficiency testing

The laboratory should participate regularly in proficiency tests for accredited institutions. If this is not possible, or if the frequency is less than the number of times a year mentioned in 3.9.2., additional blind sample tests are to be executed.

The matrix of the proficiency testing samples should correspond to the samples analysed in practice.

For each component the z-score is calculated according to:

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

in which

X_i represents the value found
 X_{ref} is the assigned value (exclusive of outliers; according to ISO 5725-2 and, for instance, Grubbs-test)

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

$$S_{W,eis} > S_{ring} \rightarrow s = (s_{W,eis}^2 + (s_{ring}^2/n))^{1/2}$$

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

in which:

$S_{W,eis}$ is the requirement regarding the intralaboratory reproducibility standard deviation;

S_{ring} represents the standard deviation of the proficiency test regarding the assigned value;

n represents the number laboratories that have participated in the proficiency test.

If the number of participants in the proficiency test is smaller than 6, the standard deviation, s , equals $S_{W,eis}$

Only for components, in which the level is higher than 5 times the set DL (Dutch AG), the z values are calculated and assessed. The result of a component or group-component is significantly deviant from the assigned value if:

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

In Table 4.1, by way of illustration, the borderline-cases have been filled in. First of all, the last proficiency test is investigated. The maximum number of exceedings must be lower than the values shown in column 2 and column 3. If in the last and the last but one proficiency test no significant deviation has been detected, subsequently the last two tests are investigated in respect of the requirements presented in column 2 and 3.

In Table 4.1, the sequence of data presented in table 4.1 is random and is of no importance for testing a significant deviation. For the assessment, the crucial factors are the number and the nature of the exceedings (II or III).

Table 4.1 Criteria for the assessment of the proficiency testing¹

Number of samples to assess	One proficiency test	
	Allowed	Not allowed
1	II	III
2	I, II	II, II
		I, III
3	I, II, II	II, II, II
		I, I, III
4	I, I, II, II	I, II, II, II
		I, I, I, III
5	I, I, I, II, II	I, I, II, II, II
		I, I, I, I, III
6	I, I, I, I, II, II	I, I, I, II, II, II
		I, I, I, I, I, III
7	I, I, I, I, I, II, II, II	I, I, I, I, II, II, II, II
		I, I, I, I, I, I, III
8	I, I, I, I, I, II, II, II	I, I, I, I, II, II, II, II
		I, I, I, I, I, I, I, III

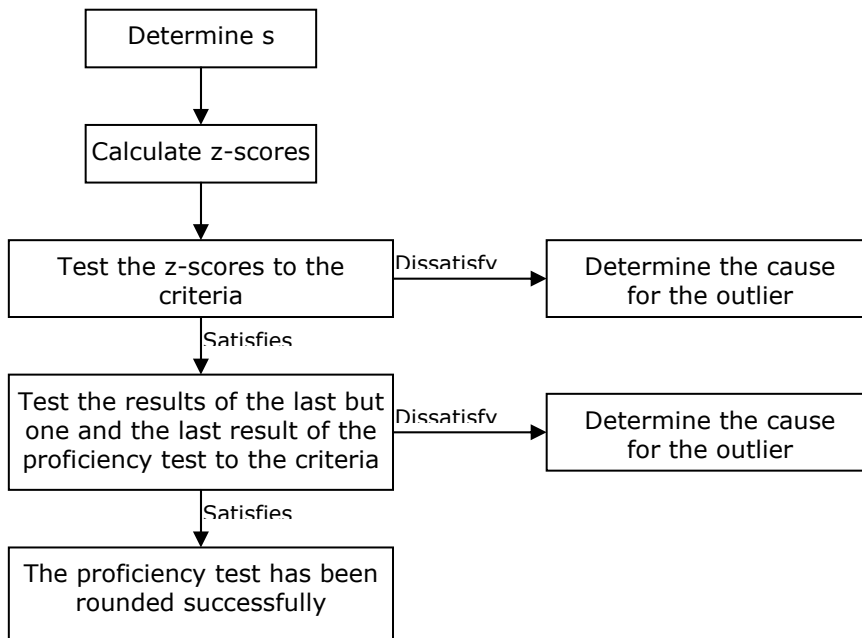
¹ **I** = $|z| < 2$ sigma (the content comes within the 95% confidence interval), **II** = $2 \text{ sigma} < |z| < 3 \text{ sigma}$ (the content comes outside the 95% confidence interval) and **III** = $|z| > 3 \text{ sigma}$ (in case of an exceeding:

determine cause of this); ²) z-scores pro parameter, which are allowed within one proficiency test; ³) z-scores pro parameter, of which the cause of the deviation has to be determined.

While assessing the table and determining whether the laboratory has undertaken timely action, the reporting time of the proficiency tests has to be taken into account. It may occur, that proficiency test n is only reported, when the results of proficiency test n+1 have been sent in. Only after the sending in of proficiency test n+2, it can be determined whether the results of a certain component showed any deviations in both proficiency test n and proficiency test n+1. It may happen that in proficiency test n+2 (which has already been sent in) the same deviation also occurs, while the laboratory is not to blame for not having taken timely action.

The diagram below outlines the necessary steps graphically.

Figure 4.1 Diagram of the steps to be taken for auditing the results of proficiency testing



If a result is significantly deviant, the laboratory must investigate the possible cause of this.

During this investigation the following actions are to be taken:

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

If during the investigation into the deviation(s) no cause has been established, one of the following actions have to be taken:

- to execute a blind sample test;
- if the destruate/extract generated by the laboratory is still present, the content is to be determined by another laboratory accredited for AS SIKB 3000.

3.9.4 Semiannual Performance Investigation

During the semiannual performance investigation, the performance characteristics of the control chart sample are audited. During this, it has to be established if the reproducibility meets the analysis requirements recorded in the related performance sheet. The requirement in respect of which the test is performed, should be regarded as an average limiting value as defined in NEN 7777. In this respect, only the average value of the reproducibility is tested, no audit is performed on an individual level. Besides this, during the semiannual performance investigation, the accuracy is to be determined according to any method.

If during the semiannual performance investigation the performance characteristics no longer appear to meet the set requirements, corrective measures should be taken, before the relevant performance characteristics are being determined again by means of a validation investigation (see NEN-EN-ISO/IEC 17025 § 4.10.2).

4 Requirements regarding the Accrediting Institution

4.1 Accrediting Institution

During the initial- or test accreditation investigation it has to be assessed, whether the analyses are being executed in conformity with NEN-EN-ISO/IEC 17025, with this AS SIKB 3000 and with the attached protocols. This assessment is to be executed either by the (Dutch) Council for Accreditation or by an organisation with which the Council for Accreditation has entered into a Multi Lateral Agreement MLA (EA/IAF) or any other equivalence agreement.

The accrediting institution may issue accreditation certificates according to this AS only, if it has entered into an agreement with the manager of this scheme, i.e. the Accreditation Council for Soil Management. Said agreement must explicitly refer to this AS.

The accrediting institution must adhere to the current rules for accreditation. Regarding accreditations for this AS SIKB 3000, for the Council for Accreditation the Dutch rules and regulations for accreditation (RAC; RvA-R2) are applicable, supplemented by the regulations described in this Chapter. Document RvA-R2 may be downloaded from the website of the Council for Accreditation (RvA), www.rva.nl.

4.2 Accreditation Investigation

It is the purpose of the accreditation investigation by the accrediting institution to determine, whether the organisation complies with and is potentially deemed to be able to permanently comply with, all parts of this accreditation scheme AS SIKB 3000 as well as the attached protocols for which an accreditation is applied.

An accreditation issued is valid for four years. During this period, this accreditation scheme AS SIKB 3000, inclusive of the attached protocols for which the accreditation has been issued, has to participate in the annual random test by the accrediting institution every year. As a rule, each separate performance should be assessed at least once every four years.

4.3 Communication between the Accrediting Institution and the Accreditation Council for Soil Management

Periodical consultations

At least once a year, representatives of the accrediting institution(s) and the Accreditation Council for Soil Management consult each other. During this consultation, the accrediting institution(s) grant o.a. insight into the content, the extent and the frequency of the accreditations in accordance with this AS SIKB 3000 of the previous year. At the same time, overall shortcomings of the previous year, observed in the branche, will be discussed. In this way, possible problem points that occurred in practice can be traced and if need be, alterations can be made in the AS.

During the annual consultation, the attention points that need to be tackled for the coming year for the overall branche, will also be discussed, as well as the functioning of the annual report described below.

Written Communication

The Accreditation Council for Soil Management will inform the accrediting institutions as soon as possible of any alterations in the AS SIKB 3000 and the attached protocols.

The accrediting institution(s) will annually report to the Accreditation Council for Soil Management the auditing results and the settling of complaints according to the rules and regulations of the previous year. For the execution of this report the agreements made between the Accreditation Council for Soil Management and the accrediting institution(s), are in force.

5 Literature

- Draft-NEN 5709
2004 Soil - Sample pretreatment for determination of organic and anorganic parameters in soil
- NEN 5740 1999 Soil - Investigation strategy for exploratory survey. Investigation of the environmental quality of soil and soil lots
- NEN 5861 1999 Environment - Procedures for the transfer of samples
- NEN 5740 1999 Soil - Investigation strategy for exploratory survey
- NEN 5861 1999 Environment - Procedures for the transfer of samples
- NPR 6603 1988 Water and sludge - Guidelines for internal quality control by the use of control charts with chemical analyses
- NEN 7777 2003 Environment - Performance characteristics of measurement methods
- NEN 7778 2003 Environment - Equivalence of measurement methods
- ISO 3534-1 1993 Statistics - Vocabulary and symbols - Part 1: Probability and general statistical terms
- ISO 8466-2 1993 Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics - Part 2: Calibration strategy for non-linear second order calibration functions
- ISO 5725-1 1994 Accuracy (trueness and precision) of measurement methods and results. Part 1: General principles and definitions
- ISO 5725-2 1994 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-EN-ISO/IEC
17025 2000 General requirements for the competence of testing and calibration laboratories

Appendix 1 Explanatory Notes to Chapter 2

1.1 Preservation and Transfer

1.1.1 Preservation

The taking of (sub)samples is an activity by which an amount of material is withdrawn from its original ("natural") environment. From this moment onwards, a sample and the environment from which the sample was taken, lead an existence independent of each other. The aim of analyzing a sample in a laboratory is, to indicate the contents in the delivered sample at the moment of sampling. For this reason, after sampling preservation techniques are applied in order to reduce the chance of changes in the sample in comparison with the original to an acceptable minimum. Preservation will only be sufficiently effective for a certain amount of time. This amount of time is the so-called preservation period. Within this period, the samples have to be treated in such a way that the concentration is secured, for instance by an extraction or by measuring. A list of the methods of preservation and the preservation periods are represented in Protocol SIKB 3001.

Preservation

A combination of precautions taken to prevent the contents of measured analytes from deviating more than acceptably after a certain period of time from the contents that would have been measured, if the analysis had been performed immediately after sampling.

These precautions can be specific actions:

- Cooling
- Adding reagentia that slow down microbial and chemical degradation.
- Filtration of the sample (e.g. for metals in on-site ground water).

Or they can be precautions related to the way of packaging:

- Packaging in an inert container
- Packaging in an air-tight container
- Packaging with a minimal layer of air above it
- Packaging in a light-tight container (in order to protect it from light. This can also be achieved by a procedure, i.e. by storage in a dark room).

If a deviation from the guidelines is established, a standard remark will be included in the report. This standard remark is as follows:

Differences from the guidelines have been established that may have influenced the reliability the marked results in this analysis report.

Under this "heading" the established points are named. Such as:

The sample for the analysis concerned has been delivered in an unsuitable receptacle.

According to statement of the client, the sample has not been delivered in a preserved state.

It is not known if the sample has been preserved before delivery.

The preservation period for the analysis concerned has been exceeded.

The date of sampling is unknown.

Remark

The standard remark is especially important to indicate that the deviant treatment (e.g. no on-site preservation) may lead to a difference compared to the original concentration (in the sample immediately after sampling). In this respect, it is not sufficient to just note that the sample has not been preserved prior to delivery.

On the request of the client, laboratories may make further information available which includes an explanatory note on the established differences regarding the guidelines and/or on the reliability of the analysis results.

1.1.2 Preservation Periods

Backgrounds of the preservation period

For a sample that has been appropriately preserved according to the guidelines, the preservation period is especially important in order to achieve a result that complies with the expected measurement uncertainty. For this reason, it has to be clear where the responsibility of the client (of the analysis) ends and that of the laboratory begins. The laboratory operates on the basis of a working week of five days, from Monday till Friday. For the preservation periods of the samples, the clock will not stop at the weekend, in other words, it runs on 24 hours a day, 7 days a week.

Remark

With the term (analysis) client the party is referred to that commissions the laboratory to perform analyses (this means, it does not refer to, for instance, a "hidden" client of a consultancy firm). In the context of this document, it may also be a party acting in the name of this client (of the analysis), such as a sample taker.

With the expiration of the preservation period, it ought to be taken into account that either re-analyses or conformation investigations might be necessary. Re-analyses are inevitable in any laboratory and may have various different causes. Of course, a laboratory will strive to have any necessary re-analyses started and the contents secured, before the preservation period expires. It is not always possible to perform this. Since re-analyses are inherent to the laboratory process and in more than just an exceptional case may cause the exceeding of the preservation period, the laboratory will not be able to definitely guarantee the analysis being performed the preservation period. This is a case of force majeure. A force majeure concerns all those elements that are intrinsically linked to the laboratory procedure.

For a conformation investigation as an additional investigation, by which the results of a standard investigation are confirmed or for which separation of sum parameters or decomposition of a composite sample is required, the same limitations as in re-analyses apply.

Especially parameters with a short preservation period of four days or less, are particularly critical. For parameters with a preservation period of only 1 day, this is obvious, for parameters with preservation period of 2 to 4 days, the preservation period is critical as well, if the sampling takes place at the end of the week. For this reason, it is of prime importance that samples are delivered in time and that the delivery of samples with a short preservation period is announced in advance.

Process safeguarding of the preservation period

In order to meet the requirements of the preservation period, agreements between the laboratories and (analysis) clients is of utmost importance. The parties start from the following agreements:

- All samples should be delivered at the laboratory the latest one day after sampling.
 - Samples with parameters, the preservation period of which is less than two days, should be delivered on the day of sampling.
- If samples with parameters with a the preservation period of two or three days have been taken on Thursday or Friday, they should be delivered on the day of sampling.

Day 0 Date of sampling

Day 1 Date of transfer of the samples to the laboratory.

Over and above this, the following core conditions apply:

- The assignment has been completely accepted (the laboratory has received from the (analysis) client all information it needs).
- The samples comply with the technical specifications with regard to packaging and preservation.
- The capacity must be sufficient (assessment on day 2, unless the assignment has been announced).
- The matrix and analysis are available in the packaging offered by the laboratory in question.
 - Parameters, of which the preservation period is shorter than or equals four days, have been announced in advance.
 - The acceptance conditions that specifically apply to the laboratory concerned, must have been complied with.

Remark

In exceptional cases, analyses have to be executed within a few hours after sampling. In these cases separate agreements are made between the (analysis) client and the laboratory.

If the above main conditions are met, the laboratory takes care of securing the contents within the preservation period(s).

If the sample is delivered later than the intended day, the laboratory will not be liable for the consequences of the possible expiration of the preservation periods.

The (analysis) client should assess possible consequences of too late a delivery of the samples and alter the desired delivery time accordingly.

Preservation period

This term refers to the time that elapsed between sampling (immediately followed by preservation) and the moment of securing the contents. The term has been adopted from NEN-EN-ISO 5667-3:1996 (in this standard both the term preservation time and preservation period are being used).

If a sample is taken within one calendar day, it is obvious that the date of sampling is the date of that specific day. This is also the date, on which the preservation period starts. If a composite sample is prepared from samples taken that same day, it is also obvious that this will be the date of sampling and that this is also the date, on which the preservation period starts.

Sampling proportional to time or proportional to volume, during which one physical sample is obtained, like in wastewater sampling or sampling from flows of material, often include more than one calendar day. The date, on which the preservation period starts, is the date of the last day of sampling. This date will be passed on to the laboratory as the date of sampling.

Remark

Time integrated measurements are not advisable, if the period of time over which the sampling takes place will exceed the preservation period(s) of the intended analyses. In those cases, the interim securing of the contents of the first subsamples can be resorted to.

The moment the preservation period starts: composite samples

In case of a composite sample prepared from samples taken on different calendar days, the date on which the preservation period starts, is the date of the oldest sample taken. This date will be passed on to the laboratory as the date of sampling.

Remark

The day of sampling is taken as day 0. This means that a preservation period of 2 days will expire at the end of day 2. (The preservation period of a sample taken on Monday expires on Wednesday. The preservation period of a sample taken on Friday lasts until and including Sunday).

Securing the content

The moment during sample pretreatment or during analysis on which the sample has undergone such "treatment", that the contents of the analytes will not change any more.

Explanatory note:

During the preservation period, for example, the chemical- or microbiological decomposition processes are (strongly) inhibited. From the moment of securing the content, the processes that may lead to a change of the content must be excluded as much as possible.

An example of a moment of securing the content is the addition of the extraction solvent to a soil sample in mineral oil determination. Another example is the addition of a bichromate solution in the CSV determination. In case of a sulphate analysis with ionchromatography, the moment of the securing of the content is only the moment the measurement has been executed.

The prescribed preservation procedures consisting of package, preservative, storage circumstances and the preservation period for each parameter, are mentioned in Protocol SIKB 3001 "Methods of Preservation and Preservation periods for Environmental Samples". The most recent version is available on www.sikb.nl; this version is binding.

1.1.3 Transfer of samples and sample data, transport and storage of samples

Transfer of samples

During the period of time between sampling and the acceptance of the samples by the laboratory, the samples are stored and transported after having been prepared according to the guidelines of SIKB Protocol 3001. If during this period the samples change the "owner", this will be noted on a sample-transfer form. During the transfer, the amount of samples (number of receptacles/bottles) is established. In case any samples (or receptacles/bottles) are missing as compared to the number given by the sampletaker, this will be recorded on the form. The individual samples should be coded uniquely (e.g. bar code, project – and sample description)

Transfer of sample data

The transfer of sample data occurs in two ways: directly, if the sampletaker takes them to the laboratory, or indirectly, via the advisor or the client. This may be either electronically or on paper.

In this process, a number of possible problems can lead to a standard remark by the laboratory, a so-called disclaimer:

- First of all, the delivery of the data by the intermediary may be too late, so that the preservation period expires. For this reason it is important that the person responsible for sending the sample data to the laboratory can be reached.
- Every transfer of the sample data may present a possible extra source of error. Missing data may lead to a disclaimer.
- The delivery of recognisably false data also leads to a disclaimer. In theory, corrective measurements are sometimes possible, but these often lead to the exceeding of the maximum preservation periods and have a very disturbing influence on the process. The delivery of unclear or provisional data is therefore most undesirable.

Some attention points are:

Unambiguous (bar) codes, unambiguous commissioning, right research environment, matrix, analysis etc.

Interim storage of samples

By interim storage of samples the short-term storage, prior to transportation to the laboratory, is referred to. This may take place on the location where the samples have been taken or in a central location where the samples are stored up. The responsibility of the storage conditions will generally speaking ly with the sampletaker.

Remark

If the storage is not short-term, the circumstances of storing should be according to what has been mentioned in connection with the transportation of samples.

If according to the Protocol SIKB 3001 cooling is required for preservation, samples are stored in a mechanically cooled room or are packed in a cooling box. The samples must to be stored at a temperature of 2 to 5 °C.

The desired low temperature cannot be realized continuously in the room and in the samples. On the one hand, by opening the cooled room or by placing samples with a higher temperature in it, the room temperature may rise. On the other hand, the newly added samples with a higher temperature will not immediately adopt the desired lower temperature. In both cases, after a period of time, the conditions will stabilize within the desired temperature range for both the samples and the room.

When a mechanically cooled room is made use of, it has to be established, whether it has operated during this period. Suitability of a room and/or the package can be established (once or at random) by a validation under normal conditions of usage. In this case, continuous measuring is not necessary.

Remark

The volume of the sample (e.g. an AP04 sample) will be not present a reason to deviate from what has been said above with regard to storage.

Transport of samples

With the notion transport of samples reference is made to both the transportation from the location of sampling to a central store room (the responsibility for which lies with the sampletaker) and the transportation to the laboratory. In latter case, the sampletaker is responsible for the transport conditions, unless the transportation is taken care of by a transporter of the laboratory.

As described under the interim storage of samples, samples must be transported in a conditioned state.

Remark

The volume of the sample (e.g. an AP04 sample) will be not be a reason to deviate from what has been said above with regard to storage.

Storage of samples in the laboratory

During storage in the laboratory, the samples will be preserved cooled at a temperature of 1 to 5 °C. The responsibility for the storage conditions rests with the laboratory.

1.1.4 Acceptance of assignments by the laboratory

Starting points

With the acceptance of the assignment, it has to be established whether the guidelines mentioned in Protocol SIKB 3001 and any additional agreements in connection with the preservation period have been fulfilled.

By (assignment)acceptance reference is made to:

The subprocess at the moment in which the laboratory has received all samples as well as the complete assignment (including sample data) from the (analysis) client on the location of the laboratory. Only from the moment of acceptance onwards, the laboratory can start with the sample pretreatment – and with analysis performances to secure the content.

Remark

The precise nature of the subprocess may be different for each laboratory. Usually, the introduction and/or the release of data in LIMS will be part of it.

Remark

If the (analysis) client transfers the samples to a courier of the laboratory, from that moment onwards, the laboratory is responsible for the samples (with respect to transport- and storage conditions).

The laboratory will start the research at all times, when both assignment and samples are complete, and when it may take for granted that the (analysis) client knows the preservation guidelines.

Date of sampling

The (analyses) client guarantees that the sample will be delivered to the laboratory. The laboratory will not check on the date of sampling and will not make its performances depend on the date of sampling.

With respect to the samples presented, the laboratory determines whether the research can be conducted within the period of time set for the laboratory (i.e. the preservation period or the preservation period minus one day).

If the date of sampling is unknown, the standard remark will be recorded in the report. A note will be added, that this arises from the fact that the date of sampling is unknown.

After reporting, this may lead to requests by (analysis) clients to enter a sampling date as yet, and to generate another report (without the standard remark). The laboratory will only perform this, after the (analysis) client has sent in a written confirmation. It will be made clear by a remark that it concerns a corrected report.

Applied preservation

If according to the specifications of (analysis) client the samples have been preserved according to Protocol SIKB 3001, the sample will be treated as such.

If according to the specifications of the (analysis) client, the sample has not been preserved, the laboratory will carry this out as yet.

Remark

The delivery - according to the (analysis) client - in a non-preserved condition and the preservation in the laboratory (by adding the preservative in the laboratory), is to be recorded in the report of the laboratory.

The laboratory will not check, whether preservation has been applied, it will go by the information of the responsible party, i.e. the (analysis) client. If information about the preservation is lacking, the sample will be treated as such (no preservation performances will be conducted).

If the date of sampling is unknown, the standard remark will be recorded in the report. A note will be added, that this arises from the fact that the date of sampling is unknown.

After reporting, this may lead to requests by (analysis) clients to assume, as yet, that the sample has been preserved, and to generate another report (without the standard remark).

The laboratory will only carry this out, after the (analysis) client has sent in a written confirmation of this. In the report it will be recorded by a remark that it concerns a corrected report.

Remark

If no information about the state of preservation of the samples is delivered, but if bottles have been used that were provided by the laboratory and supplied with a preservative, it will be assumed that the samples have been preserved prior to delivery.

Necessary on-site performances and aim of the investigation

The client is responsible of the delivery of all relevant information for the execution of the investigation, so that the aim of the investigation will be sufficiently clear to the laboratory. This concerns the right choices the laboratory has to make regarding pretreatment and parameters.

Appendix 2 Regulations for the use of the Accreditation Mark 'Quality Assurance of Soil Management SIKB'

The accreditation mark 'Quality Assurance of Soil Management SIKB', hereafter called "the accreditation mark", has been developed to create for all parties involved a clear guideline for the quality assurance of activities in soil management, including 'Laboratory Analyses for Environmental Soil Research'.

The accreditation mark and the supervision of the right application of the accreditation mark is managed for the accreditation scheme AS SIKB 3000 by the Accreditation Council for Soil Management, which operates under the aegis of the Foundation Infrastructure for Quality Assurance of Soil Management (Dutch: SIKB, Stichting Infrastructuur Kwaliteitsborging Bodembeheer). The authorized accreditation institutions supervise the correct use of the accreditation mark during their audits at the accredited organisations.

Only laboratories that have been accredited for analyses described in the accreditation scheme AS SIKB 3000 and have also paid their annual contribution to the SIKB that results thereof, are allowed to use the accreditation mark. These laboratories obtain the right to use the quality mark:

- on reports on laboratory analyses for environmental soil research, provided that the analyses for the investigation concerned have been executed completely under accreditation.
- on letter-paper, provided that the letter does not make mention of laboratory analyses for environmental soil research that have not been or will not be executed under accreditation.
- in general (including for promotional purposes, company presentations), if this documentation:
 - also refers to laboratory analyses for environmental soil research that have been or will be executed under accreditation and
 - refers in no way whatsoever to laboratory analyses for environmental soil research that have not been or will not be executed under accreditation.

If a document refers to several analyses, only part of which are or will be executed under accreditation, the quality mark may be applied in the document only in such a way that it is absolutely clear, which of the analyses have been executed under accreditation.

With "under accreditation" is meant "in conformity with the requirements described in AS SIKB 3000, 'Laboratory analyses for environmental soil research' and the related protocols". For the executing organisation that version of these documents applies, which is in force at the time of the execution of the analyses.

When the quality mark is made use of, directly under the logo incorporating the text 'Quality Assurance for Soil Management SIKB', the relevant scope for the specific situation must always be legibly included, e.g. by stating 'AS SIKB 3000'.

Sanctions may be instituted against companies or institutions that infringe upon the prescribed use of the accreditation mark. Such a sanction may be the loss of the qualification to use the accreditation mark.

The accreditation mark is a registered trademark. Any misuse of the accreditation mark may be prosecuted under private law.

Appendix 3 Data Interlaboratory Investigation Fenelab Sample

As is apparent from the documentation of Fenelab, in the Fenelab samples the following concentrations have been reported. In the table below, the results of the third quarter of 2003 are presented.

PD152b Quarterly Report

2003-08-01 till 2003-10-31

Name of Parameter	Unit	Average	Pooled RSD _L	APO4 eis RSD _L	RSD _R	FeNeLab eis RSD _R	Meets FeNeLab criteria RSD _R	n	Lab s	Median	M AD	M 95%
Clay	% (m/m ds)	28.3	11.0%	10%	11.3%	12.7%	yes	428	9	28.4	4.9%	222.0-34.7
Organic Matter	% (m/m ds)	10.6	6.2%	10%	10.8%	12.7%	yes	407	8	10.6	5.7%	8.4-12.8
Arsenic	mg/kg ds	43.0	5.2%	10%	7.3%	12.7%	yes	458	9	43.0	4.7%	36.9-49.2
Cadmium	mg/kg ds	8.1	4.7%	10%	5.4%	12.7%	yes	424	9	8.1	3.7%	7.2-8.9
Chromium	mg/kg ds	181	5.0%	15%	6.5%	19.1%	yes	459	9	180	5.6%	158-204
Copper	mg/kg ds	160	4.9%	10%	6.5%	12.7%	yes	459	9	160	4.9%	140-180
Mercury	mg/kg ds	3.94	6.7%	15%	8.5%	19.1%	yes	452	9	3.95	5.2%	3.28-4.60
Lead	mg/kg ds	273	6.5%	10%	9.4%	12.7%	yes	459	9	270	7.4%	221-324
Nickel	mg/kg ds	52.2	21.9%	15%	22.6%	19.1%	no	459	9	52.0	3.8%	28.7-75.8
Zinc	mg/kg ds	974	6.1%	10%	6.8%	12.7%	yes	459	9	960	2.8%	842-1106
EOX	mg/kg ds	3.16	11.7%	20%	14.5%	25.4%	yes	381	8	3.20	9.4%	2.25-4.06
Pak-total (sum of VROM)	mg/kg ds	9.7	6.6%	15%	9.7%	19.1%	yes	436	9	9.8	6.1%	7.9-11.6
Hexachlorobenzene	mg/kg ds	0.175	11.7%	15%	15.9%	19.1%	yes	315	6	0.172	10.5%	0.122-0.228
PCB-total (sum 7)	mg/kg ds	0.398	13.2%	20%	16.2%	25.4%	yes	315	6	0.400	10.0%	0.273-0.523
DDT/DDD/DDE (sum)	mg/kg ds	0.053	66%	20%	80%	25.4%	no	253	5	0.049	31%	0.000-0.135
Cyanide	mg/kg ds	10.9	14.1%	15%	14.1%	19.1%	yes	205	5	11.0	6.4%	7.8-14.0
Barium	mg/kg ds	821	5.8%	10%	8.0%	12.7%	yes	371	7	820	4.9%	695-948
Cobalt	mg/kg ds	18.2	5.4%	10%	6.3%	12.7%	yes	378	7	18.0	5.6%	16.0-20.5
Mineral oil	mg/kg ds	308	11.0%	15%	13.4%	19.1%	yes	470	9	310	6.5%	225-391

Explanation of Table

Average	The mentioned averages are the averages of all observations
Pooled RSD _L	The "average" relative intra-laboratory reproducibility standard deviation
APO4 eis RSD _L	The required relative intra-laboratory reproducibility standard deviation as determined in APO4
RSD _R	The relative inter-laboratory reproducibility standard deviation
FeNeLab eis RSD _R	Starting from the premise that there is a more or less constant relationship between the inter-laboratory reproducibility and the intra-laboratory reproducibility, from the results of the first quarter of the year 2000 for each parameter the relationship between the RSD _R and the pooled RSD _L was calculated. This value varied from 1,08 (EOX) to 1,56 (Cr), with an average of 1,27 (the results for alfa-Endosulfan and HCH have here been left out of consideration. This average value, multiplied by the requirement (eis) made by APO4 to RSD _L , for each parameter results to a value that could serve as requirement (eis) for the RSD _R . (Note: The average value for this quarter of a year is 1,27)
Median	In order to calculate the median, the results are arranged. In an odd number of results, the median is the middlemost of the series. In an even number of results this is the average of the two middlemost results. The value of a median is less being influenced by the extremes than by the averages.

MAD %	The relative “median of absolute deviations (MAD)”. The MAD is the median of the absolute values of the results minus the median.
n	The total number of observations
Labs	The total number of participating laboratories
M 95%	The 95% reliability interval

Appendix 4 List of Analyses and the related Protocols

Table IV.1 Subdivision of organic matter into the related protocols

Name	Abbreviation	Protocol
1,1,1-Trichloroethane		3030
1,1,2-Trichloroethane		3030
1,1-Dichloroethane		3030
1,1-Dichloroethene		3030
1,2,3,4-Tetrachlorobenzene		3020
1,2,3,5-Tetrachlorobenzene		3020
1,2,3-Trichlorobenzene		3020 / 3030
1,2,4,5-Tetrachlorobenzene		3020
1,2,4-Trichlorobenzene		3020 / 3030
1,2-Dichlorobenzene		3030
1,2-Dichloroethane		3030
1,3,5-Trichlorobenzene		3020 / 3030
1,3-Dichlorobenzene		3030
1,4-Dichlorobenzene		3030
2,3,4,2'4'5'-hexachlorobiphenyl	PCB 138	3020
2,3,4,5-2'4'5'-heptachlorobiphenyl	PCB 180	3020
2,4,5,2'4'5'-hexachlorobiphenyl	PCB 153	3020
2,4,5,2'5'-pentachlorobiphenyl	PCB 101	3020
2,4,5,3'4'-pentachlorobiphenyl	PCB 118	3020
2,4-4'-trichlorobiphenyl	PCB 28	3020
2,5-2'5'-tetrachlorobiphenyl	PCB 52	3020
Aldrin		3020
Antracene		3010
Benzene		3030
Benzo(a)antracene		3010
Benzo(a)pyrene		3010
Benzo(ghi)perylene		3010
Benzo(k)fluorantene		3010
Chlordane (cis & trans)		3020
Chrysene		3010
cis-1,2-Dichloroethene		3030
cis-Heptachlor epoxide		3020
Dichloromethane	DCM	3030
Dieldrin		3020
Endrin		3020
EOX		3010
Ethylbenzene		3030
Phenantrene		3010
Fluorantrene		3010
Heptachlor		3020
Hexachlorobenzene		3020

Indeno(1,2,3,c,d)pyrene		3010
Isodrin		3020
Mineral oil	MO	3010
Monochlorobenzene		3030
m-Xylene		3030
Naphtalene		3010
o,p'-DDD		3020
o,p'-DDE		3020
o,p'-DDT		3020
Organic matter content	OM	3010
o-Xylene		3030
p,p'-DDD		3020
p,p'-DDE		3020
p,p'-DDT		3020
PAH 10-VROM		3010
Pentachlorobenzene		3020
p-Xylene		3030
Styrene		3030
Telodrin		3020
Tetrachloroethene		3030
Tetrachloromethane		3030
Toluene		3030
trans 1,2-Dichloroethene		3030
trans-Heptachlor epoxide		3020
Trichloroethene		3030
Trichloromethane		3030
α-Endosulfan		3020
α-Hexachlorocyclohexane	A-HCH	3020
β-Hexachlorocyclohexane	B-HCH	3020
γ-Hexachlorocyclohexane	Γ-HCH	3020

Table IV.2 Subdivision of anorganic matter into the related protocols

Antimony	Sb	3010
Arsenic	As	3010
Barium	Ba	3010
Beryllium	Be	3050
Bromine	Br	3040
Cadmium	Cd	3010
Calcium carbonate	CaCO ₃	3010
Chlorine	Cl	3040
Chromium	Cr	3010
Cyanide (total)	CN total	3040
Cyanide (free)	CN free	3040
Dry matter	DM	3010
Conductivity	EC	3010
Cobalt	Co	3010
Copper	Cu	3010
Mercury (non-volatile)	Hg	3010
Lead	Pb	3010
Clay	<2um	3010
Molybdenum	Mo	3010
Nickel	Ni	3010
pH	pH (CaCl ₂)	3010
Selenium	Se	3010
Sulphate	SO ₄	3040
Tellurium	Te	3050
Thallium	Tl	3050
Tin	Sn	3010
Vanadium	V	3010
Free iron		3010
Silver	Ag	3050
Zinc	Zn	3010