

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

SIKB – Protocol 3040

Additional III



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1 Place of this Protocol in AS SIKB 3000

SIKB-Protocol 3040 presented here, belongs to a group of protocols that have been specifically written for the laboratory work for the environmental investigation of soil samples. At the time of writing, this group consists of the following protocols:

- Protocol 3001: Preservation methods and preservation period of environmental samples
- Protocol 3010: Basic package, 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.
- Protocol 3020: Additional I determination of OCP, PCB and chlorobenzenes (tri- (optional), tetra-, penta-, en hexachlorobenzenes).
- Protocol 3030: Additional II, determination of benzene, toluene, ethylbenzene, (o/m/p) xylene, volatile halogenated hydrocarbons and chlorobenzenes (mono-, di- en trichlorobenzenes (optional)).
- Protocol 3040: Additional III, determination of bromide, chloride, sulphate and cyanide.
- Protocol 3050: Additional IV, determination of Ag, Be, Te and Tl

Protocol 3090, Additional V, determination of elements in soil that have not been described in other protocols – has been planned, but not yet written.



1.1 Technical Description and Quality Assurance of the related Performances

In the performance sheets presented below, for the described performances a distinction can be made between normal performances and supporting performances. For the normal performances, the quality assurance described in AS SIKB 3000 (Chapter 3), is valid. For the supporting performances, different requirements in respect of the quality assurance are in force. For the supporting performances, the laboratory does not have to participate in proficiency testing. With the start-up of a supporting performance, the laboratory must to prove that it has a sufficient control over the performance. In a number of cases, it is not necessary to establish the detection limit, e.g. for determining free-iron, as it concerns a determination which is only relevant for samples with a high level. The quality assurance is performed by means of (a) control chart(s). The determination of equivalence is performed by showing that the set performance characteristics are being complied with.



1.2 Performance sheets

1.2.1 Performance sheet Determination of anions in soil

Principle

The analysis sample is suspended in water. The suspension is shaken for 1 hour and subsequently filtered over a folding filter. In the filtrate, the ions are determined with the aid of an ionchromatograph according to NEN-EN-ISO 10304-2.

The analytes to be determined

name	CAS-number	soil/sediment(mg/kg.ds) target- value ¹⁾	intervention- value ¹⁾	AG _{r,eis}
Bromide		20		5
Chloride		200		50
Sulphate				50

¹⁾ The target- and intervention values are based on soil containing 10% organic matter and 25% clay.

PROCEDURE

Pretreatment

Draft-NEN 5709

Preservation

SIKB-protocol 3001

Reference method

VPR C85-06 and NEN-EN-ISO 10304-2 (measurement)

Related performance

Obligatory activities

Attention points

- stirring with water

- suppressor to reduce the electrical conductivity of the eluate if the determination is executed by measuring the conductivity.

- quality of the separation (resolution)

- cross-sensitivity at high concentrations of one of the anions

PERFORMANCE REQUIREMENTS

Detection limit

< AG_{r,eis} (see "The determined analytes" above)

Retrieval

Chloride 90 - 110 %

Remaining anions 85 - 110 %

(intralaboratory) reproducibility variation coefficient VC_w

Chloride < 7.5 %

Remaining anions < 10%



1.2.2 Performance sheet Determination of total inorganic fluoride in soil

Principle

The analysis sample is suspended in water. The suspension is stirred for 1 hour and subsequently filtered over a folding filter. The filtrate is subsequently potentiometrically analysed (NEN 6483). With this method both free and complex-bound fluoride is determined.

The analytes to be determined

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

¹⁾ The target value is based on soil containing 2% organic matter and 0% clay.

²⁾ The intervention value is based on soil containing 10% organic matter and 25% clay

PROCEDURE

Pretreatment

Preservation

Reference method

Related performance

Obligatory activities

Attention points

Draft-NEN 5709

SIKB-Protocol 3001

NEN 6483 or NEN 6589 (measuring)

- potentiometric measuring

- application of tisab reagents

- condition of the electrode

PERFORMANCE REQUIREMENTS

Detection limit

Retrieval

(intralaboratory) reproducibility variation coefficient VC_w

Fluoride

< AG_{r,eis} (see "The determined analytes" above)

70-110%

< 10%



1.2.3 Performance sheet Determination of cyanides (free and total) in soil

Principle

Pretreatment of the soil sample

The analysis sample is extracted with a 2,5 mol/l sodiumhydroxide-solution. The extract is diluted 100 times, after which analysis of cyanide total and/or cyanide free is performed.

Determination of the total cyanide content

Complex-bound cyanide is digested with a pH of 3,8 by the action of UV-light. An UV-B-lamp and a digestion-spiral of borosilicate glass is employed. UV-light with a wavelength smaller than 290 nm is filtered away. Subsequently, cyanehydrogen that becomes available at a pH of 3,8 and a temperature of 125 °C, is overdistilled and determined photometrically.

Determination of the free cyanide content

Prior to the distillation, a zincsulphate solution is added to the sampleflow, through which the ironcyanides present precipitate as zinccyanoferrate complex. Subsequently, cyanehydrogen that becomes available at a pH of 3,8 and a temperature of 125 °C, is overdistilled and determined photometrically.

The analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		AG _{r,eis}
		target-value ¹⁾	intervention-value ¹⁾	
Cyanide free		1	20	1 ²⁾
Cyanide total		5	50	1

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

2) For this component, with the reference method a lower detection limit is not possible.

PROCEDURE

Pretreatment

Draft-NEN 5709

Preservation

SIKB-Protocol 3001

Reference method

NEN 6655

Related performance

Obligatory activities

Attention points

none

- UV-B lamp and a digestion spiral (borosilicate glass)

- pH max. 3.8

- the R of cyanoferrate must in average be 90%; individual determinations may be lower

PERFORMANCE REQUIREMENTS

Detection limit

< AG_{r,eis} (see "The determined analytes" above)

Retrieval

Free cyanide

K₃Fe(CN)₆ 0 - 7 %

Thiocyanate

< 5 µg/l (starting from 1000 µg/l in the extract)

KCN

80 - 110 %

Total cyanide

K₃Fe(CN)₆ 80 - 110 %

Thiocyanate

< 10 µg/l (starting from 1000 µg/l in the extract)

(intralaboratory) reproducibility variation coefficient VC_w

Free cyanide

< 15 %

Total cyanide

< 15 %



2 Literature

Inorganic parameters

NEN 6483	1982	Water – Potentiometric determination of the total fluoride content.
NEN 6589	1990	Rain water – Potentiometric determination of the total inorganic fluoride content by flow analysis
NEN 6655	1997	Water and soil – Photometric determination of the content of total and free cyanide by continuous flow analysis.
NEN-EN-ISO 10304-2	1996	Determination of dissolved anions by liquid chromatography of ions. - Part 2: Determination of Bromide, Chloride, Nitrate, Nitrite, ortho-Phosphate and Sulphate in waste water.
VPR C85-03	1985	Interim code of practice soil – ground water and soil, clean-up and analysis – Fluoride.
VPR C85-06	1985	Interim code of practice soil – ground water and soil, clean-up and analysis – Bromide (dissolved c.q. soluble).

General

Draft-NEN 5709	2004	Soil – Sample pretreatment for the determination of organic and inorganic parameters in soil.
NEN 5740	1999	Soil – Investigation strategy for exploratory survey of soil.
NEN 5861	1999	Environment – Procedures for the transfer of samples.
NPR 6603	1988	Water and sludge – Guidelines for internal quality control with control charts in chemical analyses.
NEN 7777	2003	Environment – Performance characteristics of measurement methods
NEN 7778	2003	Environment – Equivalence of measurement methods.
NEN-EN-ISO/IEC 17025	2000	General requirements for the competence of testing and calibration laboratories.

