

Accreditation Scheme
Laboratory Analyses for
Environmental Soil Investigation

SIKB – Protocol 3020

Additional I



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1 Place of this Protocol in AS SIKB 3000

SIKB-Protocol 3020 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 moment 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 sheet 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 has 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 samples only with a high level. The quality assurance is performed by means of (a) control chart(s). The determination of equivalence is carried out by showing that the set performance characteristics are being complied with.

If a laboratory wants to add this protocol to the accreditation, only for those chlorobenzenes (performance sheet 2.2.2) a validation is executed that are determined as being moderately volatile, i.e.: tetrachloro-, pentachloro-, hexachlorobenzene and optionally the trichlorobenzenes. The remaining chlorobenzenes can be accredited according to SIKB Protocol 3030.

1.2 Performance sheets

1.2.1 Performance sheet Determination of Polychlorinated Biphenyls (PCB) and Organochloride Pesticides (OCP) in soil

Principle

The analysis sample is extracted with acetone/petroleum ether, which is followed by a procedure according to NEN-EN-ISO 10382. The obtained extract will be eluted over an aluminumoxide column. Subsequently, the determination is executed with the employment of a gas chromatographic instrument, equipped with an electron-capture detector (ECD) or a mass spectrometer (MS). With the use of an ECD, additional confirmation must be executed, for example by using a double column gas chromatographic instrument.

If the chromatographic separation is insufficient or if there are interfering compounds present, the extract can be divided and purified further with a silicagel column.

The analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		AG _{r,eis}
		target-value ²⁾	intervention-value ³⁾	
PCB 28 (2,4-4'-trichlorobiphenyl)	7012-37-5			0,002
PCB 52 (2,5-2'5'- tetrachlorobiphenyl)	35693-99-3			0,002
PCB 101 (2,4,5,2',5'-pentaCB)	37680-37-2			0,002
PCB 138 (2,3,4,2'4'5'-hexaCB)	35065-28-2			0,002
PCB 153 (2,4,5,2'4'5'-hexaCB)	35065-27-1			0,002
PCB 180 (2,3,4,5-2'4'5'-heptaCB)	35065-29-3			0,002
PCB 118 (2,4,5,3'4'-pentaCB)	31508-00-6			0,002
Sum of 7 polychlorinated biphenyls		0,004		1
α-Hexachlorocyclohexane (α-HCH)	319-84-6			0,001
β-Hexachlorocyclohexane (β-HCH)	319-85-7			0,001
γ-Hexachlorocyclohexane (γ-HCH)	58-89-9			0,001
Sum of 3 HCH's		0,002		2
Aldrin	390-00-2			0,001
Dieldrin	60-57-1			0,001
Endrin	72-20-8			0,001
Sum of 3 drin's		0,001		4
p,p'-DDE	72-54-9	-		0,001
o,p'-DDD	53-19-0	-		0,001
o,p'-DDT	784-02-6	-		0,001
p,p'-DDD	72-54-8	-		0,001
o,p'-DDE	3424-82-6	-		0,001
p,p'-DDT	50-29-3	-		0,003
Sum of 6 DDT,DDE,DDD		0,002		4
Heptachlor	76-44-8	0,0005		0,001
α-Endosulfan	959-98-7	0,0005		0,001
cis-Heptachlor epoxide	280044-83-9	-		
trans-Heptachlor epoxide	1024-5703	-		0,001
Sum of 2 heptachlor epoxide		0,0005		
Telodrin ¹⁾	297-78-6			
Isodrin ¹⁾	465-73-6			
Chlordane (cis & trans) ¹⁾	87-66-3	0,002	-	0,001

¹⁾ This component has not been validated for the related standard.

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

³⁾ The intervention value is based on soil containing 10% organic matter and 25% clay.

Procedure

Pretreatment:

Draft-NEN 5709

Preservation:

SIKB-Protocol 3001

Reference method:

NEN-EN-ISO 10382

Related performance

Obligatory activities

- acetone as digestive
- confirmation by using ECD
- internal standard
- desulphurization
- aluminumoxide purification
- silica purification

Attention points:

Performance requirements

Detection limit

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

Retrieval

α-endosulfan

60 - 110%

Other

75 - 110%

(intralaboratory) reproducibility variation coefficient VC_w

All

< 25%

Components, the performance of which must be monitored by control charts

PCB 52, α-endosulfan, α-HCH, β-HCH

Components for which blind testing or proficiency testing must be executed

PCB 28, α-endosulfan, α-HCH, β-HCH, p,p'-DDT

Periodical validation:

All components

1.2.2 Performance sheet Determination of the content of chlorobenzenes in soil

Principle

The analysis sample is extracted with acetone/petroleum ether (NEN-EN-ISO 10382).

With the analysis according to NEN-EN-ISO 10382, the extract is eluted over an aluminumoxide column. Subsequently, the determination is executed with the aid of a gas chromatographic instrument equipped with an electron-capture detector (ECD) or a mass spectrometer (MS). With the use of an ECD, additional confirmation must be executed, for example by using a double column gas chromatographic instrument. If the chromatographic separation is insufficient or if there are interfering components present, the extract can be divided and purified further with a silicagel column.

The analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		AG _{r,eis}
		target-value ¹⁾	intervention-value ²⁾	
1,2,3-Trichlorobenzene	87-61-6	-		0,003
1,2,4-Trichlorobenzene	120-82-1	-		0,003
1,3,5-Trichlorobenzene	108-70-3	-		0,003
Sum trichlorobenzenes		0,002		
1,2,3,4-Tetrachlorobenzene	634-66-2	-		0,001
1,2,3,5-Tetrachlorobenzene	634-90-2	-		0,001
1,2,4,5-Tetrachlorobenzene	95-94-3	-		0,001
Sum tetrachlorobenzenes		0,002		
Pentachlorobenzene	608-93-5	0,0005		0,0004 ³⁾
Hexachlorobenzene	188-74-1	0,0005		0,0004 ³⁾
Sum Chlorobenzenes		0.006	30	

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.

3) For this component a lower analytical limit is not possible with the reference method.

Procedure

Pretreatment:

Draft-NEN 5709

Preservation:

SIKB-protocol 3001

Reference method:

NEN-EN-ISO 10382

Obligatory performances - acetone as digestive
- by using electron-capture detection (ECD), a double column capillary gas chromatographic instrument has to be employed for confirmation.

Attention points:
- internal standard
- desulphurization
- aluminumoxide purification
- silica purification

Performance requirements

Detection limit < AG_{r,eis} (see "The det..anal.")

Retrieval

trichlorobenzenes	60 - 110%
Tetrachlorobenzene	60 - 110%
Pentachlorobenzene	60 - 110%
Hexachlorobenzene	60-110%

(intralaboratory) reproducibility variation coefficient VC_w
All < 25%

Components, the performance of which must be monitored by control charts
Trichlorobenzene, HCB

Components for which blind testing or proficiency testing must be executed
Trichlorobenzene, HCB

Periodical validation:
All components

2 Literature

Organic parameters

- 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
- NPR 6405 1995 Gas chromatography – General guidelines.
- NVN 7313 1995 Leaching characteristics of solid earthy and stony building and waste materials - Sample pretreatment - Sample pretreatment for leaching tests and the analysis of organic components
- 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.
- NEN-EN-ISO 10301 1997 Water quality – Determination of highly volatile halogenated hydrocarbons – Gas chromatographic methods.
- NEN-EN-ISO 10382 2003 Soil quality – Determination of organochloride pesticides and polychlorinated biphenyls – Gas chromatographic method with electron capture detection.
- ISO 11465 1993 Soil quality - Determination of dry matter and water content on a mass basis - Gravimetric method
- ISO 13530/TR 2003 Water quality – guide to analytical quality control for water analysis

General

Draft-NEN 57092004 Soil – Sample pretreatment for the determination of organic and inorganic parameters in soil.

NEN 5740 1999 Soil – Investigation strategy for exploratory survey.

NEN 5861 1999 Environment – Procedure of 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 – Equivalency of measurement methods.

NEN-EN-ISO/IEC 17025 2000 General requirements for the competence of testing and calibration laboratories.