

Accreditation Scheme
Laboratory Analyses for
Environmental Soil Investigation

SIKB – Protocol 3030

Additional II



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1 Place of this Protocol in AS SIKB 3000

The present SIKB-Protocol 3030 belongs to a group of protocols, 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 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 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.

If a laboratory wants to extend the accreditation with this protocol, for the chlorobenzenes (performance sheet 2.2.2) a validation is executed only for those chlorobenzenes that will be determined as volatile compounds (monochloro-, dichlorobenzene and optionally the trichlorobenzenes). The remaining chlorobenzenes can be accredited under SIKB-Protocol 3020.



1.2 Performance sheets

1.2.1 Performance sheet Determination of volatile aromatics and volatile chlorohydrocarbons in soil

Principle

The analysis sample is extracted with methanol. Part of the methanol-extract is diluted with water. The volatile compounds are expelled with nitrogen and subsequently captured by an absorbent or a cold trap. The absorbed compounds are subsequently thermally desorbed, followed by the determination with a gas chromatographic instrument connected to a flame ionization detector (FID) and a electron-capture detector (ECD) or connected to a mass spectrometer (MS). When using a FID or a ECD, additional confirmation is to be executed.

By way of an alternative, the concentrations can also be determined with the aid of a so-called head-space-method coupled to an appropriate detector, such as a FID, an ECD or a MS (NEN-EN-ISO 10301 and NEN-EN-ISO 11423-1). When a FID or an ECD is made use of, additional confirmation is to be executed.

The analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds) target- value ¹⁾	intervention- value ²⁾	AG _{r,eis}
Volatile aromatic hydrocarbons				
Benzene	71-43-2	0,010	1,0	0,050 ³⁾
Toluene	108-88-3	0,010	130	0,100 ³⁾
Ethylbenzene	100-41-4	0,010	50	0,050 ³⁾
o-Xylene	95-47-6	-	-	0,100
m-Xylene	108-38-3	-	-	0,100
p-Xylene	106-42-5	-	-	0,100
Sum of xylenes		0,020	25	
Styrene	100-42-5	0,060	100	0,100 ³⁾
Volatile halogenated hydrocarbons				
Dichloromethane (DCM)	75-09-2	0.08	20	0,500
Trichloromethane	67-66-3	0,004	10	0,050 ³⁾
Tetrachloromethane	56-23-5	0,08	1	0,050 ³⁾
Trichloroethene	79-01-6	0,02	60	0,050 ³⁾
Tetrachloroethene	127-18-4	0,002	4	0,010 ³⁾
1,1-Dichloroethane	75-74-3	0.004	-	0,500
1,2-Dichloroethane	107-06-2	0.004	4	0,500
Sum dichloroethanes				
1,1-Dichlooretheen ⁴⁾	75-35-4	0.02	-	0,500 ³⁾
cis 1,2-Dichloroethene	156-59-2	-	-	0,500
trans 1,2-Dichloroethene	156-60-5	-	-	0,500
Sum of dichloroethenes		0.04		
1,1,1-Trichloroethane	79-01-6	0.014	-	0,050 ³⁾
1,1,2-Trichloroethane	79-00-5	0.08	-	0,050 ³⁾
Sum trichloroethanes				

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⁾ With the reference method, in respect of this component a lower detection limit is not possible.

⁴⁾ This component has not been validated for the realted standard.



Procedure

Pretreatment:

Draft-NEN 5709

Preservation:

SIKB-Protocol 3001

Reference method:

NEN-EN-ISO 15009, NVN 5872, NEN-EN-ISO 10301 and ISO 11423-1

Related performance

Obligatory activities

Attention points

- internal standard

- additional confirmation when using FID and ECD.

- ratio methanol / soil

- blank level of the components to be determined

Performance requirements

Detection limit

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

Retrieval

DCM

70 - 120%

Other

85 - 115%

(intralaboratory) reproducibility variation coefficient VC_w

All

< 15%

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

Benzene, 1,1-dichloroethane, dichloromethane and tetrachloromethane

Components for which blind testing or proficiency testing must be executed

Benzene, toluene, dichloromethane and tetrachloroethene

Periodical validation:

All components



1.2.2 Performance sheet Determination of the chlorobenzenes content in soil

Principle

The analysis sample is extracted with methanol (NEN-EN-ISO 15009) and/or with acetone/petroleum ether (NEN-EN-ISO 10382).

For the determination according to NEN-EN-ISO 15009, part of the methanol-extract is diluted with water. The compounds to be determined are subsequently expelled with nitrogen and captured with and absorbent or a cold trap. The absorbed compounds are subsequently thermally desorbed, after which the determination is performed employing a gas chromatographic instrument connected to a flame ionization detector (FID) and a electron-capture detector (ECD) or connected to a mass spectrometer (MS). When using a FID or an ECD, additional confirmation is to be executed.

By way of an alternative, the concentrations can also be determined with the aid of a so-called head-space-method coupled to an appropriate detector such as a FID, an ECD or a MS (NEN-EN-ISO 10301 and NEN-EN-ISO 11423-1). When using of a FID or an ECD, additional confirmation is to be executed.

The analytes to be determined

name	CAS-number	soil/sediment (mg/kg.ds)		AG _{r,eis}
		target-value ¹⁾	intervention-value ²⁾	
Monochlorobenzene	108-90-7	-	-	0,5
1,2-Dichlorobenzene	95-50-1	-	-	0,3
1,3-Dichlorobenzene	541-73-1	-	-	0,3
1,4-Dichlorobenzene	106-46-7	-	-	0,3
Sum dichlorobenzenes				
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				

- 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) In respect of the reference method, for this component a lower detection limit is not possible.

Procedure

Pretreatment:

Draft-NEN 5709

Preservation:

SIKB-Protocol 3001

Reference method:

Obligatory activities

NEN-EN-ISO 15009, NVN 5872, NEN-EN-ISO 10301 and ISO 11423-1
 - when using the electron-capture detection (ECD), a double column capillary gas chromatographic instrument has to be employed for confirmation.

- internal standard

Attention points

(volatile compounds)

- ratio methanol / soil

- blank level of the components to determine

(moderate volatile compounds):

- acetone as digestive

- desulphurization

- aluminumoxide purification

- silica purification



Performance requirements

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

Retrieval

Mono-, dichlorobenzene	85 - 110%
trichlorobenzenes	60 - 110%

(intralaboratory) reproducibility variation coefficient VC_w

All < 25%

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

Monochlorobenzene, trichlorobenzene

Compounds for which blind testing or proficiency testing must be executed

Monochlorobenzene, trichlorobenzene

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
NEN 5743	1995	Soil – Sampling of soil and sediments for the determination of volatile compounds.
NVN 5872	2003	Waste – Semiquantitative determination of volatile solvents in waste water samples using static head space analysis, gas chromatography and mass spectrometry.
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.
ISO 11423-1	1997	Water quality – Determination of benzene and some derivatives – Part 1: Head-space gas chromatographic method.
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
NEN-EN-ISO 15009	2002	Soil quality – Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons – Purge-and-trap methods with thermal desorption.



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.
NEN 5861	1999 Environment – Procedures for the transfer of samples.
NPR 6603	1988 Water and sludge – Guidelines for internal quality control by using control charts with 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.

