

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

**SIKB – Protocol 3050**

**Additional IV**



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## Table of Contents

<b>1 PLACE OF THIS PROTOCOL IN AS SIKB 3000 .....</b>	<b>4</b>
1.1 TECHNICAL DESCRIPTION AND QUALITY ASSURANCE OF THE RELATED PERFORMANCES .....	5
1.2 PERFORMANCE SHEETS .....	6
<b>1.2.1 Performance sheet Determination of Elements in Soil .....</b>	<b>6</b>
<b>2 LITERATURE .....</b>	<b>7</b>
GENERAL.....	7

## 1 Place of this Protocol in AS SIKB 3000

SIKB-Protocol 3050 presented here, belongs to a group of protocols that have been especially 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-CaCl<sub>2</sub>.
- 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.

Only part of the elements can be accredited under this protocol; these elements are: Ag, Be, Te and Tl. The remaining elements are accredited under SIKB Protocol 3010.

## 1.2 Performance sheets

### 1.2.1 Performance sheet Determination of Elements in Soil

#### Principle

The analysis sample is digested with nitric acid and hydrochloric acid (d-NEN 6961). Subsequently, the samples are analysed by a technique which is appropriate for the element.

#### Analytes to be determined

With the techniques available for the analysis of the element, usually several elements can be determined. An overview of these techniques is represented in the umbrella standard for soil analyses (NEN 6950). In the table below, for the elements mentioned in NEN 5740 the target- and intervention values and the requirement set to the analytical limit are represented

name	CAS-number	soil/sediment (mg/kg.ds)		intervention value <sup>2)</sup>	AG <sub>r,eis</sub>
		target value <sup>1)</sup>			
Beryllium <sup>3)</sup>	7440-41-7	0,29		30	0,1
Tellurium	13494-80-9	-	600	10	
Thallium <sup>3)</sup>	7440-28-0	1,0		15	3 <sup>4)</sup>
Silver	7440-22-4	-		15	1

- 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) The target value is based on the Regulation assessment of the purifiability soil remediation 2000  
 4) The detection limit is based on the application of ICP-AES. If a lower analytical limit is desired in connection with testing on target value level the choice can be made for the application of ICP-MS.

#### PROCEDURE

##### Pretreatment

Draft NEN 5709

##### Preservation

SIKB-protocol 3001

##### Reference method

Draft-NEN 6950 (umbrella standard soil); Ontw. NEN 6961 (aqua regia digestion);  
 Draft-NEN 6964 (GF-AAS); Draft-NEN 6965 (flame-AAS); Draft-NEN 6966 (ICP-AES); NEN-ISO 16772 (Hg); NEN 6426 (ICP), ISO 17294-2

##### Related performance

##### Obligatory activities

##### Attention points

- digestion with aqua regia
- relation between the temperature and the duration of the digestion
- with higher organic matter contents destruction losses must be prevented.
- ratio acid and absolute amount of organic matter

#### PERFORMANCE REQUIREMENTS

##### Detection limit

< AG<sub>r,eis</sub> (see above "The analytes to be determined")

##### Retrieval

All elements 80 - 110%

##### (intra laboratory) reproducibility variation coefficient VC<sub>w</sub>

All elements < 10 %

## 2 Literature

- Draft-NEN 5779 1994 Soil - Determination of mercury in soil with atomic absorption spectrometry (cold-vapour technique) after digestion with nitric acid and hydrochloric acid.
- Draft-NEN 6427 1999 Water – Determination of 66 elements by inductively coupled plasma emission spectrometry.
- Draft-NEN 6950 2003 Soil – Umbrella standard for the determination of 30 selected elements in soil.
- Draft-NEN 6961 2001 Digestion for the determination of 28 selected elements with aqua regia.
- o-NEN 6964 2001 Analyses of 28 selected elements - Atomic absorption spectrometry (graphite furnace technique)
- o-NEN 6965 2002 Analyses of 32 selected elements - Atomic absorption spectrometry (flame technique)
- o-NEN 6966 2003 Analyses of 30 selected elements - Atomic emission spectrometry with inductively coupled plasma.

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