
**Soil quality — Determination of
perchlorate in soil using liquid
chromatography-tandem mass
spectrometry (LC-MS/MS)**

*Qualité du sol — Détermination du perchlorate dans le sol par
chromatographie en phase liquide couplée à la spectrométrie de
masse en tandem (CL-SM/SM)*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3 *Chemical and physical characterization*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Although perchlorate (ClO_4^-) occurs naturally, it is mainly considered a manmade anion. Usually, it is combined with NH_4^+ , Na^+ , and K^+ to form ammonium perchlorate, potassium perchlorate, and sodium perchlorate, respectively. It has been reported that more than 90 % of manufactured perchlorate is used in military activities. Due to the outstanding oxidizing capacity of perchlorate, it is added to propellant for rockets, missiles, and satellites. Various routes of manmade perchlorate exposure to soil can be assumed. For example, complete or incomplete explosion of a signal bomb in a target or impact area, an oversupply of perchlorate for complete combustion at firing points, and large-scale fireworks display can be sources of perchlorate exposure to soil. Additionally, other routes could come from waste treatment processes. Because perchlorate in missiles naturally deteriorates over time, they need to be recharged. In the past, incineration was the preferred treatment for deteriorated perchlorate. When the incineration process is carried out in an open space and kept as ash onsite without caution, this becomes a detrimental route for soil contamination. Perchlorate is very stable in water and is not strongly retained by soil particle. As such, surface water or groundwater can become easily contaminated by the surface runoff or the leaching process. Perchlorate in growth medium also contaminates vegetation. This kind of pathway could subsequently affect higher level organisms through the food chain. Perchlorate contamination of drinking water and the food chain can potentially affect human health through interfering with iodide uptake by the thyroid gland. This kind of interference causes a decrease in thyroid hormone production and leads to hyperthyroidism. The permitted level of perchlorate concentration in drinking water is below 15 ppb in South Korea. Although some states in the USA have an advisory level for perchlorate in drinking water, it is very difficult to find a country which regulates the perchlorate level in soil as perchlorate contamination of soil is rare in normal circumstances. However, perchlorate is considered a major contaminant in military fields and so the management of perchlorate in soil is necessary. Accordingly, ISO 20295 proposes a standard method for determining perchlorate in soil using ion chromatography. For the standard method of ISO 20295, the limit of quantitation of perchlorate is 0,1 mg/kg soil. However, since perchlorate in soil is frequently found to be lower than the designated level, an analytical method for determining perchlorate at ppb levels is required. For this purpose, a standard method for analysing the trace level of perchlorate in soil has been developed.

Soil quality — Determination of perchlorate in soil using liquid chromatography-tandem mass spectrometry (LC-MS/MS)

1 Scope

This document specifies a method for determining of perchlorate in soil and soil-like materials using liquid chromatography connected to a tandem mass spectrometry (LC-MS/MS). It defines pretreatment (including drying and sieving) of sample, extraction, clean-up, analysis using LC-MS/MS, and calculation of perchlorate content in dry soil.

Under the conditions specified in this document, the limit of quantitation (LOQ) is approximately 4,6 µg/kg-dry soil.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

The soil and soil-like materials to be analysed are used for the experiment after pretreatment, air-drying and sieving. Perchlorate in soil and soil like materials is extracted by distilled or deionized water. Extraction of perchlorate is done by mechanical shaking and centrifugation.

After filtering the extract using 0,2 µm to 0,45 µm membrane filter (e.g. cellulose acetate, hydrophilic polypropylene or polyethersulphone filter), the internal standard solution is spiked into the filtrate. The filtrate is analysed by LC-MS/MS for the determination of perchlorate.

If the adverse effects of other anions (e.g. sulfate, chloride, and bicarbonate), cations, or organic matter are not negligible, appropriate pretreatment for the elimination of these effects should be applied. Selective removal of interfering elements with cartridge or column is one of the applicable pretreatments.

LC-MS/MS comprises of sample injection part, separation part, and detection part. Reversed-phase liquid chromatography column is used as stationary phase and acetonitrile solution is used as mobile phase. According to the characteristics of matrix, isocratic or gradient elution can be chosen.

The calibration and calculation of concentration of perchlorate in soil shall be conducted according to ISO 8466-1 or ISO 8466-2.

[Annex A](#) shows the examples of liquid chromatography-tandem mass spectrometry (LC-MS/MS) conditions. Examples of chromatogram of perchlorate ion and ^{18}O -perchlorate ion are shown in [Figure A.1](#).

5 Interferences

Any substance that has a similar retention time and shows a similar detector response with perchlorate can cause interference in the determination. Co-elution can be solved by changing columns and eluent conditions (e.g. gradient elution), modifying the eluent with organic solvents, or cleaning up of sample for selective removal of the interference in the pretreatment.

High concentration of chloride (Cl^-), sulfate (SO_4^{2-}), and carbonate (CO_3^{2-}) in extracts from saline soil can cause interference with the determination of perchlorate. According to ISO 20295, it was reported that when 800 mg/l of chloride, sulfate, and carbonate ions are injected into perchlorate standard solution (0,025 mg/l), the recovery rate of perchlorate was 80 %. Using cartridge for removal of selective ion or dilution of extracts can be an appropriate solution for the interference by the ions.

High organic carbon in soil can interfere with the determination of perchlorate by elevated baseline or increased noise in chromatogram. Using special type of cartridge (e.g. C_{18}) or dilution of extracts can be a good solution for this interference.

High concentrations of hydrogen sulfate ions ($\text{H}^{34}\text{SO}_4^-$) in the extracts can tail into the retention time of the perchlorate peak and elevate its baseline at Mass-to-charge ratio (m/z) 99. Quantitation of perchlorate can be performed by selecting m/z 83, 85, and 89 to minimize the potential interference by $\text{H}^{34}\text{SO}_4^-$.

6 Reagents

Use only reagents of recognized analytical grade. Prepare alternative concentrations or volumes of solutions as described in [6.1](#) to [6.3](#), if necessary. Alternatively, use commercially available solutions of the required concentration. And consequently, act on [6.4.1](#) to [6.4.5](#) accordingly.

6.1 Eluents

6.1.1 Acetonitrile (CH_3CN), HPLC grade

6.1.2 Acetic acid ($\text{CH}_3\text{CO}_2\text{H}$), glacial

6.1.3 Acetonitrile / acetic acid eluent

Mix 500 ml acetonitrile ([6.1.1](#)) with 500 ml water and spike 1 ml acetic acid ([6.1.2](#)). The solution contains 49,95 % acetonitrile and 0,1 % acetic acid solution.

6.2 Extractant

6.2.1 Water, distilled or deionized water or water of equivalent purity, with a resistivity of $\geq 18,2 \text{ M}\Omega \text{ cm}$ ($25 \text{ }^\circ\text{C}$).

6.3 Reagents for standard solutions

6.3.1 Sodium perchlorate (NaClO_4)

6.3.2 ^{18}O -sodium perchlorate ($\text{NaCl}^{18}\text{O}_4$)

6.4 Standard solutions

6.4.1 Stock standard solution, $c(\text{ClO}_4^-) = 1\ 000\ \text{mg/l}$

For example, prepare a stock standard solution by dissolving 0,123 g of anhydrous sodium perchlorate (6.3.1) in water (6.2.1) in a 100 ml volumetric flask. The solution shall be stored in the refrigerator at 2 °C to 8 °C for a period of up to 12 months.

6.4.2 Working standard solution, $c(\text{ClO}_4^-) = 10\ \text{mg/l}$

For example, prepare a working standard solution by diluting 1,0 ml of stock standard solution (6.4.1) to 100 ml with water (6.2.1). It shall be stored in the refrigerator at 2 °C to 8 °C for a period of up to 3 months.

6.4.3 Calibration standards

Dilute the working standard solution (6.4.2) to designated concentrations.

6.4.4 Internal standard stock solution, $c(\text{Cl}^{18}\text{O}_4^-) = 10\ \text{mg/l}$

For example, prepare an internal standard stock solution by dissolving 1,21 mg of ^{18}O -sodium perchlorate (6.3.2) in water (6.2.1) in a 100 ml volumetric flask and dilute to volume with water (6.2.1). Stock standards shall be stored in the refrigerator at 2 °C to 8 °C for a period up to 12 months.

6.4.5 Internal standard spiking solution, $c(\text{Cl}^{18}\text{O}_4^-) = 1\ 000\ \mu\text{g/l}$

For example, prepare an internal standard spiking solution by diluting 1,0 ml of the internal standard stock solution (6.4.4) to 10 ml with water (6.2.1). It shall be stored at $\leq 4\ ^\circ\text{C}$ when not in use.

7 Apparatus

General laboratory glassware.

7.1 Centrifuge tube

15 ml and 50 ml conical tube or glass tube with screw cap.

7.2 Equipment for extraction

7.2.1 Horizontal mechanical shaker

A shaker can retain shaking rate as 100 cycles per minute and have shaking width of about 10 cm.

7.2.2 Centrifuge

A centrifuge has a capacity of centrifuge rate of 1 800 *g*.

7.3 Filtration device

Suitable for using 0,2 µm to 0,45 µm membrane filter (e.g. syringe).

7.4 Membrane filter

0,2 µm to 0,45 µm membrane filter (e.g. cellulose acetate, hydrophilic polypropylene or polyethersulphone filter), chemically inert.

7.5 Clean-up cartridge

Cartridge (e.g. Ba, Ag, and H cartridge) for removing selective interference ions and C₁₈ cartridge or equivalent for removing organic matters in extracts.

7.6 Micro pipette and Pasteur pipette

0,2 ml, 1 ml, and 5 ml micro pipettes and Pasteur pipettes.

7.7 Liquid chromatograph/tandem mass spectrometer

7.7.1 Liquid chromatograph

7.7.1.1 General

The instrument shall be equipped with a programmable solvent delivery system and all necessary accessories including injection loop, analytical column, column heater, chromatography pump, in-line degasser, etc. If a solvent gradient is used to obtain separation, then a binary solvent delivery system is necessary. The chromatographic system shall be capable of interfacing with a tandem mass spectrometer. [Table A.1](#) shows an example of a LC setup for separating perchlorate from samples ([Annex A](#)).

7.7.1.2 Analytical column

Reversed-phase HPLC column [e.g. 4,6 (i.d.) × 250 mm (length), 5 µm (particle size)] for analysing perchlorate. The column utilizes the extra-dense bonding of organo-silane ligands.

7.7.2 Mass spectrometer

7.7.2.1 Electrospray ionization (ESI) source

The ESI source generates gas phase ions of perchlorate from the liquid phase. [Table A.2](#) shows an example of an ESI setup for LC-MS/MS analysis with fragmentation.

7.7.2.2 Tandem mass spectrometer

A triple quadrupole tandem mass spectrometer (MS/MS). Refer to the manufacturer's instructions for instrument tuning, conditions and verification. Tuning parameters are available from the instrument manufacturer, along with mass tuning solutions and instructions on how to optimize the mass spectrometer.

8 Procedure

8.1 Sampling and storage

Field moist soil sample is collected based on, for example, ISO 18400-101, ISO 18400-104, and ISO 18400-107, using sampling devices recommended by ISO 18400-102. The sample should be extracted within 28 days of its acquisition, and the extract shall be analysed within 28 days of its preparation.

8.2 Pretreatment

8.2.1 Drying

Field moist soil sample is dried in the air or oven. Spread soil sample in a layer not thicker than 5 cm on the tray. The tray shall not absorb any moisture from the sample and contaminate the sample. In case of air drying, direct sunlight shall be avoided. In case of oven drying, temperature in the oven shall not exceed 40 °C and ventilation device to be equipped.

8.2.2 Sieving

Dried sample is passed through 2 mm sieve. Remaining particle on the 2 mm sieve is crushed using suitable apparatus. Apparatus for crushing (e.g. rubber hammer, pestle) should be used to crush large soil particles and enable the crushed particles to pass through the 2 mm sieve. The crushed particles passed through 2 mm sieve are used as test portion.

The other details for drying and sieving shall be carried out according to ISO 11464.

8.3 Extraction

8.3.1 Mechanical shaking

Weigh (10,0 ± 0,1) g of dried and sieved sample into 50 ml of centrifuge tube. Add 30 ml of water (6.2.1). Shake it using horizontal mechanical shaker (7.2.1) for 1 h at 100 cycles per minute.

8.3.2 Centrifuge

After shaking process, centrifuge the mixture at 1 800 *g* for 1 h.

8.3.3 Filter and clean-up

Filter the extract using filtration device (e.g. plastic syringe) (7.3) with 0,2 µm to 0,45 µm membrane filter (7.4). The filtrate is used for LC-MS/MS analysis (7.7). If necessary, the filtrate is passed through specific cartridge or C₁₈ column (7.5) for clean-up of filtrate before analysing.

8.4 Liquid chromatography-Tandem mass spectrometry (LC-MS/MS)

8.4.1 Liquid chromatography

Optimize the liquid chromatograph for optimal perchlorate separation. An example of conditions of liquid chromatograph for separation of perchlorate from sample is provided in [Table A.1](#).

8.4.2 Tandem mass spectrometry

Tandem mass spectrometry shall have capacity of triple quadrupole or equivalent. Optimize the mass spectrometry for optimal perchlorate detection. An example of condition of triple quadrupole mass spectrometry is provided in [Table A.2](#). The recommended mass numbers of perchlorate are provided in [Table 1](#).

Table 1 — The recommended mass numbers of perchlorate for determining perchlorate using LC-MS/MS

Ion	Molecular weight	Selected ion (<i>m/z</i>)
$^{85}\text{ClO}_4^-$	99	83
$^{87}\text{ClO}_4^-$	101	85
$^{85}\text{Cl}^{18}\text{O}_4^-$	107	89

8.4.3 Calibration using internal standard material

This calibration method is applied to determine mass content independently. It is not affected by injection error, water content and matrix effects of sample. The recovery of target chemical in sample shall be equal to the recovery of standard solution.

Spike 50 µl of the internal standard spiking solution (6.4.5) in 10 ml of calibration standards and sample solutions. The concentration of internal standard material ($\text{Cl}^{18}\text{O}_4^-$) is 5 µg/l. The standard solutions (6.4.3) are analysed by LC-MS/MS. After plotting the ratio of mass in calibration solutions to peak area (or height) based on [Formula \(1\)](#), calculate the relative ratio of perchlorate (ClO_4^-) to ^{18}O labelled perchlorate ($\text{Cl}^{18}\text{O}_4^-$).

$$\frac{A_s}{A_{is}} = a \cdot \frac{C_s}{C_{is}} + b \quad (1)$$

where

A_s is the peak area of perchlorate in calibration solutions;

A_{is} is the peak area of ^{18}O labelled perchlorate in calibration solutions;

a is the slope of calibration curve;

C_s is the mass concentration of perchlorate in calibration solutions (µg/l);

C_{is} is the mass concentration of ^{18}O labelled perchlorate in calibration solutions (µg/l);

b is the y-intercept of calibration curve.

Inject the same volume for calibration as for the sample measurement. While analysing samples, check the validity of the calibration by injecting the middle range of the calibration standard solution. If measured values deviate from the associated nominal values by more than 10 %, a recalibration is necessary.

9 Quality control

After the calibration, initial calibration verification is performed using blank (method blank) and low-level standard solution. Also, continuing calibration verification is performed in every batch. Generally, the determined value range shall be ±10 % of nominal value.

Accuracy is evaluated by recovery test. The ratio of determined value to spiked value range shall be in between 80 % and 120 %. Precision is assessed as relative standard deviation (RSD). The RSD range shall be ≤ ±25 %.

The validation data are given in [Annex B](#).

10 Calculation

Calculate the perchlorate mass concentration in the soil sample using [Formula \(2\)](#).

$$X_s = \frac{(A_s / A_{is}) - b}{a} \cdot C_{is} \cdot f \cdot \frac{V}{m} \quad (2)$$

where

X_s is the mass concentration of perchlorate in the soil sample ($\mu\text{g/g}$);

A_s is the peak area of perchlorate in calibration solutions;

A_{is} is the peak area of ^{18}O labelled perchlorate in calibration solution;

a is the slope of calibration curve;

b is the y-intercept of calibration curve;

C_{is} is the mass concentration of ^{18}O labelled perchlorate in calibration solutions ($\mu\text{g/l}$);

f is the dilution factor in the LC-MS/MS analysis or other dilution steps (if necessary);

V is the total sample volume (ml);

m is the dried mass of soil sample (g).

11 Expression of results

Results shall be reported to a maximum of two significant figures.

EXAMPLE

Perchlorate (ClO_4^-) 2,1 $\mu\text{g/kg}$

Perchlorate (ClO_4^-) $1,8 \cdot 10^2 \mu\text{g/kg}$

12 Test report

This test report should contain at least the following information:

- the test method used, together with a reference to this document, i.e. ISO 5120:2023;
- all information necessary for complete identification of the sample;
- the results of the determination according to [Clause 10](#);
- any details that are not specified in this document or that are optional, as well as any other factors that may have affected the result.

Annex A (informative)

Examples of Liquid chromatography – tandem mass spectrometry (LC-MS/MS) conditions

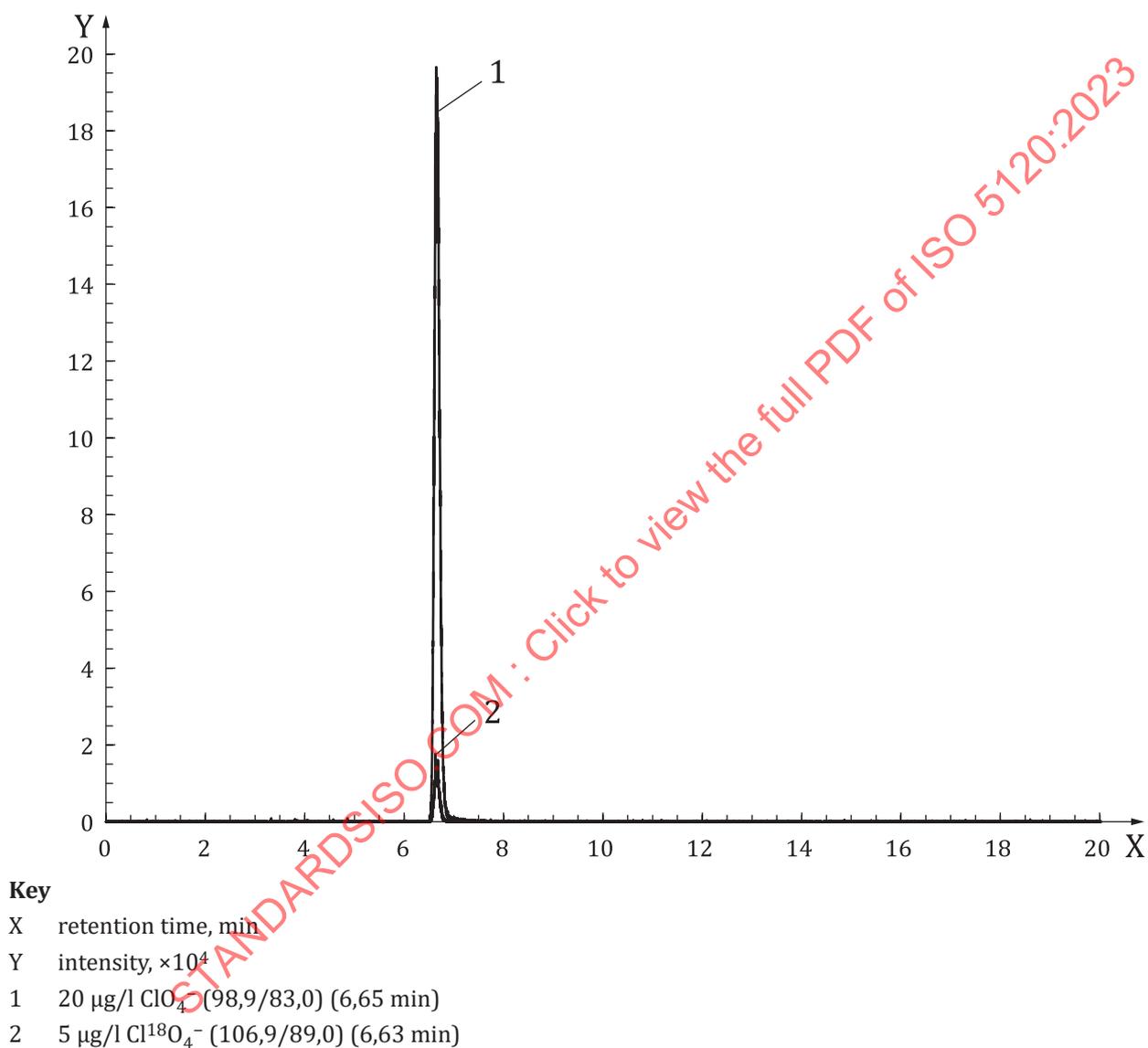


Figure A.1 — Example of perchlorate analysed by LC-MS/MS

Table A.1 — Example instrument setting for liquid chromatograph for determining perchlorate

Liquid chromatograph parameters	Example of setting
Analytical column	Reversed-phase LC column (4,6 (i.d.) × 250 mm (length), 5 µm (particle size))
Column temperature	30 °C
Injection volume	10 µl to 30 µl
Pump flow rate	0,5 ml/min
Mobile phase	49,95 % acetonitrile, 0,1 % acetic acid

NOTE This example instrument setting is based on 1200 series LC system (Agilent, USA).

Table A.2 — Example instrument setting for a tandem mass spectrometer for determining perchlorate

Mass spectrometry parameters	Example of setting
Ionization mode	Electrospray ionization (ESI)
Spray voltage	-4,5 kV
Polarity	Negative
Temperature	350 °C
Curtain gas	25
Ion source gas 1	50
Ion source gas 2	50
Detection mode	Multiple reaction monitoring (MRM) mode
Fragmentor voltage	200 V
Dry gas flow	12,0 l/min
Capillary voltage	Negative 3 000

NOTE This example instrument setting is based on API 3200 MSD (AB SCIEX, USA).

Annex B (informative)

Performance data

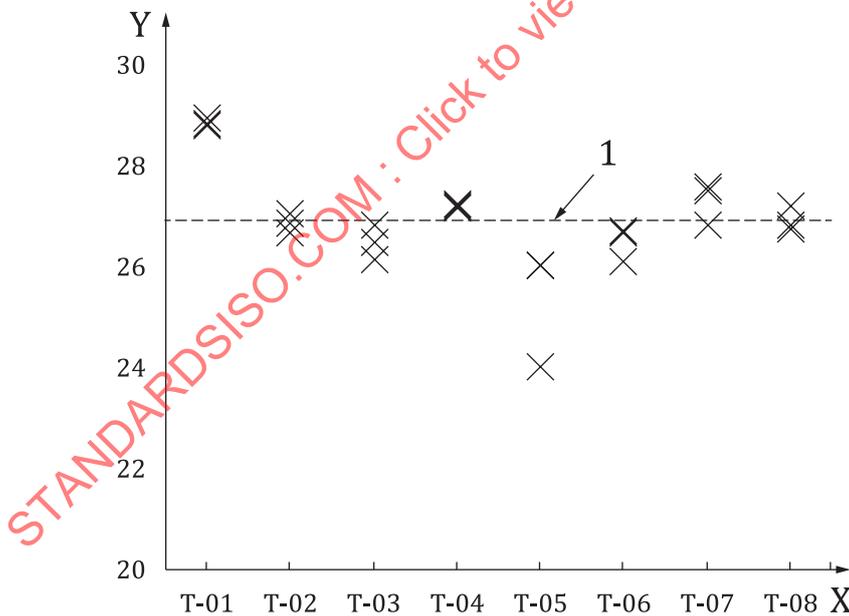
Environmental Soil Science and Contaminant Control Lab in Korea University organized an inter-laboratory validation study in September 2022.

The test soil materials were collected from perchlorate- and related compounds-free site and were ground to less than 150 µm of particle size. Each sample was prepared by adding perchlorate standard solution to test soil materials at a certain concentration. Test samples were homogenized with V-mixer.

8 laboratories from 2 countries participated in the validation study. All participants received two soil samples (1 bottle of known soil sample, 1 bottle of unknown soil sample, namely NICEM – VS503 and NICEM – VS504, respectively). The amount of soil sample in a brown glass bottle was approximately 50 g (Table 1).

For both samples, all participants carried out triplicate analysis. Figure B.1, Figure B.2, Table B.1 and Table B.2 show the summarized results of the inter-laboratory validation study. Data evaluation has been performed according to ISO 5725-2.

For a summary, the average of the coefficient of variation, repeatability ($C_{V,r}$) for NICEM – VS503 and NICEM – VS504 were 0,98 % and 1,07 %, respectively. And the average of the coefficient of variation, reproducibility ($C_{V,R}$) for both samples were 3,11 % and 4,09 %, respectively.



Key

- X lab code
- Y perchlorate concentration (µg kg⁻¹)
- 1 mean

Figure B.1 — Scatter plot of test results for NICEM – VS503 sample