



SLOVENSKI STANDARD

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Gnojila - Določevanje elementov v sledovih - Določevanje kroma (VI) s fotometrijo (metoda A) in z ionsko kromatografijo s spektrofotometrijsko detekcijo (metoda B)

Fertilisers - Determination of trace elements - Determination of chromium(VI) by photometry (method A) and by ion chromatography with spectrophotometric detection (method B)

Düngemittel - Bestimmung von Elementspuren - Bestimmung von Chrom(VI) mit Photometrie (Verfahren A) und mit Ionenchromatographie mit spektrometrischer Detektion (Verfahren B)

Engrais - Dosage des éléments traces - Dosage du chrome (VI) par spectrophotométrie (méthode A) et chromatographie ionique avec détection spectrophotométrique (méthode B)

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English Version

Fertilisers - Determination of trace elements - Determination of chromium(VI) by photometry (method A) and by ion chromatography with spectrophotometric detection (method B)

Engrais - Dosage des éléments traces - Dosage du chrome (VI) par spectrophotométrie (méthode A) et chromatographie ionique avec détection spectrophotométrique (méthode B)

Düngemittel - Bestimmung von Elementspuren - Bestimmung von Chrom(VI) mit Photometrie (Verfahren A) und mit Ionenchromatographie mit spektrometrischer Detektion (Verfahren B)

This draft European Standard is submitted to CEN members for unique acceptance procedure. It has been drawn up by the Technical Committee CEN/TC 260.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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Foreword

This document (FprEN 16318:2013) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

This document is currently submitted to the Unique Acceptance Procedure.

This document will supersede CEN/TS 16318:2012.

The following changes have been made to the former edition:

- a) the CEN Technical Specification has been adopted as a European Standard;
- b) a reference to Annex A has been added to Clause 7 concerning the results of inter-laboratory tests on the precision of method A;
- c) the statistical results of an inter-laboratory test performed by the Technical Group Fertilizers of the German VDLUFA have been added as Annex A (informative);
- d) the document has been editorially revised.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

FprEN 16318:2013 (E)

1 Scope

This European Standard specifies two methods for the determination of the content of soluble chromate in fertilisers.

Method A specifies the determination of chromate after extraction with water by photometry. This method can be used to determine Cr(VI)-mass fractions in solids higher than 1 mg/kg.

Method B specifies the determination of chromate by alkaline digestion and ion chromatography with spectrophotometric detection. This method can be used to determine Cr(VI)-mass fractions in solids higher than 0,1 mg/kg.

NOTE In case of reducing or oxidising fertiliser matrix, no valid Cr(VI) content can be reported.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN 15192:2006, *Characterisation of waste and soil — Determination of Chromium(VI) in solid material by alkaline digestion and ion chromatography with spectrophotometric detection*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Sampling and sample preparation

Sampling is not part of the methods specified in this European Standard. A recommended sampling method is given in EN 1482-1 [2].

Sample preparation shall be carried out in accordance with EN 1482-2.

Reasonable precautions have to be taken to prevent oxidation of chromate present in the sample. Samples shall be taken using appropriate devices and placed in containers that do not contain stainless steel (e.g. plastic, glass).

After digestion, the sample shall be analysed as soon as possible.

5 Method A: Determination by water extraction and spectrophotometric detection

5.1 Principle

Chromate is extracted from the sample with water at room temperature. The chromate concentration in the extract is measured by colorimetry using 1,5-diphenylcarbazide.

When chromate reduces the 1,5-diphenylcarbazide a magenta coloured complex of 1,5-diphenylcarbazone and chromium is formed which can be measured colorimetrically at 540 nm.

5.2 Reagents

Use only reagents of recognised analytical grade and water conforming to 5.2.1.

5.2.1 Water, according to EN ISO 3696, grade 2 (electrical conductivity max. $0,1 \text{ mS} \times \text{m}^{-1}$ equivalent to resistivity greater than $0,01 \text{ M}\Omega\cdot\text{m}$ at $25 \text{ }^\circ\text{C}$).

It is recommended that the water used is obtained from a purification system that delivers ultrapure water having a resistivity greater than $0,18 \text{ M}\Omega\cdot\text{m}$.

5.2.2 Hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$; 37 % volume fraction, $\rho \approx 1,18 \text{ g/ml}$.

5.2.3 Hydrochloric acid, diluted, $c(\text{HCl})$ approximately 6 mol/l .

Mix equal volumes of hydrochloric acid (5.2.2) and water (5.2.1).

5.2.4 Diphenylcarbazide solution.

Dissolve $1,0 \text{ g}$ Diphenylcarbazide [$\text{CO}(\text{NHNHC}_6\text{H}_5)_2$] in 100 ml acetone and add one drop of acetic acid. If this solution is stored in a glass bottle in the dark at $4 \text{ }^\circ\text{C}$, it may be used for approximately two weeks. This solution shall be clear without colour. If the diphenylcarbazide solution has become coloured, it shall be discarded and freshly prepared before use.

5.2.5 Chromate standard stock solution, $\rho(\text{chromate}) = 1\,000 \text{ mg/l}$.

Dissolve $2\,829 \text{ mg}$ potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in $1\,000 \text{ ml}$ water (5.2.1) using a volumetric flask. This solution may be used for two weeks if stored in the dark at $4 \text{ }^\circ\text{C}$.

5.2.6 Chromate standard stock solution, $\rho(\text{chromate}) = 50 \text{ mg/l}$.

Dilute 5 ml chromate stock solution (5.2.5) to 100 ml with water (5.2.1) using a volumetric flask. This solution shall be freshly prepared on the day of use.

5.2.7 Standard solutions.

Prepare the standard solutions by diluting aliquots of the 50 mg/l chromate standard stock solution (5.2.6) with water (5.2.1) in 100 ml volumetric flasks. The aliquots and final concentrations of chromate are given in Table 1. Other volumes and concentrations may be used for the preparation of standard solutions if appropriate. See 5.4.3 for further instructions on how to prepare the standard solutions.

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Table 1 — Aliquots and final concentrations for the preparation of chromate standards from a 50 mg/l chromate standard stock solution in 100 ml volumetric flasks

Aliquot ml	Final concentration of chromate mg/l
0	0 (calibration blank)
0,05	0,025
0,20	0,10
0,50	0,25
1,00	0,50
1,50	0,75

A blank solution shall be prepared in a separate volumetric flask by adding water (5.2.1) only.

The standard solutions shall be prepared freshly on the day of use.

5.3 Apparatus

5.3.1 Common laboratory glassware.

5.3.2 Analytical balance, capable of weighing to an accuracy of 1 mg.

5.3.3 Membrane filters and vacuum filtration apparatus.

The filters should be made of cellulose nitrate, pore size 5 µm and fit into the vacuum filtration apparatus.

5.3.4 Mechanical shaker.

5.3.5 Pipettes and/or dilutor, pipettes with fixed or variable volume and volumetric glassware to perform dilutions.

Alternatively, a dilutor may be used, which is an instrument used for automated volumetric dilutions. The precision and accuracy of this equipment for volumetric dilutions shall be proved, controlled, and documented regularly.

5.3.6 Spectrophotometer, capable to measure transmitted light at 540 nm.

5.3.7 100 ml bottles, made from PE or PP, equipped with watertight caps.

5.4 Procedure

5.4.1 Preparation of the test solution

Weigh approximately 10 g, to the nearest 0,01 g, of the prepared sample and transfer to a suitable extraction vessel (5.3.7).

Add 40 ml of water (5.2.1). If the sample material (e.g. organic material) absorbs a high amount of water, add additionally water (5.2.1) to obtain a fluid suspension. Record the amount of additional water added. It may be necessary to use larger extraction vessels in such cases. Close the cap tightly and shake vigorously for 15 min on a mechanical shaker (5.3.4). Filter the suspension immediately through membrane filters with 5 µm pore size using the vacuum filtration apparatus (5.3.3).