

SLOVENSKI STANDARD SIST-TS CEN/TS 17762:2023

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Anorganska gnojila - Določevanje bakra v gnojilih iz amonijevega nitrata z veliko vsebnostjo dušika

Inorganic fertilizers - Determination of the copper content in ammonium nitrate fertilizers of high nitrogen content

Anorganische Düngemittel - Bestimmung des Kupfergehaltes in Ammoniumnitratdüngemitteln mit hohem Stickstoffgehalt

Engrais inorganiques - Détermination de la teneur en cuivre dans les engrais à base de nitrate d'ammonium et à forte teneur en azote

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65.080 Gnojila Fertilizers

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TECHNICAL SPECIFICATION
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ICS 65.080

English Version

Inorganic fertilizers - Determination of the copper content in ammonium nitrate fertilizers of high nitrogen content

Engrais inorganiques - Détermination de la teneur en cuivre dans les engrais à base de nitrate d'ammonium et à forte teneur en azote Anorganische Düngemittel - Bestimmung des Kupfergehaltes in Ammoniumnitratdüngemitteln mit hohem Stickstoffgehalt

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (CEN/TS 17762:2022) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

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1 Scope

This document specifies a method for the determination of the copper content in ammonium nitrate fertilizers of high nitrogen content.

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.

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

EN 12944-1, Fertilizers and liming materials — Vocabulary — Part 1: General terms

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1 and EN 12944-2 apply. ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

4 Principle

The sample is dissolved in dilute hydrochloric acid and the copper is determined by atomic absorption spectrophotometry (AAS) or by inductive coupled plasma - optical emission spectroscopy (ICP-OES).

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

- **5.1 Hydrochloric acid,** density $\rho = 1,18$ g/ml, at 20 °C.
- **5.2 Hydrochloric acid,** substance concentration c = 6 mol/l.
- **5.3 Hydrochloric acid,** c = 0.5 mol/l.
- 5.4 Ammonium nitrate.
- **5.5 Hydrogen peroxide,** 30 % m/V.
- 5.6 Copper stock solution.

Weigh, to the nearest 0,001 g, 1 g of pure copper (Cu), dissolve in 25 ml of hydrochloric acid (5.2), add 5 ml of hydrogen peroxide (5.5) in portions and dilute to 1 l with water. One ml of this solution contains 1 000 μ g of copper.

A commercially available stock copper solution may be used.

5.7 Copper standard solution.

Dilute 10 ml of copper stock solution (5.6) to 100 ml with water and then dilute 10 ml of the resulting solution, to 100 ml with water, 1 ml of the final dilution contains 10 μ g of copper.

Prepare this solution at the time of use.

6 Apparatus and equipment

Usual laboratory glassware and equipment and, in particular, the following.

6.1 Atomic absorption spectrophotometer with a copper lamp (324,8 nm), or **inductive coupled plasma - optical emission spectrometer**.

The choice of the apparatus is depending on availability of equipment.

- **6.2 Balance,** capable of weighing to the nearest 0,001 g.
- **6.3 Beaker,** capacity 400 ml.
- **6.4 Graduated flask,** capacity 250 ml and 1 000 ml.
- 6.5 Copper-free filter paper.

7 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

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

8 Procedure

8.1 Preparation of the solution for analysis ds/sist/cc3f3122-e557-4635-ba8b-

Weigh, to the nearest 0,001 g, 25 g of the sample, place it in a 400-ml beaker (6.3), add carefully 20 ml of hydrochloric acid (5.1) (there may be a vigorous reaction due to carbon dioxide formation). Add more hydrochloric acid, if necessary. When gas formation has stopped, evaporate to dryness on a steam bath, stirring occasionally with a glass rod. Add 15 ml hydrochloric acid (5.2) and 120 ml of water. Stir with the glass rod, which should be left in the beaker, and cover the beaker with a watch glass. Boil the solution gently until dissolution is complete and then cool.

Transfer the solution quantitatively into a 250-ml graduated flask (in case of ICP-OES: transfer into 1 000-ml graduated flask), by washing the beaker with 5 ml hydrochloric acid (5.2), and twice with 5 ml of boiling water, fill up to the mark with hydrochloric acid (5.3) and mix carefully.

Filter through a copper-free filter paper (6.5), discarding the first 50 ml.

8.2 Blank solution

Prepare a blank solution from which only the sample has been omitted and allow for this in the calculation of the final results.

8.3 Determination

8.3.1 Preparation of sample and blank solutions

Dilute the sample solution (see 8.1) and the blank solution (see 8.2) with hydrochloric acid (5.3) to a concentration of copper within the optimal measuring range of the spectrophotometer. Normally no dilution is needed.