
**Gnojila - Določevanje ionov kompleksno vezanih mikrohranil v gnojilih -
Identifikacija lignosulfonatov**

Fertilizers - Determination of complexed micro-nutrient ions in fertilizers - Identification of lignosulfonates

Düngemittel - Bestimmung der in Düngemitteln komplexgebundenen
Spurennährstoffionen - Identifizierung von Ligninsulfonaten

Engrais - Dosage des oligo-éléments complexés dans les engrais - Identification des lignosulfates

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ICS:

65.080

Gnojila

Fertilizers

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EUROPEAN STANDARD
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**Fertilizers - Determination of complexed micro-nutrient ions in
fertilizers - Identification of lignosulfonates**

Engrais - Dosage des oligo-éléments complexés dans les
engrais - Identification des lignosulfonates

Düngemittel - Bestimmung der in Düngemitteln
komplexgebundenen Spurennährstoffionen - Identifizierung
von Ligninsulfonaten

This European Standard was approved by CEN on 1 October 2011.

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Foreword

This document (EN 16109:2011) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2012, and conflicting national standards shall be withdrawn at the latest by May 2012.

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1 Scope

This document specifies two complementary methods (method A and method B) that allow lignosulfonates to be indentified as soluble complexing agents in fertilizers.

NOTE Lignosulfonate, as a complexing agent, is a natural polymer produced as a by-product of the sulfite method for manufacturing paper from wood pulp in the paper industry. As a natural polymer, it presents a poorly defined and variable chemical structure. It is an intricate mixture of small- to moderate-sized polymeric compounds with sulfonate groups attached to the molecule, and diverse complexing capacity.

The methods are applicable to EC fertilizers covered by Regulation (EC) No 2003/2003 [1].

2 Normative references

The following referenced documents are indispensable for the application 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 12944-1:1999, *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 ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

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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 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.

5 Method A: Determination of phenolic hydroxyl content and 232,5 nm absorption for the identification of lignosulfonates

5.1 Principle

The method for the determination of the phenolic hydroxyl content is based on the ultraviolet absorption of phenols in alkaline solution (phenolate). The absorbance of an alkaline solution of the sample is measured directly against an acid solution of the same sample. The phenolic hydroxyl content of the sample is calculated from the molar extinction coefficient maximum of the resulting curve and the molar extinction coefficient of reference compounds determined in the same way.

The determination of the absorption at 232,5 nm is normally considered the method for the quantification of lignosulfonates, providing that no other ultraviolet absorbing organic compounds are present.

NOTE For additional information see [3] and [4].

5.2 Apparatus

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

5.2.1 Magnetic stirrer.

5.2.2 Balance, capable of weighing to an accuracy of 1 mg.

5.2.3 Filter paper for qualitative analysis, pore size 15 µm to 20 µm.¹

5.2.4 pH-meter, equipped with a glass electrode.

5.2.5 UV-Vis spectrophotometer, equipped with 1 cm quartz cells.

5.3 Reagents

5.3.1 General

- a) reagents shall be of recognized analytical grade,
- b) water used for the preparation of sample solutions shall conform to EN ISO 3696, grade 2 and free of organic contaminants.

5.3.2 Hydrochloric acid solution, $c(\text{HCl}) = 6 \text{ mol/l}$.

5.3.3 Sodium hydroxide solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$.

5.3.4 Analytical grade fine mesh strong cation exchange resin ²

Styrene/DVB type, 8 % crosslinked. Hydrogen form. Functional group: sulphonic acid. Nominal exchange capacity: 1,7 mmol_e/ml. Mesh: 50 to 100.

5.4 Procedure

5.4.1 Preparation of stock solution

Weigh, to the nearest 1 mg, 0,15 g to 0,20 g of the sample in a 100 ml beaker. Add 4 g of cation exchange resin (5.3.4) and about 20 ml to 25 ml of water. Allow the ion-exchange process to take place for 20 min, ensuring proper mixing by means of a magnetic stirrer.

1) Albet 412 filter paper or equivalent is an example of suitable product commercially available. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

2) Biorad AG 50 W-X8 (50-100) Cat. No. 142-1431 is an example of suitable product commercially available. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

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Filter (5.2.3) into a 250 ml volumetric flask to remove the resin and thoroughly wash the filter. Dilute to the mark with water (stock solution).

5.4.2 Solution A (acid)

Take an aliquot (40 ± 5 ml) of the stock solution into a 100 ml beaker and adjust pH between 2,0 and 2,2 with few drops of hydrochloric acid solution (5.3.2). Pipette 5 ml of the pH-adjusted solution into a 50 ml volumetric flask and dilute to the mark. Final concentration 0,06 g/l to 0,08 g/l.

5.4.3 Solution B (basic)

Pipette 5 ml of the stock solution into a 50 ml volumetric flask. Add 10 ml of sodium hydroxide solution (5.3.3) to adjust pH over 11,0. Dilute to the mark. Final concentration 0,06 g/l to 0,08 g/l. Check that the pH of the solution is over 11,0, if not prepare solution B adding more sodium hydroxide.

5.4.4 Solution C

Pipette 10 ml of the stock solution into a 100 ml beaker and fill with water to 60 ± 5 ml. Adjust the pH of the solution between 4,0 and 5,0 with the sodium hydroxide solution (5.3.3). Transfer quantitatively into a 100 ml volumetric flask, dilute to the mark with water and homogenize. See 5.4.6.

5.4.5 Measurement of phenolic hydroxyl content

Fill both cells in the UV spectrophotometer with water. Enter background correction. Scan from 340 nm to 220 nm to check baseline.

Fill the sample cell with solution B (5.4.3), and the reference cell with solution A (5.4.2). Scan from 340 nm to 220 nm. Rinse cells with water.

5.4.6 Measurement of 232,5 nm absorption

Fill the sample cell with solution C (5.4.4), and the reference cell with water and record absorbance at 232,5 nm. The absorbance of the final solution should be between 0,2 and 0,8 to minimize deviations from Beer's Law instrumental error. If necessary, the volume to be taken from stock solution (5.4.1) to prepare solution C (5.4.4) should be adapted.

5.5 Calculation**5.5.1 Phenolic hydroxyl content**

Plot the spectrum in terms of absorbance. Record wavelength and absorbance for the maximum peak at 240 nm to 260 nm and for the minimum on either the right or the left side of the maximum. Subtract minimum absorbance from the maximum height (ΔAbs_{max}) (see Figure 1).

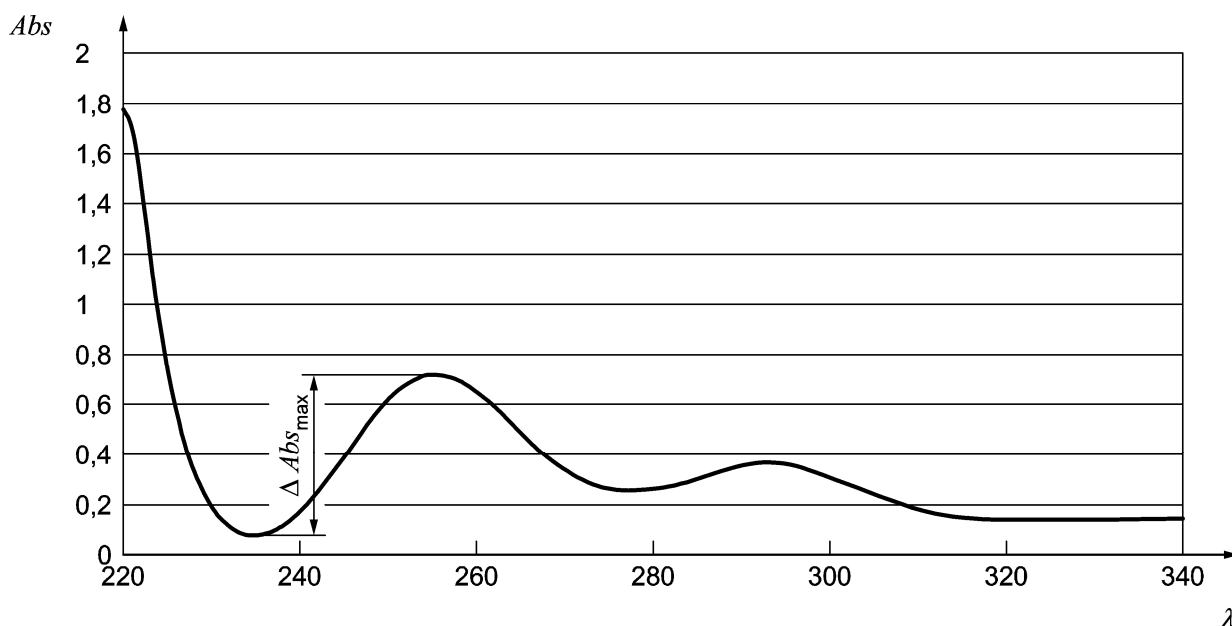


Figure 1 — Example of the spectrum of solution B against A for the determination of phenolic hydroxyl content of a lignosulfonate showing the maximum and the minimum (in this case at the left side) absorbances

Calculate the phenolic hydroxyl content, w_{ph} , of the sample, expressed as mass fraction in percent using the ΔAbs_{max} value of the sample and an average $\Delta \epsilon_{max}$ value for reference compounds ($8\,867,5\text{ l}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$) by the following formula:

$$w_{ph} = \frac{\Delta Abs_{max}}{m} \times d \times 17 \times \frac{1}{\Delta \epsilon_{max}} \times \frac{100}{1000} \quad (1)$$

$$d = \frac{50 \times 250}{5} \quad (2)$$

where

m is the mass of the test portion in grams;

d is the dilution factor included in 5.4.1, 5.4.2. and 5.4.3, in millilitres;

ΔAbs_{max} is the value obtained subtracting the minimum from the maximum absorbance;

17 is the number of OH mol $\frac{17\text{gOH}}{\text{molOH}}$;

$\Delta \epsilon_{max}$ is the average molar extraction coefficient for reference compounds ($8\,867,5\text{ l}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$).

5.5.2 232,5 nm absorption as lignosulfonic acid content

Calculate the 232,5 nm absorption as lignosulfonic acid content, w_{la} , of the sample, expressed as mass fraction in percent, by the following formula:

$$w_{la} = \frac{A_{232,5} \times d}{m \times f \times 10} \quad (3)$$