

CEN/TC 260

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Organo-mineral fertilizers -

Extraction of phosphorus, which is soluble in neutral ammonium citrate

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European Foreword

This document (CEN/prEN 17779) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

This is a working document.

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

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1 Scope

This document specifies a method for the extraction of phosphorus soluble in neutral ammonium citrate.

The extracts are suitable for analysis using [CEN/TS 17774](#).

NOTE Alternatively, inductively coupled plasma mass spectrometry (ICP-MS) can be used for the measurement if the user proves that the method gives the same results.

This method is applicable to organo-mineral fertilizers and to the fertilizing product blends where the EU fertilising product organic and organo-mineral fertilizer contained in the blend represents the highest % by mass in the blend. In case of equal shares, the user may apply either this or the standard(s) applicable to the other component product(s).

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.

CEN/TS 17774, Organic and organo-mineral fertilizers — Determination of the content of specific elements by ICP-AES after extraction by water

3 Terms and definitions

No terms and definitions are listed in this document.

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

Phosphorus is extracted at a temperature of 65 °C using a neutral ammonium citrate solution of pH = 7.

5 Sampling

Sampling should be performed following the principle described in EN 1482 (all parts) with appropriate adaptations, required to account for specificities of organo-mineral fertilizers.

6 Reagents

6.1 Water, Water with a specific conductivity not higher than 0,2 mS/m at 25 °C, free from the elements to be determined.

6.2 Ammonium Hydroxide Solution, containing 28 % to 29 % of NH₃

Commenté [BA1]: Description of preparation?

6.3 Solution with crystalline citric acid (C₆H₈O₇ · H₂O), pH = 7, ρ(C₆H₈O₇ · H₂O) = 185 g/l, specific gravity = 1,09 at 20 °C.

Prepare the reagent as follows:

Dissolve 370 g of crystalline citric acid ($C_6H_8O_7 \cdot H_2O$) in about 1,5 l of water and make an approximately neutral solution by adding 345 ml of ammonium hydroxide solution (6.2). If the NH_3 concentration is lower than 28 % add a correspondingly larger quantity of ammonium hydroxide solution and dilute the citric acid in correspondingly smaller quantities of water.

Commenté [BA2]: If using the right AH-solution, this would not be necessary..

Cool and make exactly neutral by keeping the electrodes of a pH-meter immersed in the solution. Add the ammonia, at 28 % to 29 % of NH_3 , drop by drop, stirring continuously (with a mechanical stirrer) until obtaining exactly a pH of 7 at a temperature of 20 °C. At this point make up the volume with water (6.1) to 2 l and check the pH again. Keep the reagent in a closed container and check the pH at regular intervals. Alternatively, a commercially certified solution can be used.

7 Apparatus

7.1 Common laboratory equipment and glassware.

~~7.2. Beaker~~, capacity 2 l.

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~~7.3. pH-meter~~.

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~~7.4. Erlenmeyer flask~~, capacity 200 ml or 250 ml.

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~~7.5. Volumetric flasks~~, capacity 500 ml and 2 000 ml.

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~~7.6. Water bath~~, to be set thermostatically at (65 ± 2) °C, equipped with a suitable stirrer.

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~~7.7. Dry pleated filter~~, medium speed, phosphate free.

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8 Procedure

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8.1 Test portion

Transfer 1 g to the nearest 0.01 g of the laboratory sample to be analysed into a 200 ml or 250 ml Erlenmeyer flask (7.3) containing 100 ml of ammonium citrate solution (6.2) previously heated to (65 ± 2) °C.

a supprimé: or 3 g (for fertilizers with pure or partially digested rock phosphate) with an accuracy of 0,01 g

NOTE: For fertilizers with pure or partially digested rock phosphate use 3 g of material

8.2 Extraction

Stopper the Erlenmeyer flask (7.3) and shake in order to suspend the test portion without forming lumps. Remove the stopper for an instant in order to balance the pressure and close the Erlenmeyer flask again. Place the flask in a water bath (7.5) set to maintain the contents of the flask at 65 ± 2 °C and connect it to the stirrer. During stirring, the level of the suspension in the flask shall stay constantly below the level of the water in the water bath. If a mechanical stirrer is not available, the flask may be shaken by hand every 5 min.

Regulate mechanical stirring to ensure complete suspension.

After stirring for 60 ± 1 minute, remove the Erlenmeyer flask from the water bath.

Cool immediately under running water to ambient temperature and, immediately, quantitatively transfer the contents from the Erlenmeyer flask into a volumetric 500 ml flask (7.4) with a jet of water (wash bottle). Make up the volume with water. Mix thoroughly. Filter through a dry pleated filter (7.6) into a dry container, discarding the first part of the filtrate (about 50 ml).

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Collect about 100 ml of clear filtrate.

The determination of phosphorus after extraction shall be done according to CEN/TS 17774.

9 Test Report

In the test report of the determination method (CEN/TS 17774), a reference to this standard shall be included, as well as the date of extraction and potential deviation while applying this met

Bibliography

- [1] EN 1482 (all parts), *Fertilizers and liming materials — Sampling and sample preparation*