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Fertilizers — Extraction of phosphorus soluble in Joulie's alcaline ammonium citrate

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Fertilizers - Extraction of phosphorus soluble in Joulie's alcaline ammonium citrate

Engrais - Extraction du phosphore soluble dans le citrate d'ammonium alcalin de Joulie

Düngemittel - Extraktion des in alkalischem Ammoniumcitrat nach Joulie löslichen Phosphors

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Foreword

This document (EN 15923:2011) 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of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

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1 Scope

This document specifies the procedure for the extraction of phosphorus soluble in Joulie's alkaline ammonium citrate.

The method is applicable to all the straight and compound phosphate fertilizers, in which the phosphate occurs in an alumino-calcic form.

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 15475, *Fertilizers — Determination of ammoniacal nitrogen*

CEN/TS 15959, *Fertilizers — Determination of extracted phosphorus*

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 Principle

Extraction by shaking vigorously with an alkaline solution of ammonium citrate of defined specification (and where appropriate in the presence of oxine) at about 20 °C.

5 Sampling

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. Grinding of the laboratory sample is recommended for homogeneity reasons.

6 Reagents

6.1 Water, distilled or demineralized.

6.2 Joulie's alkaline solution of ammonium citrate

6.2.1 This solution contains 400 g of citric acid and 153 g of NH_3 per litre. Its free ammonia content is approximately 55 g per litre. Prepare the solution according to 6.2.2 or 6.2.3.

6.2.2 In a 1 l graduated flask, dissolve 400 g of citric acid ($C_6H_8O_7$. H_2O) in approximately 600 ml of ammonia (d_{20} = 0,925 g/ml i.e. 200 g of NH_3 per litre). Add citric acid successively in quantities of 50 g to 80 g maintaining the temperature below 50 °C. Make up the volume to 1 l with ammonia.

6.2.3 In a 1 l graduated flask, dissolve 432 g of dibasic ammonium citrate ($C_6H_{14}N_2O_7$) in 300 ml of water (6.1). Add 440 ml of ammonia (d_{20} = 0,925 g/ml). Make the volume up to 1 l with water (6.1).

6.2.4 Verify the total ammonia content as follows. Take an amount of 10 ml of the citrate solution and place it in a 250 ml flask. Make up the volume with water (6.1). Determine the ammoniacal nitrogen content on 25 ml of this solution according to EN 15475.

1 ml of H_2SO_4 0,25 mol/l = 0,008 516 g of NH_3.

Under these conditions, the reagent is considered to be correct when the amount of millilitres found upon titration lies between 17,7 ml and 18 ml.

If this is not the case, add 4,25 ml of ammonia (d_{20} = 0,925 g/l) per 0,1 ml below 18 ml indicated above.

6.3 8-hydroxyquinoline (oxine), powdered.

7 Apparatus

Use common laboratory equipment and glassware, in particular equipment according to 7.2 to 7.4.

7.1 Small mortar, glass or porcelain with pestle.

7.2 500 ml graduated flasks.

7.3 1 000 ml graduated flask.

7.4 Rotary shaker, 35 to 40 turns per minute.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,000 5 g, 1 g of the prepared sample and place in a small mortar (7.1). Add about 10 drops of ammonium citrate solution (6.2) to moisten it and break it up very carefully with the pestle.

8.2 Extraction

Add 20 ml of ammonium citrate solution (6.2) and mix to a paste, leave it to settle for about 1 min.

Decant the liquid into a 500 ml graduated flask (7.2), straining off particles which might have escaped the preceding moist disintegration. Add 20 ml of citrate solution (6.2) to the residue, grind as above and decant the liquid into the graduated flask. Repeat the process four times, so that by the end of the fifth time all the product can be poured into the flask. The total quantity of citrate used for these processes shall be approximately 100 ml.

Rinse the pestle and mortar above the graduated flask with 40 ml of water (6.1).

Shake the stoppered flask for 3 h on the rotary shaker (7.4).

Leave the flask standing for 15 h to 16 h, shake it again under same conditions for 3 h. The temperature during the whole process shall be kept at (20 ± 2) °C.

Make up to the graduation mark with water (6.1). Filter through a dry filter, discard the first portion of the filtrate and collect the clear filtrate in a dry flask. Continue the filtering until a sufficient quantity of filtrate is obtained to carry out the phosphorus determination according to CEN/TS 15959.

9 Application to fertilizers containing magnesium

The use of oxine makes it possible to apply this method to fertilizers containing magnesium. This use is recommended when the ratio of magnesium and phosphoric anhydride contents is higher than 0,03 ($Mg/P_2O_5 > 0,03$). If this is the case, add 3 g of oxine to the moistened test portion. The use of oxine in the absence of magnesium is not, moreover, likely to interfere subsequently with the determination. In the known absence of magnesium it is, however, possible not to use oxine.

Bibliography

[1] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

[2] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21/11/2003, P. 0001-0194, Annex IV, method 3.1.5.3

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