
STATUTORY RULES OF NORTHERN IRELAND

1991 No. 540

AGRICULTURE

**The Fertilisers (Sampling and Analysis)
Regulations (Northern Ireland) 1991**

Made - - - - 19th December 1991

Coming into operation 17th February 1992

**THE FERTILISERS (SAMPLING AND ANALYSIS)
REGULATIONS (NORTHERN IRELAND) 1991**

1. Title, commencement and interpretation
 2. Prescribed amount for the purposes of the definition of sampled portion
 3. Manner of taking, marking, sealing and fastening up of samples
 4. Methods of sending part of a sample
 5. Application of the methods of analysis
 6. Form of certificate of analysis
 7. Modification of the Agriculture Act 1970
 8. Revocations
- Signature

SCHEDULE **MANNER OF TAKING, MARKING, SEALING AND
1 FASTENING UP OF SAMPLES**

PART I — DEFINITIONS

PART II — GENERAL INSTRUCTIONS FOR THE TAKING OF SAMPLES

1. In the case of fertiliser in containers, only unopened containers...
2. The sample shall be taken and prepared as quickly as...
3. A sample shall not be drawn from any part of...
4. When stones are naturally present in a fertiliser, they shall,...
5. An inspector who intends to take a sample in accordance...
6. The sampling apparatus shall be made of materials which cannot...
7. In the case of a sampling spear its dimensions shall...
8. Notwithstanding the provisions of these Regulations, a sampling spear shall...
9. Mechanical apparatus may be used for the sampling of moving...

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10. Apparatus designed to divide the sample into approximately equal parts...
11. A sample taken in accordance with the methods described below...

PART III — QUANTITATIVE REQUIREMENTS

1. **Sampled portion**
2. **Incremental sample**
3. **Aggregate sample**
4. **Final sample**

PART IV — TAKING AND PREPARATION OF SAMPLES

1. **Incremental samples**
2. **Aggregate sample**
3. **Reduced sample**
4. **Final samples**

PART V — MARKING, SEALING AND FASTENING UP OF THE FINAL SAMPLE

1. Each container of a final sample shall be so secured...
2. A label shall be attached to the container or receptacle...
3. The container or receptacle may also be sealed, or the...

PART VI — SAMPLING TABLES

SCHEDULE **METHODS OF ANALYSIS**

2

PART I

1. **General**
2. **Reagents and Apparatus**
3. **Methods of Analysis**
 1. Preparation of the sample for analysis
 2. Determination of ammoniacal nitrogen
 3. (a) Determination of nitrate and ammoniacal nitrogen-Ulsch method
 4. (a) Determination of nitrogen in calcium cyanamide-in the absence of...
 5. Determination of total nitrogen in urea
 6. Determination of cyanamide nitrogen
 7. Determination of biuret in urea
 8. (a) Determination of different forms of nitrogen-in the presence of...
 9. (a) Extraction of total phosphorus-by mineral acids
 10. Determination of extracted phosphorus
 11. Determination of water-soluble potassium
 12. (a) Determination of water-soluble magnesium-atomic absorption spectro-photometric method
 13. (a) Determination of total magnesium-atomic absorption spectrophoto-metric method
 14. Determination of chlorides, in the absence of organic matter
 15. (a) Determination of fineness of grinding—dry method
 16. Methods of analysis and test procedures for ammonium nitrate fertiliser...
- A Methods for the application of thermal cycles
- B Determination of oil retention
- C Determination of the combustible ingredients
- D Determination of the pH value
- E Determination of particle size
- F Determination of the chloride content (as chloride ion)
- G Determination of copper

1.

PREPARATION OF THE SAMPLE FOR ANALYSIS

1. SCOPE
2. PRINCIPLE
 - 2.1 Solid fertilisers: the preparation of a final sample received at...
 - 2.2 Fluid fertilisers: the final sample is mixed by shaking to...
3. APPARATUS
 - 3.1 Sample divider (optional).
 - 3.2 Sieves with apertures of 0.2 mm and 0.5 mm.
 - 3.3 250 ml flasks, stoppered.
 - 3.4 Porcelain pestle and mortar or grinder.
4. CHOICE OF TREATMENT TO BE USED
 - 4.1 ***Final samples which must not be ground***
 - 4.2 ***Final samples which must be divided and part of which must be ground***
 - 4.3 ***Final samples in respect of which all determinations are carried out on a ground product***
5. METHOD
6. SPECIAL CASES
7. FLUID FERTILISERS

2.

DETERMINATION OF AMMONIACAL NITROGEN

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. REAGENTS
 - 4.1 Hydrochloric acid solution, 50% (V/V): dilute an appropriate volume of...
 - 4.8 Sodium hydroxide solution, 30 g per 100 ml ammonia free....
 - 4.9 Indicator solutions: Mixed indicator: (4.9.1) Solution A: dissolve 1 g...
 - 4.10 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
 - 4.11 Ammonium sulphate.
5. APPARATUS
 - 5.1 Distillation apparatus consisting of a round-bottomed flask of suitable capacity...
 - 5.2 Rotary shaker, 35 to 40 turns per minute.
6. PREPARATION OF SAMPLE
7. PROCEDURE
 - 7.2 ***Determination***

Note:

 - 7.3 ***Blank***
 - 7.4 ***Control test***
8. EXPRESSION OF THE RESULT
TABLE FOR METHOD 2
 - Variant (a)***
 - Variant (b)***
 - Variant (c)***

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3a.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — ULSCH METHOD

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. REAGENTS
 - 4.1 Hydrochloric acid solution, 50% (V/V): dilute an appropriate volume of...
 - 4.2 Sulphuric acid, 0.1 N solution.
 - 4.3 Sodium or potassium hydroxide, 0.1 N solution, carbonate free.
 - 4.4 Sulphuric acid solution, approximately 30% H₂SO₄ (W/V), ammonia free.
 - 4.5 Powdered iron reduced in hydrogen. (The prescribed quantity of iron...
 - 4.6 Sodium hydroxide solution, 30 g per 100 ml, ammonia free....
 - 4.7 Indicator solutions: Mixed indicator: (4.7.1) Solution A: dissolve 1 g...
 - 4.8 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
 - 4.9 Sodium nitrate.
5. APPARATUS
6. PREPARATION OF SAMPLE
7. PROCEDURE
 - 7.1 ***Preparation of the solution***
 - 7.2 ***Determination***
 - 7.3 ***Blank test***
 - 7.4 ***Control test***
8. EXPRESSION OF THE RESULTS

3b.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — ARND METHOD

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. REAGENTS
 - 4.1 Hydrochloric acid solution, 50% (V/V): dilute an appropriate volume of...
 - 4.8 Sodium hydroxide solution, approximately 2 N.
 - 4.9 Arnd's alloy—powdered so as to pass through a sieve with...
 - 4.10 20% Magnesium chloride solution: dissolve 200 g magnesium chloride (MgC₁₂.6H₂O)...
 - 4.11 Indicator solutions: Mixed indicator: (4.11.1) Solution A: dissolve 1 g...
 - 4.12 Anti-bump granules of pumice stone washed in hydrochloric acid and...
 - 4.13 Sodium nitrate.
5. APPARATUS
6. PREPARATION OF SAMPLE
7. PROCEDURE
 - 7.1 ***Preparation of the solution for analysis***

- 7.2 **Determination**
- Note:*
- 7.3 **Blank test**
- 7.4 **Control test**
- 8. EXPRESSION OF THE RESULTS

3c.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — DEVARDA METHOD

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
 - 4.1 Hydrochloric acid solution, 50% (V/V): dilute an appropriate volume of...
 - 4.8 Devarda's alloy—powdered so that 90 to 100% will pass through...
 - 4.9 Sodium hydroxide solution, 30 g per 100 ml ammonia free...
 - 4.10 Indicator solutions: Mixed indicator: (4.10.1) Solution A: dissolve 1 g...
 - 4.11 Ethanol, 95%.
 - 4.12 Sodium nitrate.
- 5. APPARATUS
 - 5.1 Distillation apparatus consisting of a round bottomed flask of suitable...
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
 - 7.1 **Preparation of the solution for analysis**
 - 7.2 **Determination**
 - (Note:*
 - 7.3 **Blank test**
 - 7.4 **Control test**
- 8. EXPRESSION OF RESULTS

4a

DETERMINATION OF THE TOTAL NITROGEN IN CALCIUM CYANAMIDE — IN THE ABSENCE OF NITRATE

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
 - 4.1 Sulphuric acid solution 50% (V/V): dilute an appropriate volume of...
 - 4.2 Potassium sulphate.
 - 4.3 Copper oxide (CuO) — 0.3 to 0.4 g for each...
 - 4.4 Sodium hydroxide solution, 30 g per 100 ml, ammonia free....
 - 4.11 Indicator solutions: Mixed indicator: (4.11.1) Solution A: dissolve 1 g...
 - 4.12 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
 - 4.13 Potassium thiocyanate.
- 5. APPARATUS

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- 5.1 Distillation apparatus. See Method 2.
- 6. PREPARATION OF SAMPLE
- 7. PROCEDURE
- 7.1 **Preparation of the solution**
- 7.2 **Determination**
- 7.3 **Blank test**
- 7.4 **Control test**
- 8. EXPRESSION OF THE RESULT

4b.

*DETERMINATION OF TOTAL NITROGEN IN CALCIUM
CYANAMIDE — IN THE PRESENCE OF NITRATE*

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Sulphuric acid. (d = 1.84 g/ml).
- 4.2 Powdered iron reduced in hydrogen.
- 4.3 Potassium sulphate, finely pulverised. Sulphuric acid. 0.1 N solution. Sodium or potassium hydroxide, 0.1 N solution, carbonate free. for...
- 4.10 Indicator solutions:
- 4.11 Solution of stannous chloride:
- 4.12 Sodium hydroxide solution, 30 g per 100 ml, ammonia free....
- 4.13 Standard nitrate-ammoniacal solution:
- 4.14 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
- 5. APPARATUS
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Preparation of the solution**
- 7.2 **Determination**
- 7.3 **Blank test**
- 7.4 **Control test**
- 8. EXPRESSION OF THE RESULTS

5.

DETERMINATION OF TOTAL NITROGEN IN UREA

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Sulphuric acid, concentrated, (d = 1.84 g/ml).
- 4.2 Sodium hydroxide solution, 30 g per 100 ml, ammonia free....
- 4.9 Indicator solutions:
- 4.10 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
- 4.11 Urea.
- 5. APPARATUS
- 5.1 Distillation apparatus. See Method 2.
- 6. PREPARATION OF THE SAMPLE

- 7. PROCEDURE
- 7.1 **Preparation of the solution**
- 7.2 **Determination**
- 7.3 **Blank test**
- 7.4 **Control test**
- 8. EXPRESSION OF THE RESULT

6.

DETERMINATION OF CYANAMIDE NITROGEN

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Glacial acetic acid.
- 4.2 Ammonia solution: dilute 1 volume of ammonia (d = 0.88...
- 4.3 Ammoniacal silver solution, according to Tollens, freshly prepared: mix 500...
- 4.4 Concentrated sulphuric acid (d = 1.84 g/ml).
- 4.5 Potassium sulphate.
- 4.6 Copper oxide (CuO), 0.3-0.4 g for each determination or an...
- 4.7 Sodium hydroxide solution, 30 g per 100 ml, ammonia free....
- 4.8 Sulphuric acid, 0.1 N solution.
- 4.9 Sodium or potassium hydroxide, 0.1 N solution.
- 4.10 Indicator solutions: Mixed indicator: (4.10.1) Solution A: dissolve 1 g...
- 4.11 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
- 4.12 Potassium thiocyanate.
- 5. APPARATUS
- 5.1 Distillation apparatus. See Method 2
- 5.2 500 ml graduated flask (for example Stohmann).
- 5.3 Rotary shaker, 35-40 turns per minute.
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Safety precaution**
- 7.2 **Preparation of the solution for analysis**
- 7.3 **Determination**
- 7.4 **Blank test**
- 7.5 **Control test**
- 8. EXPRESSION OF RESULTS

7.

DETERMINATION OF BIURET IN UREA

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Methanol.
- 4.2 Sulphuric acid solution, approximately 0.1 N.
- 4.3 Sodium hydroxide solution, approximately 0.1 N.

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- 4.4 Alkaline solution of potassium sodium tartrate: in a 1 litre...
- 4.5 Copper sulphate solution:
- 4.6 Biuret standard solution:
- 4.7 Methyl red indicator solution:
- 5. APPARATUS
- 5.1 Spectrophotometer.
- 6. PREPARATION OF SAMPLE
- 7. PROCEDURE
- 7.1 **Preparation of the standard curve**
- 7.2 **Preparation of solution for analysis**
- 7.3 **Determination**
- 8. EXPRESSION OF RESULTS

8a.

DETERMINATION OF DIFFERENT FORMS OF NITROGEN IN THE SAME SAMPLE — IN THE PRESENCE OF CYANAMIDE NITROGEN

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 3.1 **Total soluble and insoluble nitrogen**
- 3.2 **Forms of soluble nitrogen**
- 4. REAGENTS
- 4.1 Potassium sulphate.
- 4.2 Iron powder, reduced with hydrogen (the prescribed quantity of iron...
- 4.3 Potassium thiocyanate.
- 4.4 Potassium nitrate.
- 4.5 Ammonium sulphate.
- 4.6 Urea.
- 4.7 Sulphuric acid solution: dilute an appropriate volume of sulphuric acid...
- 4.8 Sulphuric acid, 0.2 N solution.
- 4.9 Sodium hydroxide solution, 30 g per 100 ml. ammonia free....
- 4.10 Sodium or potassium hydroxide, 0.2 N solution, free from carbonates....
- 4.11 Stannous chloride solution: dissolve 120 g of stannous chloride (SnC...
- 4.12 Sulphuric acid, concentrated (d = 1.84 g/ml).
- 4.13 Hydrochloric acid solution: dilute an appropriate volume of hydrochloric acid...
- 4.14 Glacial acetic acid.
- 4.15 Sulphuric acid solution, approximately 30% (W/V) H₂SO₄.
- 4.16 Ferrous sulphate, crystalline, FeSO₄.7H₂O.
- 4.17 Sulphuric acid, 0.1 N solution.
- 4.18 Octan-1-o 1.
- 4.19 Potassium carbonate, saturated solution.
- 4.20 Sodium or potassium hydroxide, 0.1 N solution, free from carbonate....
- 4.21 Barium hydroxide, saturated solution.
- 4.22 Sodium carbonate solution, 10 g per 100 ml.
- 4.23 Hydrochloric acid, 2 N solution.

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- 4.24 Hydrochloric acid, 0.1 N solution.
- 4.25 Urease solution:
- 4.26 Xanthidrol solution, 5 g per 100 ml in ethanol or...
- 4.27 Copper oxide (CuO): 0.3 to 0.4 g per determination or...
- 4.28 Anti-bump granules washed in hydrochloric acid and ignited.
- 4.29 Indicator solutions: Mixed indicator solution: (4.29.1) Solution A: dissolve 1...
- 4.30 Indicator papers:
- 4.3 Ethanol or methanol: solution 95%.
- 5. APPARATUS
- 5.1 Distillation apparatus. See Method 2.
- 5.2 Apparatus for the determination of ammoniacal nitrogen according to analytical...
- 5.3 Apparatus for I-tie estimation of urea nitrogen according to the...
- 5.4 Rotary shaker, 35-40 turns per minute.
- 5. pH meter.
- 5.6 Laboratory oven.
- 5.7 Sintered glass crucibles, diameter of pores 5 to 15 microns....
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Total soluble and insoluble nitrogen**
- 7.2 **Forms of soluble nitrogen**
- 8. VERIFICATION OF THE RESULTS
- 8.1 In certain cases, a difference may be found between the...
- 8.2 Before each analysis, check that the apparatus is working properly...

8b.

DETERMINATION OF DIFFERENT FORMS OF NITROGEN IN THE SAME SAMPLE — IN THE ABSENCE OF CYANAMIDE NITROGEN

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 3.1 **Total soluble nitrogen**
- 3.2 Total soluble nitrogen except nitric nitrogen, by Kjeldahl digestion after...
- 3.3 Nitric nitrogen, by difference: between 3.1.2 and 3.2 and/or between...
- 3.4 Ammoniacal nitrogen, by cold distillation of a weak alkaline solution;...
- 3.5 Urea nitrogen, either: (3.5.1) By conversion using urease, into ammonia,...
- 4. REAGENTS
- 4.1 Potassium sulphate.
- 4.2 Iron powder, reduced with hydrogen (the prescribed quantity of iron...
- 4.3 Potassium nitrate.
- 4.4 Ammonium sulphate.
- 4.5 Urea.
- 4.6 Sulphuric acid, 0.2 N solution.
- 4.7 Sodium hydroxide solution, 30 g per 100 ml, ammonia free....
- 4.8 Sodium or potassium hydroxide, 0.2 N solution, free of carbonates....

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- 4.9 Sulphuric acid (d = 1.84 g/ml).
- 4.10 Hydrochloric acid solution: dilute an appropriate volume of hydrochloric acid...
- 4.11 Glacial acetic acid.
- 4.12 Sulphuric acid, solution approximately 30% (W/V) H₂SO₄.
- 4.13 Ferrous sulphate, crystalline FeSO₄.7H₂O.
- 4.14 Sulphuric acid, 0.1 N solution.
- 4.15 Octal-l-ol.
- 4.16 Potassium carbonate, saturated solution.
- 4.17 Sodium or potassium hydroxide, 0.1 N solution.
- 4.18 Barium hydroxide, saturated solution.
- 4.19 Sodium carbonate solution, 10 g per 100 ml.
- 4.20 Hydrochloric acid, 2 N solution.
- 4.21 Hydrochloric acid, 0.1 N solution.
- 4.22 Urease solution: suspend 0.5 g active urease in 100 ml...
- 4.23 Xanthidrol solution, 5 g per 100 ml in ethanol or...
- 4.24 Catalyst: copper oxide (CuO), 0.3 to 0.4 g per determination,...
- 4.25 Anti-bump granules of pumice stone washed with hydrochloric acid and...
- 4.26 Indicator solutions: Mixed indicator (4.26.1) Solution A: dissolve 1 g...
- 4.27 Indicator papers: litmus, bromothymol blue (or other papers sensitive to...
- 4.28 Ethanol or methanol, 95% (V/V).
- 5. APPARATUS
- 5.1 Distillation apparatus. See Method 2.
- 5.2 Apparatus for determination of ammoniacal nitrogen. An example of recommended...
- 5.3 Apparatus for determination of urea nitrogen by the urease method...
- 5.4 Rotary shaker: 35 — 40 turns per min.
- 5.5 pH meter.
- 5.6 Sintered glass crucibles, diameter of pores 5 to 15 microns....
- 6. PREPARATION OF SAMPLE
- 7. PROCEDURE
- 7.1 ***Preparation of solution for analysis***
- 7.2 ***Total nitrogen***
- 7.3 ***Total nitrogen excluding nitric nitrogen***
- 7.4 Nitric nitrogen is obtained: by difference between (7.2.4) — (7.5.3...
- 7.5 ***Ammoniacal nitrogen***
- 7.6 ***Ureic nitrogen***
- 1. ***Remarks***
- 2. The titration may also be carried out using an indicator...
- 8. VERIFICATION OF RESULTS
- 8.1 Before each analysis, check the functioning of the apparatus and...

9a.

EXTRACTION OF TOTAL PHOSPHORUS BY MINERAL ACIDS

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS

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- 4.1 Sulphuric acid (d = 1.84 g/ml).
- 4.2 Nitric acid (d = 1.40 g/ml).
- 5. APPARATUS
- 5.1 A Kjeldahl flask, with a capacity of at least 500...
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**

9b.

EXTRACTION OF PHOSPHORUS BY 2% FORMIC ACID

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENT
- 4.1 Formic acid, 2% (20 g per litre): dilute 82 ml...
- 5. APPARATUS
- 5.1 500 ml graduated flask (for example Stohmann).
- 5.2 Rotary shaker, 35 — 40 turns per minute.
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**

9c.

EXTRACTION OF PHOSPHORUS BY 2% CITRIC ACID

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENT
- Note:**
- 5. APPARATUS
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**

9d.

EXTRACTION OF PHOSPHORUS BY NEUTRAL AMMONIUM CITRATE

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Neutral ammonium citrate solution (pH = 7.0). This solution must...
- 5. APPARATUS
- 5.1 pH meter.
- 5.2 Water bath which can be set thermostatically at 65°C, equipped...
- 6. PREPARATION OF THE SAMPLE

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- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**

9e.

*EXTRACTION OF PHOSPHORUS BY ALKALINE
AMMONIUM CITRATE (PETERMANN'S METHOD) AT 65°C*

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Petermann's solution
 - Characteristics:**
 - Preparation from diammonium citrate:**
 - Preparation from citric acid and ammonia:**
 - Check the ammoniacal nitrogen content as follows:**
- 5. APPARATUS
- 5.1 Water bath which can be maintained at a temperature of...
- 5.2 500 ml graduated flask (for example Stohmann flask).
- 6. PREPARATION OF SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**

9f

*EXTRACTION OF PHOSPHORUS BY ALKALINE AMMONIUM
CITRATE (PETERMANN'S METHOD) AT AMBIENT TEMPERATURE*

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENT
- 5. APPARATUS
- 5.1 250 ml graduated flask (for example Stohmann).
- 5.2 Rotary shaker, 35 — 40 turns per minute.
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**

9g.

EXTRACTION OF PHOSPHORUS BY JOULIE'S ALKALINE AMMONIUM CITRATE

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Joulie's alkaline solution of ammonium citrate: This solution contains 400...
- 4.2 8 — Hydroxyquinoline, (oxine), powdered.

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- 5. APPARATUS
- 5.1 Rotary shaker, 35 — 40 turns per minute.
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**
- 8. APPENDIX

9h.

EXTRACTION OF PHOSPHORUS BY WATER

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. APPARATUS
- 4.1 500 ml graduated flask (for example Stohmann).
- 4.2 Rotary shaker, 35 — 40 turns per minute.
- 5. PREPARATION OF THE SAMPLE
- 6. PROCEDURE
- 6.1 **Extraction**
- 6.2 Determination Determine the phosphorus according to Method 10, on an...

10.

DETERMINATION OF EXTRACTED PHOSPHORUS

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Concentrated nitric acid (d = 1.40 g/ml).
- 4.2 Molybdate reagent: Preparation of the reagent based on sodium molybdate:...
- 5. APPARATUS
- 5.1 Filter crucible with porosity of 5 to 20 microns.
- 5.2 Drying oven regulated at 250°C f 10°C.
- 5.3 Sintered glass funnel with porosity of 5 to 20 microns....
- 6. PROCEDURE
- 6.1 **Treatment of the solution**
- 6.2 Hydrolysis Bring the contents of the Erlenmeyer flask to the...
- 6.3 **Weighing the crucible**
- 6.4 **Precipitation**
- 6.5 **Filtering and washing**
- 6.6 **Drying and weighing**
- 6.7 **Blank test**
- 6.8 Control test
- 7. EXPRESSION OF THE RESULTS

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11.

DETERMINATION OF WATER-SOLUBLE POTASSIUM

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. REAGENTS
 - 4.1 Formaldehyde, 25 — 35% solution, filter if necessary before use....
 - 4.2 Potassium chloride.
 - 4.3 Sodium hydroxide. 10 N solution. Care should be taken to...
 - 4.4 Indicator solution: dissolve 0.5 g phenolphthalein in 100 ml 90%...
 - 4.5 EDTA solution: 4 g of the dihydrated disodium salt of...
 - 4.6 STPB solution: dissolve 32.5 g sodium tetraphenylborate in 480 ml...
 - 4.7 Liquid for washing: dilute 20 ml of the STPB solution...
 - 4.8 Bromine water: saturated bromine solution in water.
5. APPARATUS
 - 5.1 Filter crucibles with a porosity of 5 to 20 microns....
 - 5.2 Oven regulated at $120 \pm 10^\circ\text{C}$.
6. PREPARATION OF THE SAMPLE
7. PROCEDURE
 - 7.1 Extraction Weigh to the nearest 0.001 g, 10 g of...

Note:

 - 7.2 *Determination*
 - 7.3 *Weighing the crucible*
 - 7.4 *Precipitation*
 - 7.5 *Filtering and washing*
 - 7.6 *Drying and weighing*
 - 7.7 *Blank test*
 - 7.8 *Control test*
8. EXPRESSION OF RESULTS
 - 8.1 *Method of calculation and formulae*

12a.

*DETERMINATION OF WATER-SOLUBLE MAGNESIUM —
ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD*

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. REAGENTS
 - 4.1 Hydrochloric acid, N solution (approximately).
 - 4.2 Hydrochloric acid, 0.5 N solution.
 - 4.3 Magnesium standard solution: dissolve 1.013 g magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in...
 - 4.4 Strontium chloride solution: dissolve 15 g strontium chloride ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$) in...
5. APPARATUS
 - 5.1 Atomic absorption spectrophotometer with a magnesium lamp (285.2 nm).
6. PREPARATION OF SAMPLE
7. PROCEDURE
 - 7.1 *Extraction*

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- 7.2 **Preparation of the sample solution**
- 7.3 **Blank solution**
- 7.4 **Standard solutions for calibration**
- 7.5 **Measurement**
- 8. EXPRESSION OF THE RESULTS

2b.

DETERMINATION OF WATER-SOLUBLE MAGNESIUM—EDTA METHOD

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
 - 4.1 Magnesium solution, 0.05 M: weigh out 2.016 g magnesium oxide,...
 - 4.2 EDTA solution, 0.05 M: dissolve 18.61 g of the dihydrated...
 - 4.3 Calcium solution 0.05 M: weigh out 5.004 g of dry...
 - 4.4 Calcein indicator: carefully mix in a mortar 1 ,g calcein...
 - 4.5 Calcon carbonic acid indicator: dissolve 400 mg calcon carbonic acid...
 - 4.6 Eriochrome black-T indicator: dissolve 300 mg eriochrome black-T in a...
 - 4.7 Potassium cyanide solution, 2 g per 100 ml.
 - 4.8 Solution of potassium hydroxide and potassium cyanide: dissolve 280 g...
 - 4.9 pH 10 buffer solution: dissolve 33 g ammonium chloride in...
- 5. APPARATUS
 - 5.1 Magnetic or mechanical stirrer.
 - 5.2 pH-meter.
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
 - 7.1 **Extraction**
 - 7.2 **Control test**
 - 7.3 **Determination**
- 8. EXPRESSION OF THE RESULTS

13a.

*DETERMINATION OF TOTAL MAGNESIUM -ATOMIC
ABSORPTION SPECTROPHOTOMETRIC METHOD*

- 1. SCOPE This method is for the determination of total magnesium....
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENT
 - 4.1 Hydrochloric acid solution 50% (V/V): dilute an appropriate volume of...
 - 4.2 Hydrochloric acid, N solution (approximately).
 - 4.3 Hydrochloric acid, 0.5 N solution.
 - 4.4 Magnesium solution: dissolve 1 ,013 g magnesium sulphate (MgSO₄.7H₂O) in...
 - 4.5 Strontium chloride solution: dissolve 75 g strontium chloride (SrCl₂.6H₂O) in...
- 5. APPARATUS

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- 5.1 Atomic absorption spectrophotometer with a magnesium lamp (285.2 nm).
- 6. PREPARATION OF SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Preparation of the sample solution**
- 7.3 **Blank solution**
- 7.4 **Standard solutions for calibration**
- 7.5 **Measurement**
- 8. EXPRESSION OF THE RESULTS

13b.

DETERMINATION OF TOTAL MAGNESIUM —EDTA METHOD

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Magnesium solution, 0.05 M: weigh out 2.016 g of magnesium...
- 4.2 EDTA solution 0.05 M: dissolve 18.61 g of the dihydrated...
- 4.3 Calcium solution 0.05 M: weigh out 5.004 g of dry...
- 4.4 Calcein indicator: carefully mix in a mortar 1 g of...
- 4.5 Calcon carbonic acid indicator: dissolve 400 mg of calcon carbonic...
- 4.6 Eriochrome black-T indicator: dissolve 300 mg of eriochrome black-T in...
- 4.7 Potassium cyanide solution, 2 g per 100 ml.
- 4.8 Solution of potassium hydroxide and potassium cyanide: dissolve 280 g...
- 4.9 pH 10.5 buffer solution: dissolve 33 g ammonium chloride in...
- 4.10 Hydrochloric acid solution: 50% (V/V): dilute an appropriate volume of...
- 4.11 Sodium hydroxide solution, 5 N.
- 5. APPARATUS
- 5.1 Magnetic or mechanical stirrer.
- 5.2 pH meter.
- 6. PREPARATION OF THE SAMPLE
- 7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Control test**
- 7.3 **Determination**
- Note:**
- 8. EXPRESSION OF THE RESULTS

14.

DETERMINATION OF CHLORIDES IN THE ABSENCE OF ORGANIC MATERIAL

- 1. SCOPE
- 2. FIELD OF APPLICATION
- 3. PRINCIPLE
- 4. REAGENTS
- 4.1 Nitrobenzene or diethyl ether.

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- 4.2 Nitric acid, 10 N solution.
- 4.3 Indicator solution: dissolve 40 g of ferric ammonium sulphate $[\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}]$...
- 4.4 Silver nitrate, 0.1 N solution.
- 4.5 Ammonium thiocyanate, 0.1 N solution. Preparation: since this salt is...
5. APPARATUS
- 5.1 Rotary shaker, 35 — 40 turns per minute.
6. PREPARATION OF SAMPLE
7. PROCEDURE
- 7.1 **Extraction**
- 7.2 **Determination**
- Note:**
- 7.3 **Blank test**
- 7.4 **Control test**
8. EXPRESSION OF THE RESULT

15a.

DETERMINATION OF FINENESS OF GRINDING — DRY METHOD

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. APPARATUS
- 4.1 Mechanical sieve shaker.
- 4.2 Sieves with apertures of 0.160 mm and 0.630 mm respectively...
5. PROCEDURE
6. EXPRESSION OF THE RESULTS

15b.

DETERMINATION OF THE FINENESS OF GRINDING OF SOFT NATURAL PHOSPHATES

1. SCOPE
2. FIELD OF APPLICATION
3. PRINCIPLE
4. REAGENTS
5. APPARATUS
- 5.1 Sieves with apertures of 0.063 mm and 0.125 mm respectively...
- 5.2 Glass funnel of 20 cm diameter mounted on a stand....
- 5.3 Laboratory oven.
6. PROCEDURE
7. EXPRESSION OF THE RESULTS
8. REMARK

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16.

**METHODS OF ANALYSIS AND TEST PROCEDURES FOR AMMONIUM NITRATE
FERTILISERS CONTAINING MORE THAN 28% NITROGEN BY WEIGHT**

A.

Methods for the Application of Thermal Cycles

1. SCOPE AND FIELD OF APPLICATION
2. THERMAL CYCLES
 - 2.1 Field of application This procedure is for thermal cycling prior...
 - 2.2 **Principle and definition**
 - 2.3 **Apparatus**
 - 2.4 **Procedure**

B.

Determination of Oil Retention

1. SCOPE AND FIELD OF APPLICATION
2. DEFINITION
3. PRINCIPLE
4. REAGENT
5. APPARATUS
 - 5.1 Balance, capable of weighing to the nearest 0.01 gram.
 - 5.2 Beakers, of capacity 500 ml.
 - 5.3 Funnel, of plastic materials, preferably with a cylindrical wall at...
 - 5.4 Test sieve, aperture 0.5 mm, fitting into the funnel (5.3)....
 - 5.5 **Note:**
 - 5.6 Absorbent tissue (laboratory grade).
6. PROCEDURE
 - 6.1 Two individual determinations are carried out in quick succession on...
 - 6.3 Remove particles smaller than 0.5 mm using the test sieve...
 - 6.3 Filter the entire contents of the beaker through the funnel...
 - 6.4 Lay two sheets of filter paper (5.5) (about 500 ×...
 - 6.5 **Repeating the rolling procedure and reweighing**
7. EXPRESSION OF RESULTS
 - 7.1 **Method of calculation and formula**

C.

Determination of the Combustible Ingredients

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. REAGENTS
 - 3.1 Analytical-grade chromium VI oxide; Cr-(VI)-03.
 - 3.2 Sulphuric acid diluted to 60% by volume: pour 360 ml...
 - 3.3 Silver nitrate: 0.1 M solution.
 - 3.4 Barium hydroxide: weigh out 15 grams of barium hydroxide ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$),...

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- 3.5 Hydrochloric acid: 0.1 M standard solution.
- 3.6 Sodium hydroxide: 0.1 M standard solution.
- 3.7 Bromophenol blue: solution of 0.4 grams per litre in water...
- 3.8 Phenolphthalein: solution of 2 grams per litre in 60% by...
- 3.9 Soda lime: particle dimensions, about 1.0 to 1.5 mm.
- 3.10 Demineralised water, freshly boiled to remove carbon dioxide.
- 4. APPARATUS
- 4.1 Standard laboratory equipment, in particular: filter crucible with a plate...
- 4.2 Compressed nitrogen supply.
- 4.3 Apparatus made up of the following parts and assembled, if...
Caution:
- 5. PROCEDURE
- 5.1 **Sample for analysis**
- 5.2 **Removal of carbonates**
- 5.3 **Oxidation and absorption**
- 5.4 **Measurement of the carbonates originating from organic material**
- 6. BLANK TEST
- 7. EXPRESSION OF RESULTS

D.

Determination of the pH Value

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 **Buffer solution. pH 6.88 at 20°C**
- 3.2 **Buffer solution pH 4.00 at 20°C**
- 3.3 Commercially available pH standard solutions may be used.
- 4. APPARATUS
- 5. PROCEDURE
- 5.1 **Calibration of the pH meter**
- 5.2 **Determination**
- 6. EXPRESSION OF RESULTS

E.

Determination of the Particle Size

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. APPARATUS
- 3.1 200 mm diameter woven-wire test sieves to BS 410 (1986)...
- 3.2 Balance to weigh to 0.1 gram.
- 3.3 Mechanical sieve shaker (if available) capable of imparting both vertical...
- 4. PROCEDURE
- 4.1 The sample is divided representatively into portions of approximately 100...
- 4.2 Weigh one of these portions to the nearest 0.1 gram....
- 4.3 Arrange the nest of sieves in ascending order; receiver 0.5...
- 4.4 Shake by hand or machine, imparting both a vertical and...
- 4.5 Remove the sieves from the nest in turn and collect...

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- 4.6 Weigh the material retained on each sieve and that collected...
- 5. EVALUATION OF THE RESULTS
- 5.1 Convert the fraction masses to a percentage of the total...
- 5.2 At least two separate analyses should be carried out and...
- 6. EXPRESSION OF RESULTS

F.

Determination of the Chlorine Content (as Chloride Ion)

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
 - 3.1 Acetone AR.
 - 3.2 Concentrated nitric acid (density at 20°C = 1.40 g/ml).
 - 3.3 Silver nitrate 0.1 M standard solution. Store this solution in...
 - 3.4 Silver nitrate 0.004 M standard solution — prepare this solution...
 - 3.6 Potassium chloride, 0.1 M standard reference solution. Weigh, to the...
 - 3.6 Potassium chloride, 0.004 M standard reference solution — prepare this...
- 4. APPARATUS
 - 4.2 Bridge, containing a saturated potassium nitrate solution, connected to the...
 - 4.3 Magnetic stirrer, with a Teflon-coated rod.
 - 4.4 Microburette with fine-pointed tip, graduated in 0.01 ml divisions.
- 5. PROCEDURE
 - 5.1 ***Standardisation of the silver nitrate solution***
 - 5.2 ***Blank Test***
 - 5.3 ***Check test***
 - 5.4 ***Determination***
- 6. EXPRESSION OF RESULTS

G.

Determination of Copper

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
 - 3.1 Hydrochloric acid (density at 20°C = 1.18 g/ml).
 - 3.2 Hydrochloric acid, 6 M solution.
 - 3.3 Hydrochloric acid, 0.5 M solution.
 - 3.4 Ammonium nitrate.
 - 3.5 Hydrogen peroxide, 30%.
 - 3.6 Copper solution (stock): weigh, to the nearest 0.001 gram, 1...
- 4. APPARATUS
- 5. PROCEDURE
 - 5.1 ***Preparation of the solution for analysis***
 - 5.2 ***Blank solution***
 - 5.3 ***Determination***
 - 5.4 ***Measurement***
- 6. EXPRESSION OF THE RESULTS

PART II

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1. **General**
2. **Reagents and Apparatus**
3. **Methods of Analysis**

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1.

Preparation of the sample for analysis

2.

Determination of moisture

3.

Determination of total nitrogen — chromium powder reduction method

4.

Determination of urea

5.a

Extraction of phosphorus — by mineral acids (total phosphorus)

b

Extraction of phosphorus — by 2% citric acid

6.

Determination of extracted phosphorus — spectrophotometric method

7.a

Determination of potassium — gravimetric method

b

Determination of potassium — flame photometric method

8.

Determination of total magnesium

9.a

Determination of boron — titrimetric method

b

Determination of boron — spectrophotometric method

10.

Determination of cobalt

11.

Determination of molybdenum

12.

Determination of copper

2. SCOPE AND FIELD OF APPLICATION
3. PRINCIPLE
- 3.1 Solid fertilisers: the whole final sample is ground to the...
- 3.2 Fluid fertilisers: the final sample is thoroughly mixed before each...
4. APPARATUS
- 4.1 Sample grinder capable of grinding the fertiliser to pass the...
- 4.2 Mortar and pestle of suitable material and size.
- 4.3 Sieves having square apertures of 0.18 mm, 0.5 mm and...
- 4.4 Sample containers of non-corrodible materials, with air-tight closures.
5. PROCEDURE
- WARNING
- 5.1 ***Grinding and sieving***
- 5.2 Place the prepared sample in a clean container (4.4) and...
- 5.3 Before taking each test portion for analysis, the whole sample...
- 5.4 If the sample contains foreign matter which cannot be ground...
6. SPECIAL CASES
- 6.1 ***Samples not to be ground***
- 6.2 ***Products which may be difficult to grind mechanically, including products with abnormal moisture or products which become doughy through grinding***
- 6.3 ***Organic materials***
- 6.4 ***Fertilisers comprising several different materials***
7. FLUID FERTILISERS

2.

DETERMINATION OF MOISTURE

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. APPARATUS
- 3.1 Suitable containers with lids ensuring air-tight closure; the dimensions should...
- 3.2 Electrically heated oven, suitably ventilated and capable of being maintained...
4. PREPARATION OF SAMPLE
5. PROCEDURE
6. EXPRESSION OF THE RESULT

3.

DETERMINATION OF TOTAL NITROGEN — CHROMIUM POWDER REDUCTION METHOD

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. REAGENTS
- 3.1 Sodium hydroxide solution: 40 g per 100 ml, ammonia free....
- 3.2 Sulphuric acid, 0.1 N solution.
- 3.3 Sulphuric acid, 0.2 N solution.
- 3.4 Sulphuric acid, 0.5 N solution.
- 3.5 Sodium hydroxide, 0.2 N solution, carbonate free.
- 3.6 Chromium metal powder, 100 mesh, low nitrogen content.

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- 3.7 Anti-bump granules of pumice stone, washed in hydrochloric acid and...
- 3.8 Anti-foaming agent, paraffin wax.
- 3.9 Sulphuric acid (d = 1.84 g/ml).
- 3.10 Hydrochloric acid (d = 1.18 g/ml).
- 3.11 Catalyst mixture: 1,000 g potassium sulphate and 50 g copper...
- 3.12 Indicator solutions: Mixed indicator: (3.12.1) mix 50 ml of 2...
- 3.13 pH indicator paper, wide range.
- 4. APPARATUS
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
- 6.1 **Reduction**
- 2.0 g of the prepared sample, containing not more than 0.06...
- 6.2 **Hydrolysis, when the fertiliser is known not to contain organic matter**
- 6.3 **Digestion, when the fertiliser is known to contain organic matter**
- Note:**
- 6.4 **Distillation**
- 6.5 **Blank test**
- 7. EXPRESSION OF THE RESULTS

4.

DETERMINATION OF UREA

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Activated charcoal.
- 3.2 Carrez solution I: dissolve 21.9 g zinc acetate dihydrate in...
- 3.3 Carrez solution II: 10.6 g potassium ferrocyanide per 100 ml...
- 3.4 Hydrochloric acid solution, 0.02 N.
- 3.5 Sodium acetate solution: 136 g sodium acetate trihydrate per litre....
- 3.6 4-dimethylamino-benzaldehyde solution: dissolve 1.6 g of 4-dimethylamino-benzaldehyde (4-DMAB) in 100...
- 3.7 Urea standard solution: 1.0 g per 100 ml (1...
- 4. APPARATUS
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
- 6.1 **Preparation of the solution for analysis**
- 6.2 **Determination**
- 6.3 **Calibration curve**
- 7. EXPRESSION OF THE RESULTS

5a.

EXTRACTION OF PHOSPHORUS BY MINERAL ACIDS (TOTAL PHOSPHORUS)

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Sulphuric acid (d = 1.84 g/ml).
- 3.2 Nitric acid (d = 1.42 g/ml).
- 4. APPARATUS

- 4.1 A Kjeldahl flask, with a capacity of at least 500...
- 5. PREPARATION OF THE SAMPLE
- 6. PROCEDURE
- 6.1 **Extraction**
- 6.2 **Determination**
- Note**

5b.

EXTRACTION OF PHOSPHORUS BY 2% CITRIC ACID

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENT
- 3.1 2% citric acid solution (20 g per litre), prepared from...
- 4. APPARATUS
- 4.1 Rotary shaker: 35 — 40 turns per minute.
- 5. PREPARATION OF THE SAMPLE
- 6. PROCEDURE
- 6.1 **Extraction**
- 6.2 **Determination**

6.

DETERMINATION OF EXTRACTED PHOSPHORUS — SPECTROPHOTOMETRIC METHOD

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Nitric acid (d =.42 g/ml).
- 3.2 Molybdovanadate reagent: dissolve separately 20 g ammonium molybdate and 0.47...
- 3.3 Phosphorus standard solution: dissolve 4.387 g potassium dihydrogen phosphate, previously...
- 3.4 Sodium hydroxide, approximately 5 N solution.
- 5. APPARATUS
- 5. PROCEDURE
- 5.1 **Determination**
- 5.2 **Calibration**
- 6. EXPRESSION OF THE RESULTS

7a.

DETERMINATION OF POTASSIUM — GRAVIMETRIC METHOD

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Formaldehyde, 25 – 35% solution, filtered if necessary before use....
- 3.2 Potassium chloride.
- 3.3 Sodium hydroxide, 10 N solution. Care should be taken to...
- 3.4 Indicator solution: Dissolve 0.5 g phenolphthalein in 100 ml 90%...
- 3.5 EDTA solution: 4 g of the dihydrated disodium salt of...
- 3.6 STPB solution: dissolve 32.5 g sodium tetraphenylborate in 480 ml...

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- 3.7 Liquid for washing: dilute 20 ml of the STPB solution...
- 3.8 Hydrochloric acid (d = 1.18 g/ml).
- 4. APPARATUS
- 4.1 Filter crucibles with a porosity of 5 to 20 microns....
- 4.2 Oven regulated at 120°C + 10°C.
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
- 6.1 ***Preparation of the solution for analysis***
- 6.2 ***Determination***
- 6.3 ***Weighing the crucible***
- 6.4 ***Precipitation***
- 6.5 ***Filtering and washing***
- 6.6 ***Drying and weighing***
- 6.7 ***Blank test***
- 6.8 ***Control test***
- 7. EXPRESSION OF THE RESULTS

7b.

DETERMINATION OF POTASSIUM — FLAME PHOTOMETRIC METHOD

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Ammonia solution (30% V/V): dilute 30 ml concentrated ammonia solution...
- 3.2 Ammonium oxalate solution: saturated aqueous solution.
- 3.3 Hydrochloric acid (d = 1.18 g/ml).
- 3.4 Potassium dihydrogen phosphate: dried for one hour at 105°C.
- 3.5 Potassium solution (stock): dissolve 3.4807 g potassium dihydrogen phosphate (3.4)...
- 3.6 Potassium solution (dilute): dilute 50 ml stock solution (3.5) to...
- 4. APPARATUS
- 4.1 Flame photometer.
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
- 6.1 ***Preparation of the solution for analysis***
- 6.2 ***Blank solution***
- 6.3 ***Determination***
- 6.4 ***Measurement***
- 7. EXPRESSION OF THE RESULTS

8.

DETERMINATION OF TOTAL MAGNESIUM

- 8.1 EXTRACTION OF TOTAL MAGNESIUM
- 1. SCOPE AND FIELD OF APPLICATION
- 1.1 This method is applicable to all fertilisers.
- 2. PRINCIPLE
- 2.1 Solubilisation by boiling in dilute hydrochloric acid.
- 3. REAGENTS
- 3.1 Diluted hydrochloric acid: One volume of hydrochloric acid (d =...
- 4. APPARATUS

- 4.1 Electric hot plate with adjustable temperature.
- 5. PREPARATION OF THE SAMPLE
- 5.1 See Method 1.
- 6. PROCEDURE
- 6.1 **Test sample**
- 6.2 **Preparation of the solution**

8.2

DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 2.1 Determination of magnesium by atomic absorption spectrophotometry after appropriate dilution...
- 3. REAGENTS
- 3.1 Hydrochloric acid, 1 M solution.
- 3.2 Hydrochloric acid, 0.5 M solution.
- 3.3 Standard solution of magnesium, 1.00 mg/ml. (3.3.1) Dissolve 1.013 grams...
- 3.4 **Strontium chloride solution**
- 4. APPARATUS
- 4.1 Spectrophotometer fitted for atomic absorption, with a magnesium lamp, set...
- 4.2 Air-acetylene flame.
- 5. PREPARATION OF THE SAMPLE
- 5.1 See Method 8.1
- 6. PROCEDURE
- 6.1 **Test sample**
- 6.2 **Preparation of the solution**

8.2

DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 2.1 Determination of magnesium by atomic absorption spectrophotometry after appropriate dilution...
- 3. REAGENTS
- 3.1 Hydrochloric acid, 1 M solution.
- 3.2 Hydrochloric acid, 0.5 M solution.
- 3.3 Standard solution of magnesium, 1.00 mg/ml. (3.3.1) Dissolve 1.013 grams...
- 3.4 **Strontium chloride solution**
- 4. APPARATUS
- 4.1 Spectrophotometer fitted for atomic absorption, with a magnesium lamp, set...
- 4.2 Air-acetylene flame.
- 5. PREPARATION OF THE SAMPLE
- 5.1 See Method 8.1
- 6. PROCEDURE
- 6.1 If the fertiliser has a declared magnesium (Mg) content of...
- 6.2 Using a pipette, take 10 ml of the extraction solution...

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- 6.3 Dilute this solution (6.2) with the 0.5 M hydrochloric acid...
- 6.4 **Preparation of blank solution**
- 6.5 **Preparation of calibration solutions**
- 6.6 **Measurement**
- 7. EXPRESSION OF RESULTS

9a.

DETERMINATION OF BORON — TITRIMETRIC METHOD

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
 - 3.1 Calcium oxide.
 - 3.2 Mannitol.
 - 3.3 Sodium carbonate.
 - 3.4 Hydrochloric acid solution 50% (V/V): dilute 50 ml concentrated hydrochloric...
 - 3.5 Hydrochloric acid, 0.5 N solution.
 - 3.6 Lead nitrate solution, 10 g per 100 ml.
 - 3.7 Sodium hydroxide, 0.5 N solution.
 - 3.8 Sodium hydroxide, 0.05 N solution, carbonate free.
 - 3.9 Methyl red indicator solution: dissolve 0.1 g of methyl red...
 - 3.10 Phenolphthalein indicator solution: dissolve 0.25 g phenolphthalein in 1.50 ml...
- 4. APPARATUS
 - 4.1 pH meter.
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
 - 6.1 **Preparation of the solution for analysis**
 - 6.2 **Determination**
- 7. EXPRESSION OF THE RESULT

9b.

DETERMINATION OF BORON — SPECTROPHOTOMETRIC METHOD

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
 - 3.1 Calcium oxide.
 - 3.2 Sulphuric acid (d = 1.84 g/ml).
 - 3.3 Carminic acid solution: dissolve 0.025 g carminic acid in sulphuric...
 - 3.4 Hydrochloric acid solution 20% (V/V): dilute 20 ml hydrochloric acid...
 - 3.6 Hydrazine hydrate (approximately 60% W/W solution).
WARNING:
- 4. APPARATUS
 - 4.1 Spectrophotometer with 10 mm cells.
- 5. PREPARATION OF THE SAMPLE
- 6. PROCEDURE
 - 6.1 **Preparation of the solution for analysis**
 - 6.2 **Blank test**
 - 6.3 **Determination**

- 6.4 **Calibration curve**
 - 7. EXPRESSION OF THE RESULTS
- Note**

10.

DETERMINATION OF COBALT

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
 - 3.1 Sodium sulphate, anhydrous.
 - 3.2 Toluene.
 - 3.3 Hydrochloric acid, 2 N solution.
 - 3.4 Hydrochloric acid solution, 50% (V/V): dilute 50 ml concentrated hydrochloric...
 - 3.5 Hydrogen peroxide solution, 3% (10 volume).
 - 3.6 Nitric acid solution, 30% (V/V): dilute 30 ml nitric acid...
 - 3.7 2-nitroso-1-naphthol solution: dissolve 1 g of 2-nitroso-1-naphthol in 100 ml...
 - 3.8 Sodium citrate solution: 40 g per 100 ml.
 - 3.9 Sodium hydroxide, 2 N solution.
 - 3.10 Cobalt solution (stock): weigh to the nearest 0.001 g, 0.670...
 - 3.11 Cobalt solution (working standard): dilute the stock cobalt solution (3.10)...
- 4. APPARATUS
 - 4.1 Spectrophotometer with 10 mm cells.
- 5. PREPARATION OF THE SAMPLE
- 6. PROCEDURE
 - 6.1 **Preparation of the solution for analysis**
 - 6.2 **Determination**
 - 6.3 **Calibration Curve**
- 7. EXPRESSION OF THE RESULTS

11.

DETERMINATION OF MOLYBDENUM

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
 - 3.1 Hydrochloric acid, 50% (V/V): dilute 50 ml concentrated hydrochloric acid...
 - 3.2 Hydrochloric acid, 2 N solution.
 - 3.3 Hydrochloric acid, N solution.
 - 3.4 Nitric acid solution, 30% (V/V): dilute 30 ml nitric acid...
 - 3.6 Ammonium ferrous sulphate solution, 4 g per 100 ml.
 - 3.7 Potassium thiocyanate solution, 40 g per 100 ml.
 - 3.8 Sodium sulphate, anhydrous.
 - 3.9 Stannous chloride solution: suspend 40 g stannous chloride dihydrate in...
 - 3.10 Solvent mixture: mix equal volumes of carbon tetrachloride and 3-methylbutan-1-ol....
- 4. APPARATUS

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- 4.1 Spectrophotometer with 10 mm cells.
- 5. PREPARATION OF THE SAMPLE
- 6. PROCEDURE
- 6.1 ***Preparation of the solution for analysis***
- 6.2 ***Determination***
- Note:***
- 6.3 ***Note:***
- 7. EXPRESSION OF THE RESULTS

12.

DETERMINATION OF COPPER

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Hydrochloric acid (d = 1.18 g/ml).
- 3.2 Hydrochloric acid, 6 N solution.
- 3.3 Hydrochloric acid, 0.5 N solution.
- 3.4 Hydrogen peroxide, approximately 100 volume, 30% by weight.
Copper solution...
- 4. APPARATUS
- 4.1 Atomic absorption spectrophotometer with a copper lamp (324.8 nm).
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
- 6.1 ***Preparation of the solution for analysis***
- 6.2 ***Blank Solution***
- 6.3 ***Determination***
- 6.4 ***Measurement***
- 7. EXPRESSION OF THE RESULTS

13.

DETERMINATION OF IRON

- 1. SCOPE AND FIELD OF APPLICATION
- 2. PRINCIPLE
- 3. REAGENTS
- 3.1 Hydrochloric acid (d = 1.18 g/ml).
- 3.2 Hydrochloric acid, 6 N solution.
- 3.3 Hydrochloric acid, 0.5 N solution.
- 3.4 Hydrogen peroxide, approximately 100 volume, 30% by weight.
- 3.6 Lanthanum chloride solution: dissolve 12 g lanthanum oxide in 150...
- 4. APPARATUS
- 4.1 Atomic absorption spectrophotometer with an iron lamp (248.3 nm).
- 5. PREPARATION OF SAMPLE
- 6. PROCEDURE
- 6.1 ***Preparation of the solution for analysis***
- 6.2 ***Blank solution***
- 6.3 ***Determination***
- 6.4 ***Measurement***
- 7. EXPRESSION OF THE RESULTS

14.

DETERMINATION OF MANGANESE

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. REAGENTS
 - 3.1 Hydrochloric acid (d = 1.18 g/ml).
 - 3.2 Hydrochloric acid, 6 N solution.
 - 3.3 Hydrochloric acid, 0.5 N solution. Manganese solution (stock):
 - 3.5 Lanthanum chloride solution: dissolve 12 g lanthanum oxide in 150...
4. APPARATUS
 - 4.1 Atomic absorption spectrophotometer with a manganese lamp (279.5 nm).
5. PREPARATION OF SAMPLE
6. PROCEDURE
 - 6.1 **Preparation of the solution for analysis**
 - 6.2 **Blank solution**
 - 6.3 **Determination**
 - 6.4 **Measurement**
7. EXPRESSION OF THE RESULTS

15.

DETERMINATION OF THE NEUTRALISING VALUE IN LIMING MATERIALS

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. REAGENTS
 - 3.1 Hydrochloric acid, 0.5 N solution.
 - 3.2 Sodium hydroxide, 0.5 N solution of carbonate free).
 - 3.3 Phenolphthalein indicator solution: dissolve 0.25 g phenolphthalein in 150 ml...
4. PREPARATION OF SAMPLE
5. PROCEDURE
 - 5.1 **Determination**
6. EXPRESSION OF THE RESULTS

16.

DETERMINATION OF FINENESS OF PRODUCTS OTHER THAN POTASSIC BASIC SLAG

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. APPARATUS
4. PROCEDURE
 - 4.1 **For sieving through 3.55 mm, 1.0 mm and 150 micron sieves**
 - 4.2 **For sieving through 6.7 mm, 6.3 mm and 5 mm sieves**
 - 4.3 **For sieving through a 45 mm sieve**
 - 4.4 **Sieving**
5. EXPRESSION OF THE RESULTS

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DETERMINATION OF FINENESS OF POTASSIC BASIC SLAG

1. SCOPE AND FIELD OF APPLICATION
2. PRINCIPLE
3. APPARATUS
4. PROCEDURE
 - 4.1 ***Preparation of the sample***
 - 4.2 ***Sieving***
5. EXPRESSION OF THE RESULTS
 - APPENDIX TO SCHEDULE 2
 - KEY TO FIGURE 1
 - KEY TO FIGURE 2
 - KEY TO FIGURE 3
 - KEY TO FIGURE 4
 - KEY TO FIGURE 5
 - KEY TO FIGURE 6
 - KEY TO FIGURE 7
 - KEY TO FIGURE 8

SCHEDULE

3

Explanatory Note