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.

PREPARATION OF THE SAMPLE FOR ANALYSIS

2.

DETERMINATION OF AMMONIACAL NITROGEN

3a.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — ULSCH METHOD

3b.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — ARND METHOD

3c.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — DEVARDA METHOD

4a

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

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

6.

DETERMINATION OF CYANAMIDE NITROGEN

7.

DETERMINATION OF BIURET IN UREA

8a.

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

4.24 Hydrochloric acid, 0.1 N solution.
4.25 Urease solution:
4.26 Xanthydrol 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....

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 unease in 100 ml...
4.23 Xanthydrol 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

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

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.

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

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 ± 10°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 (MgSO4.7H2O) in...
4.4 Strontium chloride solution: dissolve 15 g strontium chloride (SrCl2.6H2O) in...

5. APPARATUS

5.1 Atomic absorption spectrophotometer with a magnesium lamp (285.2 nm).

6. PREPARATION OF SAMPLE
7. PROCEDURE

7.1 ***Extraction***

2b.

DETERMINATION OF WATER-SOLUBLE MAGNESIUM —EDTA METHOD

13a.

DETERMINATION OF TOTAL MAGNESIUM -ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

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.

4.2 Nitric acid, 10 N solution.
4.3 Indicator solution: dissolve 40 g of ferric ammonium sulphate [Fe2(SO4)3.(NH4)2SO4.24H2O]...
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

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)....
Note:
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 (Ba(OH)2.8H2O),...

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...

F.

Determination of the Chlorine Content (as Chloride Ion)

G.

Determination of Copper

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

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.

DETERMINATION OF MOISTURE

3.

DETERMINATION OF TOTAL NITROGEN — CHROMIUM POWDER REDUCTION METHOD

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 .O 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...

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...

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***

10.

DETERMINATION OF COBALT

11.

DETERMINATION OF MOLYBDENUM

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

17.

DETERMINATION OF FINENESS OF POTASSIC BASIC SLAG