SCHEDULE 2

METHODS OF ANALYSIS

PART II

General

1. When two or more methods are prescribed in this part of this Schedule to determine a component of a fertiliser the choice of the method shall, except where otherwise indicated, be left to the agricultural analyst concerned; the method used must, however, be indicated in the certificate of analysis.

Reagents

2. Except where otherwise specified in the method of analysis, all reagents shall be of analytical quality. Where trace elements are to be determined, the purity of the reagents used shall be checked by means of a blank test.

Water

(a) (a) Except where otherwise specified, a reference in this Part of this Schedule to water shall be a reference to demineralized or distilled water.

(b) For the determination of any form of nitrogen, water shall be free of all nitrogenous compounds and carbon dioxide.

(c) Except where the method of analysis specifies a particular solvent or diluent, all dissolution, dilution, rinsing and washing operations mentioned in the methods of analysis shall be carried out using water.

Apparatus

(a) (a) Only special instruments and apparatus and specifically required apparatus and equipment are mentioned in the methods of analysis.

(b) Apparatus and equipment shall be clean.

(c) The accuracy of graduated glassware shall be assured by reference to the appropriate standards.

Methods of Analysis

5

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. Determination of potassium — gravimetric method

6. Determination of the neutralising value in liming materials

7. Determination of fineness of products other than potassic basic slag

8. Determination of fineness of potassic basic slag

9. Determination of fineness of certain lime products by wet sieving.

1.

PREPARATION OF THE SAMPLE FOR ANALYSIS

INTRODUCTION

1. The preparation of a sample for analysis from the final sample received at the laboratory is a series of operations, usually sieving, grinding and mixing, carried out in such a way that the smallest amount weighed, as prescribed by the method of analysis chosen, is representative of the final sample. The sample should be ground to the fineness required by the method of analysis. (Over grinding must be avoided in cases where this will affect the solubility in various reagents). With some materials, fine grinding may lead to loss or gain of moisture and allowance for this must be made.

SCOPE AND FIELD OF APPLICATION

2. This method is applicable to fertilisers in Groups 1(b), l(c), 2(b), 2(c), 2(d), 3(b), 3(c), 4(a), 4(b), 4(c) and 5(b) of Section A and Group 5 and 6 of Section B of the Table in Schedule 1 of the Fertilisers Regulations 1991.

This method is also applicable to fluid fertilisers.

The determination of the fineness of fertilisers is carried out on the sample as received.

PRINCIPLE

3

Solid fertilisers:

3.1 the whole final sample is ground to the required fineness. All the ground sample is thoroughly mixed before each test portion is taken.

Fluid fertilisers:

3.2 the final sample is thoroughly mixed before each test portion is taken.

APPARATUS

4

4.1 Sample grinder capable of grinding the fertiliser to pass the specified sieve.

4.2 Mortar and pestle of suitable material and size.

4.3 Sieves having square apertures of 0.18 mm, 0.5 mm and 1.0 mm. Test sieves conforming to British Standard 410: 1986 are suitable.

4.4 Sample containers of non-corrodible materials, with air-tight closures.

PROCEDURE

5

WARNING

All operations connected with this procedure should be carried out as quickly as possible to minimise absorption or loss of water. Care should be taken during grinding that the temperature of the fertiliser does not rise above 45°C to avoid loss of volatile constituents. Grinding beyond the fineness required must in all cases be avoided.

5.1 Grinding and sieving

The procedure in 5.1.1 should be followed except when a grinding machine is not available, in which case 5.1.2 is applicable.

(5.1.1) Grind the final sample until all the sample has passed through, or for the specified time, depending on the type of grinder (4.1). To check that the grinding has been adequate, sieve a small portion of the ground sample through a 0.5 mm sieve (4.3) and discard it. If the whole of this portion does not pass the sieve, return the remainder of the sample to the grinder and repeat the grinding until satisfactory grinding is achieved.

(5.1.2) Sieve the whole final sample through the 0.5 mm sieve (4.3). Grind the residue on the sieve, using the pestle and mortar (4.2), until all the material passes through the sieve. Carefully mix the sample.

5.2 Place the prepared sample in a clean container (4.4) and seal it until required for analysis.

5.3 Before taking each test portion for analysis, the whole sample must be well mixed. Form the material into a flattened cone and using a spatula take the required test portion at random in small increments.

5.4 If the sample contains foreign matter which cannot be ground this shall be removed, weighed and allowed for in the results of the analysis. This material shall be retained and if possible its nature recorded.

SPECIAL CASES

6

Samples not to be ground

6.1 For those samples where the fineness of grinding is to be determined it shall be carried out on an unground sample. The sample should be well mixed (soft lumps may be disintegrated by lightly crushing) and divided into two parts, which are as identical as possible. All other determinations shall be carried out on the sample prepared in accordance with the directions in paragraph 5.1.

Products which may be difficult to grind mechanically, including products with abnormal moisture or products which become doughy through grinding

6.2 Some products such as superphosphate may become doughy if ground mechanically. In these cases crush the sample in a mortar (4.2) so that all the material passes through a 1.0 mm sieve (4.3). Place the material so crushed in a clean container (4.4) and seal it until required for analysis.

Organic materials

6.3 Some organic materials may be of such a nature that the procedures given above cannot be used (for example fresh guano, leather, wool and animal residues). In these cases the analyst should use the best practicable means to obtain a representative sample.

Fertilisers comprising several different materials

6.4 These fertilisers include materials with marked differences in texture or mechanical properties (hardness, density, etc). They may be difficult to grind entirely (for example mixtures of organic and inorganic materials) or they may segregate during handling (for example "Kalimagnesia"). Special procedures are necessary in these cases:

(6.4.1) for mixtures other than those in 6.4.2, follow the procedure in 5.1.1, replacing the 0.5 mm sieve by one with apertures of 0.18 mm. A grinding machine, capable of grinding the whole of the sample to the required fineness in one pass, is strongly recommended;

(6.4.2) in the case of mixtures containing one or more very hard components, or mixtures containing organic materials, it may be difficult to grind and homogenise all the components. To avoid overgrinding some of the softer components proceed as follows: —

grind the sample as in 5.1.1 or 5.1.2 to pass a 0.5 mm sieve. Re-sieve the sample through a 0.18 mm sieve and reduce the residue to a convenient size by further grinding or other practical means. Thoroughly remix the sample and place in a clean container (4.4).

FLUID FERTILISERS

7. Mix thoroughly by shaking, ensuring that any insoluble matter, particularly crystalline material, is thoroughly dispersed, immediately before drawing a portion of the sample for analysis.

2.

DETERMINATION OF MOISTURE

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers where a correction for moisture is necessary.

PRINCIPLE

2. The sample is dried to constant weight in an oven at 100°C. The loss in weight corresponds to the moisture content of the sample.

APPARATUS

3

3.1 Suitable containers with lids ensuring air-tight closure; the dimensions should allow the sample to be spread at about 0.3 g per cm_2.

3.2 Electrically heated oven, suitably ventilated and capable of being maintained at 100°C.

PREPARATION OF SAMPLE

4. See Method 1.

PROCEDURE

5. Weigh to the nearest 0.001 g, 5 g of the prepared sample and transfer to a previously weighed container (3.1). Place the uncovered container and the lid in the oven (3.2) for 2 to 3 hours. Replace the lid on the container, remove from the oven and allow to cool in a desiccator and weigh. Reheat for another hour, cool and reweigh. If the difference in weight exceeds 0.01 g continue the heating and cooling procedure until a weight constant within 0.01 g is attained.

EXPRESSION OF RESULT

6. Calculate the total loss of weight and express it as a percentage of the original weight.

3.

DETERMINATION OF TOTAL NITROGEN CHROMIUM POWDER REDUCTION METHOD

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Groups 1(b), 1(c), 3(b), 4(a) and 4(c) of Section A, Group 5 and 6 of Section B and Groups 1(c) and 1(d) of Section C of the Table in Schedule 1 of the Fertilisers Regulations 1991 in respect of which a declaration of total nitrogen is required.

PRINCIPLE

2. The nitrate is reduced to ammonia by chromium powder in an acid medium. Organic and ureic nitrogen are converted into ammonium sulfate by digestion with concentrated sulfuric acid using a catalyst. The ammonia is distilled from an alkaline solution and absorbed in a standard acid. The excess acid is titrated with standard alkali.

REAGENTS

3

3.1 Sodium hydroxide solution: 40 g per 100 ml, ammonia free.

3.2 Sulfuric acid, 0.05 M solution.

3.3 Sulfuric acid, 0.1 M solution.

3.4 Sulfuric acid, 0.25 M solution.

3.5 Sodium hydroxide, 0.2 M solution, carbonate free.

3.6 Chromium metal powder, 100 mesh, low nitrogen content.

3.7 Anti-bump granules of pumice stone, washed in hydrochloric acid and ignited.

3.8 Anti-foaming agent, paraffin wax.

3.9 Sulfuric acid ($\rho = 1.84$ g/ml).

3.10 Hydrochloric acid ($\rho = 1.18$ g/ml).

3.11 Catalyst mixture: 1,000 g potassium sulfate and 50 g copper sulfate pentahydrate. The ingredients must be ground and thoroughly mixed.

3.12 Indicator solutions:

(3.12.1) Mixed indicator:

mix 50 ml of 2 g/litre ethanolic solution of methyl red with 50 ml of 1 g/litre ethanolic solution of methylene blue.

(3.12.2) Methyl red indicator:

dissolve 0.1 g methyl red in 50 ml ethanol . This indicator may be used instead of the preceding one.

3.13 pH indicator paper, wide range.

APPARATUS

4. Apparatus for mineral acid digestion and distillation according to Kjeldahl's method.

PREPARATION OF SAMPLE

5. See Method 1.

PROCEDURE

6

Reduction

6.1 Weigh, to the nearest 0.001 g, between 0.5 and 2.0 g of the prepared sample, containing not more than 0.06 g nitric nitrogen and 0.235 g total nitrogen and transfer to a Kjeldahl flask. Add sufficient water to make the total volume 35 ml. Allow the flask to stand for 10 minutes with occasional gentle swirling to ensure solution of all nitrate salts.

Add 1.2 g chromium powder (3.6) and 7 ml hydrochloric acid (3.10), mix well and allow the flask to stand for at least 5 minutes but not more than 10 minutes at ambient temperature. Heat the flask gently so that the contents just begin to boil in about 7 minutes. Continue boiling gently for 10 minutes. Remove the flask from the heat and allow to cool.

Hydrolysis, when the fertiliser is known not to contain organic matter

6.2 Place the flask (6.1) in a fume cupboard, add a small quantity of anti-bump granules (3.7) and then carefully add 25 ml sulfuric acid (3.9). Mix the contents of the flask and heat gently until boiling. Continue heating until dense white fumes of sulfuric acid are evolved for at least 15 minutes. Allow the mixture to cool and then carefully add 250 ml water. Allow to cool to room temperature and continue as described in 6.4.

Digestion, when the fertiliser is known to contain organic matter

6.3 Add a small quantity of anti-bump granules (3.7), 10 g of the catalyst mixture (3.11) and then carefully add 25 ml sulfuric acid (3.9) (see Note). Add 0.5 g paraffin wax (3.8) to reduce foaming and mix. Heat the flask moderately at first, shaking from time to time until frothing ceases and the liquid is practically colourless. Continue the digestion for at least a further 60 minutes. Allow the mixture to cool and then carefully add 250 ml water. Allow to cool to room temperature, and continue as described in 6.4.

(Note) If organic matter other than urea exceeds 1.0 g add an additional 1.0 ml sulfuric acid for each 0.1 g organic matter in excess of 1.0 g.

Distillation

6.4 Transfer an appropriate volume of 0.1 M, 0.2 M or 0.5 M sulfuric acid (3.2, 3.3, 3.4) to the collecting flask of the distillation apparatus, according to the presumed level of nitrogen; add a few drops of indicator solution (3.12.1 or 3.12.2). Taking precautions against the loss of ammonia, carefully add to the contents of the Kjeldahl flask (6.2 or 6.3) 100 ml sodium hydroxide solution (3.1). Mix well and connect immediately to the distillation apparatus. Heat the flask so that approximately 150 ml of the liquid are distilled in 30 minutes. At the end of this time, lower the collecting flask so that the tip of the condenser is above the surface of the liquid. Test the subsequent distillate by means of the indicator paper (3.13) to ensure that all the ammonia is completely distilled. Remove

the source of heat. Titrate the excess acid with 0.2 M sodium hydroxide solution (3.5) to the end point of the indicator.

Blank test

6.5 Carry out a blank test (omitting only the sample) under the same conditions and allow for this in the calculation of the final results.

EXPRESSION OF RESULTS

7. Determine the quantity of sulfuric acid consumed.

1 ml 0.05 M sulfuric acid, = 0.0014 g nitrogen.

1 ml 0.1 M sulfuric acid, = 0.0028 g nitrogen.

1 ml 0.25 M sulfuric acid, = 0.0070 g nitrogen.

Express the result as the percentage of nitrogen (N) contained in the fertiliser as received for analysis.

4.

DETERMINATION OF UREA

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Group 1(c) of Section A, Group 5 and 6 of Section B and Group 1(d) of Section C of the Table in Schedule 1 of the Fertilisers Regulations 1991.

PRINCIPLE

2. The sample is suspended in acid solution with a clarifying agent and filtered. The urea content of the filtrate is determined after the addition of 4-dimethylaminobenzaldehyde (4-DMAB) by measuring the absorbance at 435 nm.

REAGENTS

3

3.1 Activated charcoal.

3.2 Carrez solution I:

dissolve 21.9 g zinc acetate dihydrate in water, add 3 ml glacial acetic acid and dilute to 100 ml with water.

3.3 Carrez solution II: 10.6 g potassium ferrocyanide per 100 ml.

3.4 Hydrochloric acid solution, 0.02 M.

3.5 Sodium acetate solution: 136 g sodium acetate trihydrate per litre.

3.6 4-dimethylaminobenzaldehyde solution:

dissolve 1.6 g of 4-dimethylaminobenzaldehyde (4-DMAB) in 100 ml 96% ethanol and add 10 ml of hydrochloric acid ($\rho = 1.18$ g/ml)

3.7 Urea standard solution: 1.0 g per 100 ml (1 ml of this solution = 10 mg urea).

APPARATUS

4

4.1 Mechanical shaker.

4.2 Spectrometer with 10 mm cells.

PREPARATION OF SAMPLE

5. See Method 1.

PROCEDURE

6

Preparation of the solution for analysis

6.1 Weigh to the nearest 0.001 g, 2 g of the prepared sample, or a suitable amount expected to contain between 50 and 500 mg of urea and transfer it to a 500 ml graduated flask. Add 150 ml 0.02 M hydrochloric acid solution (3.4), shake for 30 minutes using a mechanical shaker (4.1) then add 10 ml sodium acetate solution (3.5) and mix well. Add 2 g activated charcoal (3.1) to the flask, shake well and allow to stand for a further 15 minutes. Add 5 ml Carrez solution I (3.2), followed by 5 ml Carrez solution II (3.3), mixing well between additions. Dilute to volume with water and mix well. Filter a portion of the solution through a dry filter paper into a clean dry 250 ml beaker.

Determination

6.2 Transfer 10 ml of the filtrate (6.1) to a 50 ml graduated flask, add 10 ml 4-DMAB solution (3.6), dilute to 50 ml with water, mix well and allow to stand for 10 minutes. Measure the absorbance of the solution at 435 nm, in a 10 mm cell against a reference solution prepared by diluting 10 ml 4-DMAB solution (3.6) to 50 ml with water.

Calibration curve

6.3 Transfer amounts of standard urea solution (3.7) corresponding to 50, 100, 150 and 250 mg of urea into a series of 250 ml graduated flask; add 75 ml 0.02 M hydrochloric acid solution (3.4) and proceed as described above (6.1) commencing at ". . . shake for 30 minutes . . .". Measure the absorbance of the solutions and construct a calibration graph relating the absorbances to the amounts of urea present.

EXPRESSION OF RESULTS

7. Determine the amount of urea in the sample by reference to the calibration graph. Express the result in terms of percentage ureic nitrogen of the sample:

(mgurea×0.4665=mgureicnitrogen).

5.

DETERMINATION OF POTASSIUM — GRAVIMETRIC METHOD

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Groups 3(b), 3(c), 3(d) and 4(c) of Section A and Group 5 and 6 of Section B of the Table in Schedule 1 of the Fertilisers Regulations 1991 in respect of which an indication of total potassium is required.

PRINCIPLE

2. The sample is ashed and dissolved in dilute hydrochloric acid or, if it contains no organic substances, it is dissolved directly in dilute hydrochloric acid. After the removal of interfering substances the potassium is precipitated in a slightly alkaline medium in the form of potassium tetraphenylborate (KTPB).

REAGENTS

3

<1 Formaldehyde, 25 – 35% solution, filtered if necessary before use.

3

3.2 Potassium chloride.

3.3 Sodium hydroxide, 10 M solution. Care should be taken to ensure that the sodium hydroxide is free from potassium.

3.4 Indicator solution: dissolve 0.5 g phenolphthalein in 100 ml 90% ethanol.

3.5 EDTA solution: 4 g of the dihydrated disodium salt of ethylenediaminetetraacetic acid (EDTA) per 100 ml. Store this reagent in a plastic container.

3.6 STPB solution: dissolve 32.5 g sodium tetraphenylborate in 480 ml of water, add 2 ml of sodium hydroxide solution (3.3) and 20 ml of a magnesium chloride solution (100 g of $MgCl_2.6H_2O$ per litre). Stir for fifteen minutes and filter through a fine, ashless filter. Store this reagent in a plastic container.

3.7 Liquid for washing: dilute 20 ml of the STPB solution (3.6) to 1 litre with water.

3.8 Hydrochloric acid (ρ =1.18 g/ml).

APPARATUS

4

4.1 Filter crucibles with a porosity of 5 to 20 microns.

4.2 Oven regulated to 120°C10°C.

PREPARATION OF SAMPLE

5. See Method 1.

PROCEDURE

6

Preparation of the solution for analysis

6.1.—(6.1.1) *Fertilisers containing little or no organic matter*

Weigh to the nearest 0.001 g, 2.5 g of the prepared sample and transfer to a 400 ml beaker. Add 50 ml water and 5 ml hydrochloric acid (3.8) and evaporate to dryness on a steam bath. Add 5 ml hydrochloric acid (3.8) and 50 ml water. Bring the contents to the boiling point, breaking down any crystals or lumps with a glass rod. Dilute the solution with water to about 100 ml and boil gently for a few minutes. Allow to cool, transfer to a 250 ml graduated flask, dilute to the mark with water and mix; filter through a dry paper.

(6.1.2) *Fertilisers containing organic matter*

Weigh to the nearest 0.01 g, 10 g of the prepared sample into a suitable crucible and place in a cold muffle furnace. Gradually raise the temperature to about 475°C (do not exceed 500°C). Maintain at this temperature for at least 16 hours and then open the furnace and allow the crucible to cool. Grind the residue to eliminate any lumps, add 50 ml water and 10 ml hydrochloric acid (3.8) and evaporate to dryness on a steam bath. Proceed as in 6.1.1, commencing "Add 5 ml hydrochloric acid (3.8) and 50 ml water.".

Determination

6.2.—(6.2.1) Transfer by pipette an aliquot portion of the filtrate (6.1.1 or 6.1.2), containing 25-50 mg of potassium (30 – 60 mg K_2O) into a 250 ml beaker; make up to 50 ml with water.

(6.2.2) To remove interferences, add 10 ml of the EDTA solution (3.5), several drops of the phenolphthalein solution (3.4) and stir in sodium hydroxide solution (3.3), drop by drop, until the solution turns red, then finally add a few more drops of sodium hydroxide to ensure an excess (usually 1 ml of sodium hydroxide is sufficient to neutralise the sample and ensure an excess).

(6.2.3) To eliminate most of the ammonia boil gently for 15 minutes. Add water to make the volume up to 60 ml. Bring the solution to the boil, remove the beaker from the heat and add 10 ml formaldehyde (3.1). Add several drops of phenolphthalein solution (3.4), and if necessary, more sodium hydroxide solution until a distinct red colour appears. Cover the beaker with a watch glass and place it on a steam bath for fifteen minutes.

Weighing the crucible

6.3 Dry the filter crucible (4.1) to constant weight in the oven at 120°C (4.2) (about 15 minutes).

Allow the crucible to cool in a desiccator and weigh.

Precipitation

6.4 Remove the beaker from the steam bath and stir in drop by drop 10 ml of the STPB solution (3.6). This addition should take about 2 minutes; allow to stand for at least 10 minutes before filtering.

Filtering and washing

6.5 Filter under vacuum into the weighed crucible; rinse the beaker with the liquid for washing (3.7), wash the precipitate three times with the liquid for washing (60 ml in all) and twice with 5 to 10 ml of water.

Drying and weighing

6.6 Wipe the outside of the crucible with a filter paper and place in the oven (4.2) for one and a half hours at a temperature of 120°C. Allow the crucible to cool in a desiccator to ambient temperature and weigh rapidly.

Blank test

6.7 Make a blank test under the same conditions (omitting only the sample) and allow for this in the calculation of the final result.

Control test

6.8 Carry out the determination on an aliquot portion of an aqueous solution of potassium chloride, containing at the most 40 mg of K_2O.

EXPRESSION OF RESULTS

7. Calculate the percentage potassium content of the samples as K_2O, taking into account the weight of the test sample, the volume of the aliquot portion taken for the determination and the value of the blank determination. (Conversion factor, KTPB to K_2O = 0.1314.)

6.

DETERMINATION OF THE NEUTRALISING VALUE IN LIMING MATERIALS

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to products in Groups 5(a) and 5(b) of Section A of the Table in Schedule 1 of the Fertilisers Regulations 1991.

PRINCIPLE

2. The sample is dissolved in a measured quantity of standard hydrochloric acid, the excess of which is titrated with standard solution of sodium hydroxide.

REAGENTS

3

3.1 Hydrochloric acid, 0.5 M solution.

3.2 Sodium hydroxide, 0.5 M solution (carbonate free).

3.3 Phenolphthalein indicator solution: dissolve 0.25 g phenolphthalein in 150 ml 95% ethanol and dilute with water to 250 ml.

PREPARATION OF SAMPLE

4. Rapidly grind 50 g of the representative lime sample to pass through a 1 mm sieve.

PROCEDURE

5

Determination

5.1 Weigh to the nearest 0.001 g, 0.5 g of the prepared sample and transfer to a 300 ml conical flask. Add 50 ml of 0.5 M hydrochloric acid (3.1), cover the flask with a watch glass and boil the contents gently for five minutes. Cool the mixture to room temperature, add two or three drops of the phenolphthalein indicator (3.3) and titrate with 0.5 M sodium hydroxide solution (3.2) to the end point of the indicator.

EXPRESSION OF RESULTS

6. Determine the amount of hydrochloric acid consumed by the sample. 1 ml 0.5 M hydrochloric acid = 0.01402 g calcium oxide (CaO).

The neutralising value is expressed as a percentage by weight of calcium oxide (CaO) and refers to the undried sample as received.

7.

DETERMINATION OF FINENESS OF PRODUCTS OTHER THAN POTASSIC BASIC SLAG

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to "Rock phosphate" in Group 2(b) and to products in Groups 4(c), 5(a) and 5(b) of Section A of the Table in Schedule 1 of the Fertilisers Regulations 1991.

PRINCIPLE

2. By hand sieve shaking, the proportion of material passing through the prescribed sieve is determined.

APPARATUS

3. Sieves having square apertures of 45 mm, 6.7 mm, 6.3 mm, 5 mm, 3.35 mm, l.0 mm and 150 microns; lower receiver to fit sieve. Test sieves conforming to British Standard 410: 1986 are suitable.

PROCEDURE

4

For sieving through 3.5 mm, 1.0 mm and 150 micron sieves

4.1 Thoroughly mix the sample and quarter down until a portion of about 100 g is obtained. Heat this portion at 100°C until dry and thoroughly mix. Weigh to the nearest 0.01 g, 20 g and transfer to the sieve with the lower receiver attached. Proceed as described in 4.4.

For sieving through 6.7 mm, 6.3 mm and 5 mm sieves

4.2 Oven dry the sample of 100°C for 24 hours and thoroughly mix. Weigh to the nearest 0.1 g, 200 g and transfer to the sieve with the lower receiver attached. Proceed as described in 4.4.

For sieving through a 45 mm sieve

4.3 If the sample appears moist or damp, oven dry at 100°C for 24 hours, but if the sample appears dry, heating is not necessary. Thoroughly mix the sample and weigh to the nearest 0.1 g, 500 g and transfer to the sieve with the lower receiver attached. Proceed as in 4.4.

Sieving

4.4 Shake the sieve for 5 minutes, frequently tapping the side. Disintegrate soft lumps such as can be caused to crumble by the application of the fibres of a soft brush, taking care that the hard part of the brush does not make contact with the sieve and that the brush is not used to brush particles through the sieve. Brush out the powder in the lower receiver and weigh. Replace the receiver and

repeat the shaking and tapping procedure for 2 minutes. Add the powder in the receiver to the first portion and weigh. Repeat the process until not more than 0.04 g passes through the sieve during 2 minutes.

EXPRESSION OF RESULTS

5. Calculate the fineness by expressing the weight of the material passing through the sieve as a percentage of the weight of the portion of the dried (or as the case may be, undried) sample taken for sieving.

8.

DETERMINATION OF FINENESS OF POTASSIC BASIC SLAG

SCOPE AND FIELD OF APPLICATION

1. Exclusively to "Potassic basic slag" in Group 3(b) of Section A of the Table in Schedule 1 of the Fertilisers Regulations 1991.

PRINCIPLE

2. By hand sieve shaking and dissolution of the soluble salts, the proportion of slag passing through the prescribed sieve is determined.

APPARATUS

3. Sieve having square apertures of 0.5 mm (500 microns); lower receiver to fit sieve. Test sieves conforming to British Standard 410: 1986 are suitable.

PROCEDURE

4

Preparation of the sample

4.1 Thoroughly mix the sample and quarter down until a portion of about 100 g is obtained. Heat this portion at 100°C until dry and thoroughly mix.

Sieving

4.2 Weigh to the nearest 0.1 g, 20 g of the dry sample and transfer to the sieve with the lower receiver attached. Shake the sieve for five minutes, frequently tapping the sides. Disintegrate soft lumps that can be caused to crumble by the application of a soft brush, taking care that the hard part of the brush does not make contact with the sieve and that the brush is not used to brush particles through the sieve.

Transfer the finer portion from the container into a 500 ml beaker and add 200 ml of previously boiled water. Stir and then filter through a weighed glass sintered crucible. Thoroughly wash the residue with water, dry and re-weigh the crucible. Calculate the weight of slag in the mixture with a particle size of less than 0.5 mm (A).

Weigh to the nearest 0.01 g, about 20 g of the dry sample and transfer to a 500 ml conical flask. Add 200 ml previously boiled water and shake for 30 minutes. Filter through a weighed, sintered glass crucible, wash the residue thoroughly with water, dry and re-weigh the crucible. Calculate the total weight of slag in the mixture (B).

EXPRESSION OF RESULTS

Express the fineness of the slag asABx100.

9.

DETERMINATION OF FINENESS OF CERTAIN LIME PRODUCTS BY WET SIEVING

SCOPE

1. This method is applicable to products in Group 5(b) of Section A of the Table in Schedule 1 of the Fertilisers Regulations 1991 which are susceptible to clogging, caking, electrostatic changes or agglomeration on pre-drying. The method is not applicable to burnt and hydrated lime products.

PRINCIPLE

2. The liming material is suspended in water. The suspension is sieved under continuous water spraying or using a mechanical wet-sieving machine. The fractions retained on the sieves are collected and dried.

APPARATUS

3. Usual laboratory apparatus and in particular:

3.1 Balance, capable of weighing to the nearest 0.01 g

3.2 Stainless steel woven wire test sieves 100 mm diameter, complying with ISO 3310 – 1, with nominal apertures of 5.00 mm, 3.35 mm and 150 microns

3.3 Stainless steel woven wire test sieves complying with ISO 3310 – 1, with nominal apertures of 10.00 mm

3.4 Oven capable of being controlled at 105°C2

3.5 Rotating end over end shaker: 35 – 40 turns per minute.

SAMPLING

4

4.1 Procedure for samples with dry matter content <60%

Pass the laboratory sample through a sieve with nominal apertures of 10.00 mm (3.3). If necessary, lightly crush any lumps by means of a soft brush. Remove any lumps which cannot be crushed in this way and record the weight of the residue and the weight of the lumps. Take account of these lumps when recording the final results. Thoroughly mix the sieved sample and quarter down until a representative sample portion of about 50 g is obtained.

4.2 Procedure for samples with dry matter content<60% which cannot be treated as per 4.1 due to the nature of the material.

Empty the whole of the sample onto a clean dry surface and flatten to form a regular shape about 25 mm thick. Divide into four approximately equal portions and reject two opposite quarters. Take small portions from random places on all the exposed surfaces to give a sample portion of about 50 g.

PROCEDURE

5. Weigh the sample portion (4.1 or 4.2) to the nearest 0.01 g and transfer to a 500 ml flask. Add approximately 300 ml of de-mineralized water, stopper and shake vigorously by hand for 30

seconds. Remove the stopper for an instant to relieve the pressure and replace the stopper. Place the flask in the rotating end over end shaker (3.5) and shake for 60 minutes to ensure the complete suspension of the sample.

Assemble the three sieves (3.2) in ascending order of aperture size on top of the receiver.

Rinse the sample quantitatively onto the top sieve and wash under a flow of water up to 2.5 litres/min until no more material passes each sieve, or up to a maximum time period of 10 minutes.

Remove the sieves and rinse the residue on each sieve quantitatively into a separate 250 ml beaker. Decant most of the water from the top of the material and dry each of the oversize fractions in an oven set at 105 C and weigh each fraction separately.

DRY MATTER CONTENT

6. Determine the dry matter content of a portion of the original sample using the method given in Method 2.

EXPRESSION OF RESULTS

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7.1 Original dry mass

Calculate the original dry mass (Md) of material, using the following formula:

$Md=M\times DM$

where:

M is the mass of the test portion taken for the sieving test

DM is the percentage dry matter obtained in 6.

7.2 Sieve fraction

Calculate the percentage of material retained on each sieve, using the following formula:

$Xn=Mn/Md\times 100$

where:

X_n is the percentage by mass retained on sieve n

M_n is the dry mass retained on sieve n

M_d is the dry mass of the test portion

Report the percentages of material (100 – Xn) which will pass through each sieve.

Carry out two single test on separate test portions prepared from the same original sample. Record the mean of the two individual results for each sieve as the result (corrected if necessary for the presence of lumps (4.1)).