

SCHEDULE 2

METHODS OF ANALYSIS

PART II

7a.

DETERMINATION OF POTASSIUM — GRAVIMETRIC METHOD

1 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 of Section B of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990 in respect of which an indication of total potassium is required.

2 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).

3 REAGENTS

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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 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 ethylenediaminetetra-acetic 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 $MgCl_2 \cdot 6H_2O$ 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 ($d = 1.18$ g/ml).

4 APPARATUS

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

4.2 Oven regulated at $120^\circ C + 10^\circ C$.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

5 PREPARATION OF SAMPLE

5. See Method 1.

6 PROCEDURE

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Preparation of the solution for analysis

Fertilisers containing little or no organic matter

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6.1.—(6.1.1) 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.

Fertilisers containing organic matter

(6.1.2) 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 (not to 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 part of the filtrate (6.1.1 or 6.1.2), containing 25 — 50 mg of potassium (30 — 60 mg K₂O) 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, drop by drop, sodium hydroxide solution (3.3) until it 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 then weigh it.

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 of the liquid for washing) 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 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 part of an aqueous solution of potassium chloride, containing at the most 40 mg of K₂O

7 EXPRESSION OF THE RESULTS

7. Calculate the percentage potassium content of the sample as K₂O, taking into account the weight of the test sample, the volume of the aliquot part taken for the determination and the value of the blank determination. (Conversion factor, KTPB to K₂O = 0.1314).