

SCHEDULE 2

METHODS OF ANALYSIS

PART II

1.

PREPARATION OF THE SAMPLE FOR ANALYSIS

1 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.

2 SCOPE AND FIELD OF APPLICATION

2. This method is applicable to fertilisers in Groups 1(b), 1 (c), 2(b), 2(c), 2(d), 3(b), 3(c), 4(a), 4(b), 4(c) of Section A and Group 5 of Section B of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990⁽¹⁾. It is also applicable to products in Group 5(a) of Section A in the said table when the determination of total magnesium is required.

This method is also applicable to fluid fertilisers.

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

3 PRINCIPLE

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

3.2 *Fluid fertilisers:* the final sample is thoroughly mixed before each test portion is taken.

4 APPARATUS

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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 : 1976 are suitable.

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

(1) [S.R. 1990 No. 286](#)

Status: This is the original version (as it was originally made).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

5 PROCEDURE

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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.

Grinding and sieving

5.1 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 a 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.

6 SPECIAL CASES

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Samples not to be ground

6.1 For those samples where the amount of phosphorus pentoxide soluble in 2% citric acid and the fineness of grinding are to be determined, 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. The above mentioned determinations shall be carried out on the unground sample. 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 practicable means. Thoroughly remix the sample and place in a clean container (4.4).

7 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.