1981 No. 58

AGRICULTURE

Fertilisers (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1981

Made . . . . . *2nd March 1981*

Coming into operation . . . . *10th April 1981*

The Department(**a**) of Agriculture, in exercise of the powers conferred on it by sections 66(1), 74A(**b**), 75(1), 76(1), 77, 78(2), (4), and (6), 79(1), (2), and (9), 84 and 86 of the Agriculture Act 1970(**c**) and of every other power enabling it in that behalf, after consultation with such persons or organisations as appear to it to represent the interests concerned, hereby makes the following regulations:

Citation, commencement and interpretation

1.—(1) These regulations may be cited as the Fertilisers (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1981 and shall come into operation on 10th April 1981.

(2) In these regulations "the principal regulations" means the Fertilisers (Sampling and Analysis) Regulations (Northern Ireland) 1978(**d**).

(3) The Interpretation Act (Northern Ireland) 1954(**e**) shall apply to these regulations as it applies to a Measure of the Northern Ireland Assembly.

Amendment of earlier regulations

2.—(1) The principal regulations shall be amended as set out in regulations 3 to 7.

(2) The 1977 regulations shall be amended as set out in regulation 8.

Definitions added to principal regulations

3. In regulation 1(2) of the principal regulations after the definition of "the Act" there shall be inserted—

""EEC fertiliser" has the meaning assigned to it by regulation 1(2) of the Fertilisers Regulations (Northern Ireland) 1977(**f**).

"the 1977 regulations" means the Fertilisers Regulations (Northern Ireland) 1977.".

New regulation 5 in principal regulations

4. For regulation 5 of the principal regulations there shall be substituted—

"**5.**—(1) The methods by which analysis of fertilisers shall be made for the purposes of the Act shall be those set out in Schedules 2 and 2A.

(2) Analytical constitutents of materials listed in Groups 1(*a*), 1(*b*), 2(*a*) and 3(*a*) of Section A and Groups 1 to 4 of Section B of the table in Schedule 1 to the 1977 regulations shall be determined by the appropriate method of analysis prescribed in Schedule 2.

(**a**) Formerly Ministry: *see* 1973 c. 36 s. 40 and Sch. 5 paragraph 8(1)
(**b**) Inserted by 1972 c. 68 s. 4(1) and Sch. 4 paragraph 6
(**c**) 1970 c. 40
(**d**) S.R. 1978 No. 240
(**e**) 1954 c. 33 (N.I.)
(**f**) S.R. 1977 No. 288 as amended by S.R. 1978 No. 240

(3) In the case of "Phosphated Slag" and "Rock phosphate" in Group 2(*b*) of Section A of the said table in the amount of phosphorus pentoxide soluble in 2% formic acid shall be determined by the method prescribed in Schedule 2.

(4) In the case of "Basic Slag medium concentration" and "Granular basic Slag" in Group 2(*b*) of Section A of the said table fineness shall be determined by the method prescribed in Schedule 2.

(5) In respect of analytical constituents of materials not provided for in paragraphs (2), (3) and (4) the methods of analysis prescribed in Schedule 2A shall apply.".

Amendment of regulation 9 of principal regulations

5. For regulation 9 of the principal regulations there shall be substituted—

"**9.** As respects their application to fertilisers, regulations 3, 4, 14, 15 and 16 of and Parts I, II and IV of Schedule 1, Schedule 6 and Part I of Schedule 8 to the Fertilisers and Feeding Stuffs Regulations (Northern Ireland) 1973(**a**) shall cease to have effect on 10th April 1981.".

New Schedule 2A to principal regulations

6. The provisions of the Schedule to these regulations shall be inserted as Schedule 2A to the principal regulations.

Amendment of Schedule 3 to principal regulations

7. For note (11) to the Form of Certificate of Analysis set out in Schedule 3 to the principal regulations there shall be substituted—

"(11) In the case of analysis of substances for which no analytical method is prescribed by regulation 5 and Schedules 2 and 2A here indicate the method used. If analysis cannot be carried out because no suitable method exists then the certificate should state this.".

Amendment of the 1977 regulations

8. In regulation 2(2) of the 1977 regulations for "organic substance" there shall be substituted "organic nutrients of animal or vegetable origin".

Sealed with the Official Seal of the Department of Agriculture for Northern Ireland on 2nd March 1981.

(L.S.) *W. H. Parker*
Assistant Secretary

(a) S.R. & O. (N.I.) 1973 No. 494 as amended by S.R. 1976 No. 255

SCHEDULE Regulation 5

PROVISIONS TO BE INSERTED AS SCHEDULE 2A TO THE PRINCIPAL REGULATIONS

"SCHEDULE 2A

Methods of Analysis

1. *General*

(*a*) When two or more methods are prescribed in 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.

(*b*) Any reference to water in this Schedule means purified water as defined in the European Pharmacopoeia.

2. *Reagents and apparatus*

(*a*) All reagents used shall be of analytical quality.

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

(*c*) Solutions for which no solvents are prescribed must be aqueous.

(*d*) Only special instruments or apparatus requiring special standards are mentioned in the descriptions of the methods of analysis.

3. *Methods of Analysis*

1. Preparation of the sample for analysis
2. Determination of moisture
3. Determination of total nitrogen — chrome powder reduction method
4. Determination of urea
5*a*. Extraction of phosphorus — by mineral acids (total phosphorus)
5*b*. Extraction of phosphorus — by 2% citric acid
5*c*. Extraction of phosphorus — by water
6*a*. Determination of extracted phosphorus — gravimetric method
6*b*. Determination of extracted phosphorus — spectrophotometric method
7*a*. Determination of potassium — gravimetric method
7*b*. Determination of potassium — flame photometric method
8. Determination of total magnesium
9*a*. Determination of boron — titrimetric method
9*b*. Determination of boron — spectrophotometric method
10. Determination of cobalt
11. Determination of molybdenum
12. Determination of copper
13. Determination of iron
14. Determination of manganese
15. Determination of the neutralising value in liming materials
16. Determination of fineness for products other than potassic basic slag
17. Determination of fineness of potassic basic slag

1. PREPARATION OF THE SAMPLE FOR ANALYSIS

1. INTRODUCTION

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 sample for analysis. 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

This method is applicable to fertilisers in Groups 1(c), 1(d), 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) 1977. 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.

The method is also applicable to liquid fertilisers.

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

3. PRINCIPLE

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

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

4. APPARATUS

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.

5. PROCEDURE

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 laboratory 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 laboratory 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

6.1 *Samples not to be ground*

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.

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

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 and seal it until required for analysis.

6.3 *Organic materials*

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.

6.4 *Fertilisers comprising several different materials*

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.

7. LIQUID FERTILISERS

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

2. DETERMINATION OF MOISTURE

1. SCOPE AND FIELD OF APPLICATION

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

2. PRINCIPLE

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.

3. APPARATUS

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±2°C.

4. PREPARATION OF SAMPLE

See Method 1.