1970. No. 156 **[C]**

FERTILISERS AND FEEDING STUFFS

The Fertilisers and Feeding Stuffs (Amendment) Regulations (Northern Ireland) 1970

REGULATIONS, DATED THE 17TH DAY OF JUNE 1970, MADE BY THE MINISTRY OF AGRICULTURE UNDER THE FERTILISERS AND FEEDING STUFFS ACT 1926.

The Ministry of Agriculture for Northern Ireland, in exercise of the powers vested in it by sections 23 and 29 of the Fertilisers and Feeding Stuffs Act 1926(a) and of every other power enabling it in that behalf, and acting on the advice of the advisory committee appointed for Great Britain under section 23 of the said Act, hereby makes the following Regulations:—

Citation and Commencement

1. These Regulations may be cited as the Fertilisers and Feeding Stuffs (Amendment) Regulations (Northern Ireland) 1970 and shall come into operation on 1st October 1970.

Amendment of the Schedules to the Fertilisers and Feeding Stuffs Regulations (Northern Ireland) 1968

2. Schedules 2 and 4 to the Fertilisers and Feeding Stuffs Act 1926, as substituted by the Fertilisers and Feeding Stuffs Regulations (Northern Ireland) 1968(b), (hereinafter referred to as the "principal Regulations"), and Schedules 6, 8 and 9 to the principal Regulations are hereby amended as follows:—

(*a*) In Part II of Schedule 2,

(i) in the first column, for the words "Dried brewery and distillery grains" there shall be substituted the words "Dried brewery grains";

(ii) immediately after the item referred to in the last preceding sub-paragraph, there shall be inserted the item—

"Dried distillery by-products (other than malt culms and dried yeast)	Amounts of oil and protein, of fibre if present in excess of 2% and of calcium if present in excess of 2%."

(*b*) In Part I of Schedule 4, immediately after the item—

"Horns	The product obtained by crushing or grinding horn, to which no other matter has been added."

there shall be inserted the items—

"Kainit	Mineral potassium salt containing less than 3·6% of magnesium (Mg).
Magnesium kainit	Mineral potassium salt containing at least 3·6% of magnesium (Mg)."

(*c*) In part II of Schedule 4,

(i) the item—

"Dried distillery grains.	The article produced by drying the residues from distillery mash-tuns, to which no other matter has been added."

shall be omitted;

(a) 16 & 17 Geo. 5, c. 45. (b) S.R. & O. (N.I.) 1968, No. 157.

(ii) in the second column, in the item beginning "Feeding meat and bone meal" for the words "hoof and horn" there shall be substituted the words "hoof, horn and feathers";

(iii) in the second column, in the item beginning "Feeding meat meal" for the words "hoof and horn" there shall be substituted the words "hoof, horn and feathers".

(*d*) In Schedule 6,

(i) for Part I there shall be substituted the following—

"

PART I

PROVISIONS APPLICABLE TO BOTH FERTILISERS AND FEEDING STUFFS

A. *General Provisions*

1. In the case of articles in packages, bottles, drums or kegs, only unopened containers shall be selected for the purpose of the sample.

2. Samples shall not be drawn from part of any quantity where such part bears the appearance of having received damage.

3. In every case the sampling shall be done as quickly as is possible, consistent with due care, and the material shall not be exposed any longer than is absolutely necessary.

B. *Provisions Applicable where the Fertiliser or Feeding Stuff is in Solid Condition*

4. Where the weight of the whole quantity does not exceed 2 cwt., or the whole quantity is in one container, the sample may consist of such a portion of the quantity as is fairly representative of the whole, and the sample shall be of not less than $1\frac{1}{2}$ lbs. in weight.

5. In each case it shall be assumed that the quantity is composed of separate approximately equal parts and that the number of such parts is equivalent to:

(*a*) the number of packages to be selected in accordance with paragraph 1(*a*) of Part II of this Schedule, or

(*b*) the number of portions where the quantity is in bulk, to be taken in accordance with paragraph 1(*b*) of Part II of this Schedule.

The packages or portions shall be selected on the basis of at least one from each assumed approximately equal part, and shall be drawn at random.

6. Notwithstanding anything in these Regulations, a sampling spear shall not be used if objection is raised thereto, prior to the taking of a sample, on the ground that the material is unsuitable.

C. *Provisions Applicable where the Fertiliser or Feeding Stuff is in a Liquid or Semi Liquid Condition*

7. (*a*) *In bottles or containers each containing not more than one quart*

The number of bottles or containers to be selected shall be in accordance with the appropriate scale for solid fertilisers in paragraph 1(*a*) of Part II of this Schedule. The entire contents of the selected bottles or containers shall be emptied into a clean, dry vessel of glass or other suitable material and well mixed by stirring or shaking. From this mixture a sample of between about one quart and about half-a-gallon shall be drawn, the mixture being stirred or shaken until immediately before the sample is drawn.

(*b*) *In drums, kegs, or other containers each containing more than one quart and not more than forty gallons*

The number of containers to be selected shall be in accordance with the appropriate scale for solid fertilisers in paragraph 1(*a*) of Part II of this Schedule. The selected containers shall be well shaken or the contents agitated or otherwise treated to ensure uniformity. An approximately equal proportion of fluid shall then be taken immediately from each of the selected containers, emptied into a clean, dry vessel of glass or other suitable material and well mixed by stirring or shaking. From this mixture a sample of between about one quart and about half-a-gallon shall be drawn, the mixture being stirred or shaken until immediately before the sample is drawn.

(*c*) *In a bulk container or containers containing more than forty gallons*

(i) When a consignment is being withdrawn from the bulk container, and there is a tap in the outlet pipe from which it is suitable to draw a sample, a quantity in accordance with the table below shall be drawn from the tap (after first withdrawing sufficient to remove any residues in the pipe), into a clean, dry vessel of glass or other suitable material, made up of portions of not less than one pint and of approximately equal size taken at regular intervals; otherwise

(ii) if the liquid is homogeneous, about one quart shall be drawn from a convenient outlet in the container (after first withdrawing sufficient to remove any residues in the outlet) into a clean, dry vessel of glass or other suitable material, or

(iii) if the liquid is not homogeneous, the contents shall be well stirred or otherwise agitated, and sampling shall then proceed as in sub-paragraph (ii), but

(iv) if it is not possible to make the liquid homogeneous, in the manner described in sub-paragraph (iii), the contents shall be sampled by lowering an open tube (which must be long enough to reach the bottom of the container) perpendicularly into the container. One or both ends of the tube shall then be closed and the contents transferred to a clean, dry vessel of glass or other suitable material. If sampling by tube is impracticable, portions shall be taken from various levels of the container with a sampling bottle, so as to obtain a quantity representative of the whole. The appropriate process shall be repeated until a quantity in accordance with the table below has been withdrawn.

(v) Where a parcel consists of two or more containers, a sample from each, drawn in the manner described in sub-paragraphs (i) to (iv), as appropriate, shall be placed in a clean, dry vessel of glass or other suitable material.

(vi) The quantity taken as described in sub-paragraphs (i), (iv) and (v) shall be thoroughly mixed and a sample of about one quart transferred into a clean, dry vessel of glass or other suitable material.

TABLE

Quantities of Liquid Fertilisers and Feeding Stuffs to be withdrawn in accordance with sub-paragraphs (*c*)(i) *and* (iv) *above*

Where the quantity to be sampled—									Quantity to be withdrawn
does not exceed	1,000	gallons							not less than 2 pints
exceeds	1,000	gallons	but does not exceed		5,000	gallons			,, ,, ,, 3 pints
,,	5,000	,,	,,	,,	,,	,,	10,000	,,	,, ,, ,, 4 pints
,,	10,000	,,	,,	,,	,,	,,	15,000	,,	,, ,, ,, 5 pints
,,	15,000	,,	,,	,,	,,	,,	20,000	,,	,, ,, ,, 6 pints
,,	20,000	,,	,,	,,	,,	,,	50,000	,,	,, ,, ,, 7 pints
,,	50,000	,,	,,	,,	,,	,,	100,000	,,	,, ,, ,, 10 pints
,,	100,000	,,							,, ,, ,, 20 pints"

(ii) in Part II for the heading there shall be substituted the heading "PROVISIONS APPLICABLE TO SOLID FERTILISERS",

(iii) in Part II paragraph 4 shall be omitted and paragraphs 5 and 6 shall be re-numbered 4 and 5 respectively,

(iv) in Part III for the heading there shall be substituted the heading "PROVISIONS APPLICABLE TO SOLID FEEDING STUFFS",

(v) in Part III paragraph 3 shall be omitted and paragraphs 4 and 5 shall be re-numbered 3 and 4 respectively.

(*e*) In Schedule 8,

(i) at the beginning, to the list of the main divisions there shall be added the following items—

"18. Determination of Ethopabate.

19. Determination of Furazolidone.

20. Determination of Dinitolmide.

21. Determination of Calcium."

(ii) in the main division numbered 8.,

(*a*) for the first paragraph—

"For the purposes of the Fertilisers and Feeding Stuffs Act 1926 "sugar" means sucrose."

there shall be substituted the paragraph—

"For the purposes of the Fertilisers and Feeding Stuffs Act 1926 "sugar" means total reducing sugars after inversion expressed as sucrose."

(*b*) in the second paragraph, for the words—

"The sugar is then determined as invert sugar after inversion of sucrose."

there shall be substituted the words—

"The total reducing sugar content is then determined after inversion of the sucrose."

(*c*) in the sub-division numbered 8·223, for the words—

"The total copper reducing power should finally be determined in terms of sugar ($C_{12}H_{22}O_{11}$)."

there shall be substituted the words—

"The total copper reducing power should be calculated as invert sugar and diminished by 1/20th to give the sugar."

(iii) in the main division numbered 16., at the end, there shall be added the following sub-division—

"16·21 NITROFURAZONE IN THE PRESENCE OF FURAZOLIDONE

Carry out the determination of furazolidone according to 19·2. Correct the extinction obtained in the determination of nitrofurazone at 530 nm. by means of the following expression:

Corrected extinction, $E_{cor} = E_{obs} - \frac{34\ Wxy}{2500}$

Where E_{obs} = extinction observed in 16·2

W = weight of sample in grams taken for nitrofurazone method

x = content per cent. of furazolidone found by determination of 19·2

y = number of ml. taken from original dimethylformamide extract of 50 ml. at point 'transfer a suitable portion containing about 200 μg.'."

(iv) at the end, there shall be added the following main divisions and subdivisions—

"18· DETERMINATION OF ETHOPABATE
(methyl 4-acetamido-2-ethoxybenzoate)

18·1 REAGENTS

Ammonium sulphamate solution—Dissolve 1·00 g. ammonium sulphamate in water and dilute to 100 ml.

Butan-1-ol.

Chloroform.

Ethopabate, stock solution—Dissolve 40·0 mg. pure ethopabate in methanol, and dilute to 100 ml. with methanol.

Ethopabate, standard solution—Dilute 10 ml. ethopabate stock solution with 50 per cent. methanol to 100 ml.

Ethopabate, working standard solution—Dilute 5·00 ml. ethopabate standard solution to 250 ml. with 50 per cent. methanol and mix well. 20 ml. of this solution contains 16·0 μg. ethopabate.

Hydrochloric acid, dilute solution A—Dilute 10 ml. concentrated hydrochloric acid (d=1·18) with water to 100 ml.

Hydrochloric acid, dilute solution B—Dilute 25 ml. concentrated hydrochloric acid (d=1·18) with water to 1 litre.

Methanol, 50 per cent. v/v—Dilute one volume methanol to two volumes with water.

N-1-Naphthylethylenediamine dihydrochloride solution, (coupling agent)—Dissolve 50 mg. N-1-naphthylethylenediamine dihydrochloride in 25 ml. water. Prepare freshly as required.

Sodium carbonate solution—Dissolve 40 g. anhydrous sodium carbonate in water and dilute to 1 litre.

Sodium chloride.

Sodium nitrite solution—Dissolve 0·20 g. sodium nitrite in water and dilute to 100 ml. Prepare immediately before use.

18·2 PROCEDURE

Weigh out a portion of the prepared sample between 5 g. and 20 g. (ideally the portion should contain about 80 μg. ethopabate) and transfer it to a glass stoppered 250 ml. flask. Add 100 ml. 50 per cent. methanol and agitate the mixture for one hour. Collect sufficient clear extract for a test by passing the mixture through a fast filter paper or by centrifuging a suitable portion. This extract can be stored overnight at room temperature in a tightly stoppered flask.

Transfer 20·0 ml. of the clear extract into a 50 ml. centrifuge tube, and add 5 ml. dilute hydrochloric acid, solution A. Then add 10 ml. chloroform, close the tube with a polythene stopper and shake vigorously for 3 minutes. Separate into two phases by centrifuging and carefully transfer the lower chloroform layer to a second 50 ml. centrifuge tube. (This is conveniently accomplished by the use of a syringe fitted with a capillary tube.) Re-extract the aqueous portion in the first centrifuge tube with a second 10 ml. chloroform by mixing, centrifuging, and transferring the chloroform phase to the second centrifuge tube as before. Repeat this extraction procedure with a third 10 ml. chloroform and combine the chloroform extracts in the second centrifuge tube. Add 10 ml. sodium carbonate solution to the combined