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

chloroform extracts and shake vigorously for 3 minutes. Centrifuge and, without disturbing the interface, draw off most of the upper layer and discard it. Repeat the washing with another 10 ml. sodium carbonate solution and discard the aqueous layer. Wash the chloroform with 10 ml. water by closing the tube and shaking vigorously for 1 minute. Centrifuge and draw off and discard the aqueous layer. Repeat this washing with a further 10 ml. water. (Note: it is important that the chloroform interface should not be disturbed as loss of drug may occur. Also the extraction and washings should be completed in as short a time as possible, as prolonged contact with hydrochloric acid or sodium carbonate may cause partial hydrolysis of the ethopabate.)

Prepare a reagent blank and a standard in the following way:

Into separate 50 ml. centrifuge tubes transfer by pipette 20·0 ml. 50 per cent. methanol (reagent blank) and 20·0 ml. (16 μg.) ethopabate working standard solution. Add 5·0 ml. hydrochloric acid, dilute solution A to the contents of each tube and carry out the extraction procedure described above, commencing at "Then add 10 ml. chloroform, close the tube with a polythene stopper and .....".

Transfer the washed chloroform extracts obtained from the sample, the reagent blank, and the standard to three 100 ml. beakers. Rinse the centrifuge tubes with two 3 ml. portions of 50 per cent. methanol and add the rinsings to the respective beakers. Evaporate the contents of the beakers on a steam bath to about 2 ml. Add 5·0 ml. 50 per cent. methanol to each beaker and redissolve any solid material which has separated out.

Transfer these solutions quantitatively to three centrifuge tubes and rinse each beaker successively with 10, 10 and 5 ml. portions of hydrochloric acid, dilute solution B, adding the rinsings to the centrifuge tube. Immerse the tubes in a boiling water bath, so that the level of the liquid in the tubes is just below the level of the water in the bath, for 45 minutes. Remove the tubes and cool to 10—15°C.

To each tube add 1·0 ml. sodium nitrite solution, mix, and allow to stand for 2 minutes. Add 1·0 ml. ammonium sulphamate solution, mix, and set aside for 2 minutes. Add 1·0 ml. of N-1-naphthylethylenediamine dihydrochloric coupling agent solution, mix and allow to stand for 10 minutes. Then add 5·0 g. sodium chloride and 5·0 ml. butan-1-ol, stopper the tube and shake it vigorously until the sodium chloride has dissolved. Remove the stoppers and spin the tubes in a centrifuge. Measure the extinction of the butan-1-ol layer from each tube in a 10 mm. cell at 555 nm. against a cell containing pure butan-1-ol.

Calculate the quantity of ethopabate in the feed from the ratios of the extinctions of the sample and standard solutions correcting each reading for the extinction of the blank.

Calculation:

$$\text{Ethopabate in feed, per cent.} = \frac{0{\cdot}008\,(A_x - A_b)}{W\,(A_s - A_b)}$$

Where A_x = optical density of sample solution
A_b = optical density of reagent blank solution
A_s = optical density of standard solution
W = weight in grams of original sample.

19· DETERMINATION OF FURAZOLIDONE

[3-(5-nitrofurfurylideneamino)-oxazolidin-2-one]

19·1 REAGENTS

Acetone.

Aluminium oxide—neutral aluminium oxide suitable for chromatography; activity grade 1; 100-240 mesh. Prepare the aluminium oxide as follows: Slurry 500 g. aluminium oxide with 1 litre of hot distilled water, and decant the supernatant liquid. Repeat this procedure twice more. Dry the aluminium oxide at 105°C. to constant weight before use.

Amyl alcohol.

Amyl acetate.

Petroleum spirit—light petroleum—boiling range 40-60°C. or 60-80°C.

Urea solution—Dissolve 90 g. urea in 100 ml. water.

19·2 PROCEDURE

Weigh a quantity of the feed sample expected to contain between 0·9 and 1·1 mg. furazolidone into a 25×80 mm. extraction thimble and transfer it to a suitable extraction apparatus. Extract with petroleum spirit for half an hour, ensuring 13 to 17 cycles of solvent. Remove the extraction thimble from the apparatus, drain off residual solvent and dry the thimble and the extracted feed in a current of warm air. Place the dried thimble and contents in a clean extraction apparatus and extract with acetone for 1 hour, ensuring not less than 25 cycles of solvent. Protect the apparatus from light. Evaporate the acetone extract to 5-10 ml. on a steam bath and cool to room temperature.

Prepare a chromatographic column as follows—Glass column; 10 mm. internal diameter 300 mm. long with a constriction of 5 mm. at the lower end. Insert a plug of glass wool in the lower end and tamp it down with a suitable rod to a thickness of 2 to 3 mm. Prepare a slurry of aluminium oxide with acetone, pour the slurry into the column and allow to settle. The prepared column should be about 200 mm. high. Allow the acetone layer to drain down to the top of the aluminium oxide column.

Chromatography of feed extract—Transfer the acetone extract of the feed to the prepared chromatographic column, and elute with acetone until the furazolidone band has passed through the column and collect the eluate. Evaporate the acetone eluate just to dryness on a steam bath. Dissolve the residue in 10 ml. amyl alcohol and transfer the solution to an amber-glass 100 ml. separating funnel Complete the transfer using 10 ml. amyl acetate as a rinse liquid. Extract the solution with five separate 10 ml. portions urea solution and transfer each separate aqueous extract to an amber-glass 100 ml. volumetric flask. Dilute to 100 ml. with urea solution and mix.

Measure the extinction of the solution at 375 nm. against urea solution as a blank and calculate the furazolidone content of the feed from the relationship:

$$E^{1\%}_{1\ cm.} \text{ of furazolidone in urea solution} = 643.$$

Note: Solutions of furazolidone should be protected from light at all times.

20· DETERMINATION OF DINITOLMIDE

(3, 5-dinitro-*o*-toluamide)

20·1 REAGENTS

Acetone, 95 per cent.—Add 5 ml. water to 95 ml. acetone.

Acetonitrile, 85 per cent.—Add 850 ml. acetonitrile to 150 ml. water.

Aluminium oxide—Dry a suitable grade of aluminium oxide prepared for chromatography at 105°C. for 30 minutes before use.

Diaminoethane—Fresh undiscoloured diaminoethane is imperative.

Dimethylformamide, 95 per cent.—Add 5 ml. water to 95 ml. dimethylformamide.

Dinitolmide standard solution—Weigh 40·0 mg. pure dinitolmide and transfer to a 100 ml. volumetric flask. Add acetonitrile, 85 per cent., and shake until all the dinitolmide has dissolved. Dilute to 100 ml. with acetonitrile, 85 per cent. Dilute 10 ml. of this solution to 100 ml. with acetonitrile, 85 per cent., to give a solution containing 0·04 mg. per ml.

20·2 PROCEDURE

Weigh 10·0 g. of the sample and transfer to a 250 ml. conical flask. Add 65 ml. 85 per cent. acetonitrile and heat to 50 ± 5°C. (Caution: all operations involving acetonitrile should be carried out in an efficient fume hood.) Maintain at this temperature for 30 minutes, swirling occasionally. Allow the flask to cool to room temperature and add 20 g. activated aluminium oxide, gently mixing for 3 minutes. (Note: the addition of aluminium oxide is unnecessary where the content of dinitolmide is in excess of 1 per cent.)

Filter the solution with suction through a 40 mm. sintered glass funnel (porosity 3), transferring as much of the solids as possible. Transfer the remaining solids to the sintered glass funnel with 85 per cent. acetonitrile, using as little as possible, and suck the residue dry. Suspend the filter cake in the sintered glass funnel by the addition of a little 85 per cent. acetonitrile with gentle stirring but without suction. Remove the liquid by applying suction, then repeat the suspension and filtration. Keep the volume of the filtrate below 100 ml. Transfer the filtrate to a 100 ml. volumetric flask, dilute to volume with 85 per cent. acetonitrile and mix.

Having regard to the expected concentration of dinitolmide in the sample, dilute the filtrate with 95 per cent. acetone by reference to the following table.

Dinitolmide %	Further dilution	Aliquot ml.	Factor M
0·004— 0·012	none	4	1
0·012— 0·025	none	2	2
0·025— 0·050	×10	10	4
0·050— 0·100	×10	5	8
0·100— 0·250	×10	2	20
0·25 — 0·50	×100	10	40
0·50 — 1·00	×100	5	80
1·00 — 2·50	×100	2	200
2·50 — 5·00	×1000	10	400
5·00 —10·00	×1000	5	800
10·00 —25·00	×1000	2	2000

Transfer the appropriate aliquot indicated in the table to each of three beakers, A, B and C. (Omit beaker A for samples containing dinitolmide in excess of 0·25 per cent.) Add 1 ml. standard dinitolmide solution to beaker C and evaporate each of the solutions in beakers A, B and C at a temperature not exceeding 60°C. in a current of air. Transfer 10 ml. 95 per cent. dimethylformamide to beaker A and 2 ml. 95 per cent. dimethlyformamide to each of beakers B and C. Swirl the beakers intermittently for five minutes to dissolve the dinitolmide. Add 8 ml. diaminoethane to each of beakers B and C and mix. Filter the solution through a suitable filter paper if a persistent turbidity is formed.

Measure the extinctions, E, at 560 nm. in a 10 mm. stoppered cell, of each solution 5 minutes after the addition of diaminoethane. Keep the temperature of the cell compartment below 30°C. to avoid rapid fading of colour. Calculate the dinitolmide content of the sample from the expression

$$\text{Dinitolmide per cent.} = \frac{(E_b - E_a) \times M}{100\,(E_c - E_b)}$$

21· DETERMINATION OF CALCIUM

Calcium may be determined by the oxalate method or, alternatively, by the atomic absorption spectrophotometric method.

21·1 OXALATE METHOD

21·11 REAGENTS

Ammonia solution, 2 per cent. v/v—Dilute 20 ml. concentrated ammonia solution (d=0·88) with water to 1 litre.

Ammonium acetate solution—Dissolve 500 g. ammonium acetate in 500 ml. water.

Ammonium oxalate solution—saturated aqueous solution.

Bromocresol green indicator solution—Dissolve 0·05 g. bromocresol green in 20 ml. ethanol and dilute with water to 100 ml.

Citric acid—monohydrate.

Hydrochloric acid, 50 per cent. v/v—Dilute 50 ml. concentrated hydrochloric acid (d=1·18) with water to 100 ml.

Potassium permanganate, 0·1 N.

Sulphuric acid, 20 per cent. v/v—Cautiously add 100 ml. concentrated sulphuric acid (d=1·84) to 400 ml. water, and, while hot, add 0·1 N potassium permanganate drop by drop until a faint pink colour persists.

21·12 DISSOLUTION OF THE SAMPLE

Weigh to the nearest mg. 5 g. of the sample into a platinum or silica basin and incinerate at a temperature not exceeding 500°C. until all the organic matter has been destroyed. Allow to cool, moisten the ash with water and cautiously add 10 ml. 50 per cent. v/v hydrochloric acid, avoiding loss by use of a cover glass. Wash the cover glass with water, adding the washings to the basin and evaporate to dryness. Continue heating for at least one hour to dehydrate any silica which may be present. Cool, add 20 ml. water and 10 ml. 50 per cent. v/v hydrochloric acid, bring to the boil and filter into a 250 ml. volumetric flask. Wash the basin and filter with hot water collecting the washings in the flask. Cool, make up to volume and mix.

21·13 PROCEDURE

Transfer an aliquot of the filtrate, containing about 40 mg. Ca, to a 400 ml. beaker and add water to make the volume approximately 150 ml. Add sufficient bromocresol green indicator, 1-2 g. citric acid, and ammonium acetate solution drop by drop until the colour changes to yellow-green (pH 4·0). Bring the solution to the boil and while boiling, slowly add with stirring 20 ml. boiling ammonium oxalate solution. Digest the mixture at boiling point for 15 minutes, allow to cool and stand for at least 4 hours. Decant the supernatant liquid through a sintered glass crucible (porosity 4). Wash down the sides of the beaker with hot water, stir up the calcium oxalate precipitate and allow to settle. Decant the supernatant liquid through the sintered glass crucible. Transfer the precipitate to the sintered glass crucible with 2 per cent. v/v ammonia solution and wash the beaker and crucible with 2 per cent v/v ammonia solution until the washings are free from chloride. Remove the crucible and carefully rinse the outside with water, discarding the rinsings. Transfer the bulk of the precipitate to the original beaker and wash the remainder through with hot 20 per cent. v/v sulphuric acid, adding the washings to the beaker. Add 70-80 ml. boiling water and mix to dissolve the precipitate. Heat the contents to 75-80°C. and titrate with 0·1 N potassium permanganate until a faint pink colour persists for 30 seconds, transferring the crucible to the beaker towards the end of the titration.

1 ml. 0·1 N $KMnO_4 \equiv 2{\cdot}0$ mg. calcium.

21·2 ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

21·21 APPARATUS

Atomic absorption spectrophotometer.
Calcium hollow-cathode lamp.

21·22 REAGENTS

Calcium stock solution—Dry calcium carbonate at 105°C. for 1 hour. Transfer 2·497 g. into a 1 litre volumetric flask using approximately 100 ml. water. Add slowly with swirling 60 ml. N hydrochloric acid. When all the calcium carbonate has dissolved, dilute to 1 litre with water.
1 ml. $\equiv$ 1 mg. calcium.

Calcium dilute solution—Dilute 20 ml. calcium stock solution to 200 ml.
1 ml. $\equiv$ 100 μg. calcium.

Calcium working standard solutions—Add 10 ml. releasing agent to each of six 100 ml. volumetric flasks. Measure 0, 3, 6, 9, 12, 15 ml. dilute calcium solution (1 ml. $\equiv$ 100 μg. calcium) into the flasks and dilute to 100 ml. with water. The flasks contain 0, 3, 6, 9, 12, 15 μg. Ca per ml. respectively.

Lanthanum oxide solution (releasing agent)—Wet 117·3 g. lanthanum oxide, La_2O_3, low in calcium with water. Add 350 ml. concentrated hydrochloric acid (d = 1·18) slowly, and shake until all the lanthanum oxide is dissolved. Allow to cool and dilute to 1 litre with water.

21·23 PROCEDURE

Set up the instrument using the line at 422·7 nm. Use a fuel rich flame. Add releasing agent and water to a suitable aliquot of the sample solution, prepared in accordance with para. 21·12 to produce a standard volume of solution to contain between 5 and 10 μg. of calcium per ml. and 10 per cent. v/v releasing agent. Prepare a blank solution from which only the sample has been omitted. Spray water into the flame and zero the instrument. Spray successively, in triplicate, the standard solutions, sample and blank, washing the instrument through with water between each spraying. Plot the mean reading obtained for each standard solution against its calcium content. Determine the calcium content of the sample and blank solutions from the graph and from the difference between them calculate the calcium content of the sample. If a number of samples is being examined, one or more standard solutions must be resprayed at intervals during the course of the analyses."

(*f*) In Schedule 9,

(i) in Part I, in the second column, in item 13, for the words—

"Provided that the variation from each amount stated shall not exceed 1·75%."

there shall be substituted the words—

"Provided that the variation from each amount stated shall not exceed 1·75% and, where the total of the amounts stated is 25% or over, the amount of all variations taken together, after setting off deficiencies against excesses, shall not exceed 1/20th of the aforesaid total."

(ii) in Part II, in the first column, for the words—

"Dried brewery and distillery grains."

there shall be substituted the words—

"Dried brewery grains."

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

"12a. Dried distillery by-products (other than malt culms and dried yeast)	Oil, 0·75% or one fifth of the amount stated, whichever is the greater; protein, one fifth of the amount stated; fibre, if present in excess of 2%, if the actual amount exceeds that stated, one eighth of the amount stated; if the actual amount is less than that stated, one half of the amount stated; lime (expressed as calcium (Ca)), if present in excess of 2%, one fifth of the amount stated."

Sealed with the Official Seal of the Ministry of Agriculture for Northern Ireland this 17th day of June 1970.

(L.S.)

A. E. W. Steen,
Assistant Secretary.

EXPLANATORY NOTE

(This Note is not part of the Regulations but is intended to indicate their general purport.)

These Regulations amend the Fertilisers and Feeding Stuffs Regulations (Northern Ireland) 1968.

They introduce an additional limit of variation for high concentration compound fertilisers; revise methods of sampling fertilisers and feeding stuffs in liquid form; prescribe methods of analysis for certain prophylactics added to feeding stuffs; add a new composite entry in Schedule 2 covering "dried distillery by-products" and lay down methods of analysis for calcium in these products; introduce implied definitions for "kainit" and "magnesium kainit"; and effect certain minor revisions to Schedules 4 and 8.