FERTILISERS AND FEEDING STUFFS.

Appointed Day.

THE FERTILISERS AND FEEDING STUFFS (NORTHERN IRELAND) ORDER, 1928, DATED 5TH DAY OF JUNE, 1928.

1928. No. 59.

The Ministry of Agriculture by virtue and in exercise of the power for this purpose vested in it by the Fertilisers and Feeding Stuffs Act, 1926, and of every other power in that behalf enabling it hereby appoints the first day of July, one thousand nine hundred and twenty-eight as the day on which the Fertilisers and Feeding Stuffs Act, 1926, shall come into operation in Northern Ireland.

In Witness whereof the Official Seal of the Ministry of Agriculture, Northern Ireland, is hereunto affixed this fifth day of June, one thousand nine hundred and twenty-eight.

James S. Gordon,

Sécretary.

(L.S.)

General Regulations.

THE FERTILISERS AND FEEDING STUFFS (NORTHERN IRELAND) REGULATIONS, 1928. DATED 5TH DAY OF JUNE, 1928.

1928. No. 43.

The Ministry of Agriculture by virtue and in exercise of the power for this purpose vested in it by the Fertilisers and Feeding Stuffs Act, 1926, and of every other power in that behalf enabling it, hereby makes the Regulations following, that is to say :—

Manner of marking particulars on label in the case of sales of small quantities. (Section 1 (1) (ii).)

1. In the case of parcels to which Section 1 (1) (ii) of the Act relates, the label shall bear the particulars prescribed in Section 1 (1) of the Act in block capital letters and figures not less than half-an-inch long.

Limits of Variation.

2. The limits of variation for the purposes of Sections 2 (5) and 26 (5) of the Act shall be as follows:—

	Limits of Variation. (expressed as percentages of the <i>whole bulk</i> .)								
Article.	Nitro- gen.	Soluble phos- phoric acid.	Insolu- ble phos- phoric acid.	Phos- phoric acid.	Potash.				
1. Calcium cyanamide	0.5		· ·	— —					
2. Dried blood for fertilising	0.5			<u> </u>					
3. Nitrate of lime	0.5	l —							

Limits of Variation for Fertilisers.

		Limits of Variation. (expressed as percentages of the <i>whole bulk</i>										
-	Article.	Nitro gen.	Soluble phos- phoric acid.	Insolu- ble phos- phoric acid.	Phos- phoric acid.	Potash.						
4. 5.	Nitrate of soda Oil seed fertilisers, as des- cribed in the First Schedule to the Act	0.3 0.5			_	—— ——						
6. 7. 9. 10. 11. 12.	Hoofs Hoofs and Horns Horns Superphosphate Basic superphosphate Precipitated bone Potassium salts used as fertilisers, as described in the First Schedule to the Act	0.5 0.5 0.5 	0.5		 1 1							
	(a) If the percentage of potash stated does not exceed 15.		—			1						
13.	 (b) If such percentage exceeds 15. Compound fertilisers, as described in the First Schedule to the Act:— (a) If the respective per- 	• 0.3	0.5	0.5		0.3						
	centages of nitrogen and potash stated do not exceed 4. (b) If such respective per-	0.5	0.5	0.5		0.5						
	centages exceed 4 but do not exceed 5. (c) If such respective per-	0.75	0.5	0.5		0.75						
14.	 c) the second of the percentages of the percentages of phose phoric acid (soluble and insoluble) stated amounts to 14 or more, 											
	then (a) If the excess of the actual percentage of insoluble phosphoric acid over that stated is 1.5 or more.	0.3	2	<u> </u>	-							
	(b) If such excess is not less than 1, but is less than 1.5.	0.3	1.5	: <u> </u>		_						
	(c) If such excess is not less than 0.5, but is	0.3	1	-	·							
	(ii) In all other cases	0.3	0.5	0.5								

Article.	Limits of Variation. (percentages are percentages of the) whole bulk.)
15. Sulphate of ammonia	Nitrogen, 0.3 per cent. ; free acid, one-
16. Basic slag	fifth of the quantity stated.
17. Raw phosphate or phosphate rock	Phosphoric acid, one-twentieth of the amount stated; amount that will pass through a prescribed sieve, one- twentieth of the amount stated.
18. Guano, including Peruvian and other raw guanos	Nitrogen, one-fifth of the percentage stated, with a minimum of 0.25 per cent, and a maximum of 1.5 per cent.; phosphoric acid, one-tenth of the percentage stated, with a maxi- mum of 2 per cent.; and potash, one-fifth of the percentage stated.
19. Bone meal or other bone product (excluding dissolved or vitriolised bone), as described in the First Schedule to the Act.	Nitrogen, 0.5 per cent., increasing, if the actual percentage of phosphoric acid exceeds that stated, to not more than 1 per cent. at the rate of 0.25 per cent. of nitrogen for each 1 per cent. of such excess of phosphoric acid; and phosphoric acid, 1 per cent., increasing, if the actual per- centage of nitrogen exceeds that stated, to not more than 2 per cent. at the rate of 1 per cent. of phos- phoric acid for each 0.25 per cent. of such excess of nitrogen.
20. Fish residues or other fish pro-	Nitrogen, 0.5 per cent. increasing, if the actual percentage of phosphoric acid exceeds that stated, to not more than 2 per cent. at the rate of 0.25 per cent. of nitrogen for each 1 per
 duct, as described in the First Schedule to the Act. 21. Meat and bone residues as des- cribed in the First Schedule to the Act. 	cent. of such excess of phosphoric acid; and phosphoric acid, 1 per cent. increasing, if the actual per- centage of nitrogen exceeds that stated, to not more than 3 per cent. at the rate of 1 per cent. of phos- phoric acid for each 0.25 per cent.
22. Calcium hydrate; calcium hy- droxide; slaked lime.	Calcium hydroxide and equivalent of calcium oxide, one-tenth of the
23. Chalk, ground	Calcium carbonate and equivalent of calcium oxide, one-twentieth of the amounts stated.
25. Limestone, ground	Calcium carbonate and equivalent of calcium oxide, one-twentieth of the amounts stated; amount that will pass through a prescribed sieve, one- twentieth of the amount stated. Calcium oxide, one-tenth of the amount
	stated.
Limits of Variation	for <i>E</i> 'eeding Stuffs.
Article.	Limits of Variation.
1. Linseed meal </th <th>One-tenth of the amount of oil stated. One-twentieth of the amount of albu- minoids (protein) stated.</th>	One-tenth of the amount of oil stated. One-twentieth of the amount of albu- minoids (protein) stated.

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Article.		Limits of Variation.
4. Dried brewery and di	stillery grains	One-fifth of the respective amounts of
5. Linseed cakes and t	he meals of	
6. Maize, flaked		One-eighth of the respective amounts
7. Maize germ cake or m	eal }	of oil and albuminoids (protein)
8. Maize gluten feed	••	stated.
10. Sova cake or meal		
11. Coconut or copra cak	e or meal)	
12. Cotton cakes or meals cated	, not decorti-	
13. Feeding meat meal, in the First Schedule	as described to the Act	One-tenth of the respective amounts
14. Oil cakes or meals n	ot otherwise >	of oil and albuminoids (protein)
the product of spy	a which are	stated.
ticated substance o	r seed from	
which oil has been ren	noved	
15. Palm kernel cake or r	neal J	(· · · · · · · · · · · · · · · · · · ·
16. Compound cakes of	meals, as	
to the Act	ist beneutie	· ·
17. Cotton cakes or mea	ls from de-	· ·
corticated or partly	decorticated	
cotton seed	ot othorizad	
specifically mentioned	I OUTOFWING	One-tenth of the respective amounts
19. Oil cakes or meals n	ot otherwise }	of oil and albuminoids (protein) and
specifically mentione	d which are	one-eighth of the amount of fibre
the product of any	one decorti-	stated.
stance or seed from	n which oil	
has been removed		
20. Rice bran or rice r	neal, or the	
by-produced produced	i in milling	· ·
-21. Feeding bone flour.	as described	One-twentieth of the amount of phos-
in the First Schedule	to the Act	phoric acid and one-tenth of the
		amount of albuminoids (protein) stated.
2. Feeding meat and b	one meal, as	One-tenth of the respective amounts
described in the Firs	t senedule to	or oil and albuminoids (protein)
	•• ••	of phosphoric acid stated.
23. Fish meal, as describe	d in the First	One-tenth of the respective amounts
Schedule to the Act		of albuminoids (protein), oil and
		sait and one-sixth of the amount
24. Malt culms		One-fifth of the amount of albuminoida
		(protein) and one-eighth of the
		amount of fibre stated.
25. Treacle or molasses	•• ••	Une-twentieth of the amount of sugar
26. Molasses feeds, as de	scribed in the	One-tenth of the amount of sugar and
First Schedule to the	Act	one-eighth of the amount of fibre
. 07 Clorron m 1	ب	stated.
227. Ulover meal 28 Dried suger heat resid		stated
29. Wheat offals. or mille	rs' offals	Bouddett.
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· · · · · · · · · · · · · · · · · · ·		

*The Limits of Variation in respect of oil and salt in fish meal shall apply in the case of statements of percentages made in compliance with the Act in the case of fish meal, including white fish meal, but shall not operate so as to permit of the application of the name " white fish meal " to an article containing more than 6 per cent. of oil or 4 per cent. of salt.

Manner of taking samples.

3. The manner in which samples shall be taken and dealt with in cases where under the Act they are required to be taken in the prescribed manner shall be as follows:—

A. In the case of a Fertiliser.

(i) When the fertiliser is delivered or prepared for delivery in bags or other packages, a number of bags or packages shall be taken as follows, viz. :---

· .	Number taken	But not fewe
•	for sampling.	packages
	Per cent.	than
Where the quantity is from		
2 to 20 packages	20	2
Where the quantity is from		
21 to 60 packages	10	4
Where the quantity is from		
61 to 200 packages	71	6
Where the quantity is from	. –	··
201 to 500 packages	5	15
Where the quantity is from	•	
501 to 1,000 packages	4	25
Where the quantity exceeds		
1.000 packages	3	40

When the number of packages to be drawn according to the above percentage scale contains a fraction, this fraction shall be counted as a whole number.

In each case the quantity shall be treated as composed of separate approximately equal portions, the number of portions being reckoned as the same as the number of packages to be selected for the purpose of sampling, and one package shall be drawn by the inspector or official sampler at random from each, of these portions.

Where the quantity exceeds 500 packages it may, in the discretion of the inspector or official sampler, be treated as composed of separate parcels of 500 packages each, any one of which may be sampled separately; but where two or more samples are taken under this provision from one quantity, they shall be mixed together to form one sample for the purposes of the Act.

Only unopened packages shall be selected for the purpose of the sample.

(ii) The sample shall be drawn by one of the following methods :---

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(a) The selected bags or packages shall be emptied separately on a clean and dry floor and worked up with a spade and one spadeful from each set aside. The spadefuls so set aside shall then be thoroughly mixed together and any lumps broken up by the hand or spade. From this mixture a sample from about 2 lbs. to 4 lbs. in weight shall be taken. (b) As an alternative method, when the material is of a suitable nature, a portion shall be drawn from each selected package by means of a suitable sampling spear; provided that, in the case of a sample taken by an inspector, a sampling spear shall not be used if objection is raised thereto, prior to the taking of the sample, on the grounds that the material is unsuitable. The separate quantities thus taken from the selected bags or packages shall be thoroughly mixed together and a sample from 2 lbs. to 4 lbs. in weight shall be taken from the mixture.

(iii) Where the fertiliser is in bulk and is in fine condition so that it will pass into a sampling spear pressed into it, it shall be sampled with the sampling spear, which shall be pressed into the bulk in different parts in such a manner that portions are withdrawn at random from different parts of the bulk.

The number of portions taken by the spear shall be as follows :---

		TOTOTOTO.
Where the quantity does not exceed 1 ton		· 4
Where the quantity is from 1 ton to 2 tons	·	6
Where the quantity is from 2 tons to 5 tons	••	10
Where the quantity is from 5 tons to 10 tons	•••	15
Where the quantity is from 10 tons to 25 tons	••	25
Where the quantity is from 25 tons to 50 tons	• •	40
Where the quantity is from 50 tons to 100 tons	••	60

Portions

Where the quantity exceeds 50 tons, it may, in the discretion of the inspector or official sampler, be treated as composed of separate portions of 50 tons each or part thereof, and each of these portions shall be treated as separate for sampling purposes, as many portions being drawn, according to the scale above, as there are portions of 50 tons or part thereof. The portions withdrawn shall be thoroughly mixed together and a sample from 2 lbs. to 4 lbs. in weight shall be taken from the mixture.

(iv) Where the fertiliser is in a coarse or lump condition, as in the case of caustic or burnt lime, not ground, the sample shall be drawn as follows:—

(a) In Bulk.—Four spadefuls shall be taken for each ton, each such spadeful to be taken at random from a different part of the lot. The quantity thus removed shall be crushed immediately and passed through a sieve with meshes one and a quarter inch square. Any lumps and stones in the portion taken are not to be disregarded, but shall be broken up and mixed with the remainder of the sample. It shall be mixed thoroughly and rapidly and a sample of 4 lbs. to 6 lbs. taken.

(b) In Packages.—The packages shall be selected according to the scale given in (i) and treated as described in (ii) (α). The spadefuls removed shall be crushed immediately and treated as in (iv) (α).

(v) Where the fertiliser consists of materials such as caustic lime or slaked lime (calcium hydroxide) which are liable to undergo change on exposure to air and moisture, or where the fertiliser consists of materials such as calcium nitrate or ammonium nitrate, which are liable to absorb moisture, or where the material is sulphate of ammonia, the sampling shall be carried out in a dry place and as expeditiously as possible, the division of the sample into parts (see paragraph D) shall be carried out at once, and each part shall be placed in a clean, dry, stoppered bottle or airtight container. Care must be taken to see that the sample bottles or containers are completely dry before use.

(vi) When the fertiliser consists of bulky material, uneven in character and likely to get matted together, such as shoddy, wool refuse, hair, &c., the selected packages shall be emptied on a clean dry floor, or if in bulk, the portions selected at random from different parts of the bulk, shall be placed on a clean dry floor, the matted portions torn up, and all the portions thoroughly mixed together. The sample shall be taken from the mixture and shall be from 2 lbs. to 4 lbs. in weight. If the material separates into a fibrous part and a powdery part, the sample drawn shall consist of these two kinds in approximately their relative proportions by weight, as they exist in the fertiliser.

B. In the case of a Feeding Stuff.

(vii) When the feeding stuff is in the state of small lumps or meal, it shall be sampled in the same manner as prescribed for fertilisers under paragraphs (i), (ii) and (iii).

(viii) When the feeding stuff is in the state of cake, a number of cakes shall be selected from different parts of the whole quantity as follows :---

•	Cakes.
Where the quantity does not exceed 2 tons	5
Where the quantity exceeds 2 tons and does not	•
exceed 5 tons	10
Where the quantity exceeds 5 tons and does not	;
exceed 50 tons	15
Where the quantity exceeds 50 tons and does not	
exceed 100 tons	25

In each case the quantity shall be treated as composed of separate approximately equal portions, the number of portions being reckoned as the same as the number of cakes to be selected for the purpose of sampling, and one cake shall be drawn by the inspector or official sampler at random from each of these portions.

Where the quantity exceeds 50 tons it may, in the discretion of the inspector or official sampler, be treated as composed of separate parcels of 50 tons each, any one of which may be sampled separately; but where two or more samples are taken under this provision from one quantity, they shall be mixed together to form one sample for the purposes of the Act.

(ix) The selected cakes shall be broken by a cakebreaker or in some other manner so that the whole shall pass through a sieve with meshes one and a quarter inch square. The broken pieces shall then be thoroughly mixed, and from the mixture a sample of not less than 6 lbs. in weight shall be taken.

(x) Where any appreciable portion of the feeding stuff appears to be mouldy or is otherwise apparently unsuitable for feeding purposes, separate samples shall be taken of the unsuitable portion and of the residue of the feeding stuff respectively, and in the case of unsuitable cakes, the sample may consist of several large pieces representative thereof.

(xi) When the feeding stuff is in a fluid, or semi-fluid condition, packages shall be taken at random in accordance with the scale shown in paragraph (i), the contents well mixed by stirring or shaking, and a similar quantity taken from each. These portions shall then be mixed together, in a clean dry vessel, and from the mixture a sample of from 2 lbs. to 4 lbs. in weight shall be taken.

C. In the Case of both Fertilisers and Feeding Stuffs.

(xii) Where the quantity of the whole consignment does not exceed 2 cwts., the sample may consist of such a portion of the consignment or bulk as is fairly representative of the whole, and the sample shall be of such a quantity that each of the parts into which (under paragraph D) it is to be divided will be not less than half a pound in weight.

(xiii) In every case the sampling shall be done as quickly as is possible consistently with due care and the material shall not be exposed any longer than is absolutely necessary.

D. Divison of Sample.

Where a sample has been taken with a view to the institution of any civil or criminal proceedings, the person taking the sample shall divide it into three parts, as nearly as possible equal, in the following manner :—

(xiv) (a) In the case of dry or powdered substances.—The sample, drawn as prescribed in the foregoing section, shall be thoroughly mixed on heavy glazed paper or oilcloth and divided into three similar and approximately equal portions. Each such portion shall be placed in a separate tin or bottle of a kind described in paragraph (xv) and shall constitute a part of the sample.

(b) In the cases of substances in a fluid or semi-fluid condition. The sample drawn as prescribed shall be thoroughly mixed and at once divided into similar and approximately equal parts by pouring successive portions into each of three bottles.

(xv) Each of the parts into which the sample is divided shall be packed in a dry clean bottle or (except in the case of a fertiliser) a dry clean tin with close-fitting lid (such as a lever lid), so that the original composition of the fertiliser or feeding stuff may be preserved. In the case of caustic lime, slaked lime (calcium hydroxide), calcium nitrate, ammonium nitrate, ammonium sulphate and other substances likely to undergo change if not kept in an air-tight receptacle the bottle used shall have a ground-in stopper, or be provided with a metal cap with inner pad. Each of the said parts shall be so secured and sealed that the bottle or tin containing the sample cannot be opened without breaking the seal.

(xvi) Each of the said parts, shall be sealed and initialled by. the person taking the sample. It may also be sealed or initialled by the person on whose premises the sample is taken, or his representative. Each part shall be marked with the name of the article, any mark applied to the article in compliance with the Act, the date and place of the sampling, and with some distinguishing number, in such a manner that the particulars so marked can be seen without breaking the seal or seals.

(xvii) Samples shall not be drawn from parcels bearing the appearance of having received damage in transit or after delivery.

E. Analyst to whom Samples are to be sent.

Where under the Act any person desires that a sample shall be analysed by the Agricultural Analyst, the sample or parts of the sample, as the case may be, shall be sent to such Agricultural Analyst as is hereby prescribed, that is to say :—

- (1) If the sample is taken in a county, or in a county borough the Council of which have appointed or concurred in appointing an Agricultural Analyst, then to the Analyst appointed for such county or borough; respectively.
- (2) If the sample is taken in a county borough the Council of which have not appointed or concurred in appointing an Agricultural Analyst, then to the Analyst appointed for the county in which for the purposes of the Local Government Act, 1898, such borough is deemed to be situate.

Manner of marking articles and nature of marks. (Section 4 (1).)

4. A parcel required by Section 4 (1) of the Act to be marked shall be marked in writing (including printing and stencilling) on the article itself or on the wrapper or container of the package or packages composing the parcel, or by means of a label securely attached to the package or placed inside the package, or in such manner as clearly to identify the label with the parcel in question; provided that:—

- (a) the marking shall be legible;
- (b) every parcel shall be marked in such manner that the parcel shall remain marked so long as it is on the premises where it has been marked; and
- (c) where a parcel consists of a number of packages and the packages or any of them are not marked, the parcel shall be marked by a mark entered in a register in accordance with Section 4 (2) of the Act.

Form of Register of Marks specifying the particulars which the several marks are used as indicating. (Section 4 (2).)

5. Any register of marks kept in accordance with Section 4 (2) of the Act, specifying the particulars which the several marks entered in the register are used as indicating, shall be kept in such form that the particulars required by the said Section of the Act, relating to each separate parcel or consignment, shall be readily ascertainable by an inspector.

Form of Register of Parcels delivered or consigned ex ship. (Section 5 (2).)

6. The register of articles delivered or consigned direct from ships or quays to purchasers, required to be kept in accordance with Section 5 (2) of the Act, shall be kept in such form that the particulars required, relating to each separate parcel or consignment, shall be readily ascertainable by an inspector.

Method of dealing with third part of sample. (Section 13 (1).)

7. In the case of a sample taken by an official sampler, the third part shall be delivered or sent by registered post to the last seller or his agent. In the case of a sample taken by an inspector, the third part shall be delivered or sent by registered post to the person who would be liable to prosecution in the event of an offence being disclosed by the results of analysis of the sample, or to the representative of such person.

Period for which Registers and Statutory Statements shall be preserved. (Section 9 (1).)

8. The period for which a register and a statutory statement shall be preserved in accordance with Section 9 (1) of the Act shall be four months.

Period for which duplicate part of sample shall be retained by agricultural analyst. (Section 13 (2).)

9. The period for which an agricultural analyst shall retain one of the parts of each sample sent to him for analysis shall be six months from the date of the certificate relating to the sample.

Form of Quarterly Return of Results of Analyses. (Section 18.)

10. The return of results of the analysis of the samples submitted to the agricultural analyst for each county, county borough or other district, required to be made in accordance with Section 18 of the Act, shall be in the forms C and D set forth in the Schedule hereto or to the like effect.

Methods of Analysis. (Section 23 (1) (e).) Methods of Analysis of Fertilisers.

11. The methods of analysis of a fertiliser for the purposes of the Act shall be as follows :--

- (i) Preparation of the sample for analysis :---
 - (a) In the case of powdered fertilisers in a dry, or moderately dry, condition, the sample shall be passed through a sieve having apertures about one millimetre square.

Adventitious materials which cannot be conveniently crushed, e.g., fragments of metal in basic slag, shall be removed and allowed for.

- (b) Other substances which are dry enough to powder, but which are not in a fine condition, shall be pulverised until the sample passes through a sieve having apertures about one millimetre square.
- (c) Wool, hair, hoof, shoddy and similar substances shall be pulled apart and cut until in a fine condition; or, if dry, they may be passed through a shredding machine.
- (d) Moist fertilisers which do not admit of being passed through a sieve shall be thoroughly mixed by the most suitable means.
- (e) In the case of substances which gain or lose water during the process of pulverising or mixing, the proportion of water shall be determined in the coarse and in the powdered condition respectively, and the results of the analysis of the powdered sample shall be calculated to the water content of the original coarse substance.
- (f) Crystalline or saline materials, such as sulphate of ammonia, nitrate of soda or potash salts, may be prepared by being well mixed and rapidly ground in a stoneware mortar, the portion finally reserved for analysis being specially finely ground.
- (g) When the sample has been passed through the sieve and thoroughly mixed, or, if not passed through the sieve, has been thoroughly mixed, a part of it not being less than 100 grams shall be placed in a stoppered bottle and from this the portions for analysis shall be weighed.

(ii) Determination of moisture (loss on drying).—A weighed quantity of the sample shall be dried at 100°C.

(iii) Determination of nitrogen.—The presence or absence of nitrates shall first be ascertained :—

(a) Nitrogen (organic and ammoniacal) in absence of nitrates.

(A) A weighed portion of the sample shall be transferred to a Kjeldahl digestion flask, 25 millilitres of concentrated sulphuric acid (or more if necessary) shall be added and the flask gently heated until frothing ceases. 10 grams of potassium or sodium sulphate (anhydrous) shall then be added and the flask further heated until the colour of the The digestion clear liquid ceases to diminish. continued for an hour thereafter to shall be ensure complete oxidation of the organic matter. The operation may be accelerated by the addition of a small crystal of copper sulphate or a globule of mercury to the liquid in the digestion flask.

(B) The quantity of ammonia present in the liquid shall be determined by distillation into standard acid after liberation with alkali and, where mercury has been used, with the addition also of sodium or potassium sulphide solution.

(b) Nitrogen (total, i.e., organic, ammoniacal and nitric) when nitrates are present.

> (A) A weighed portion of the sample shall be transferred to a Kjeldahl digestion flask; 30 millilitres of concentrated sulphuric acid, containing one gram of salicylic acid or one gram of phenol, shall be added and the flask shall be shaken so as to mix its contents without delay. The shaking shall be continued at intervals during ten minutes, the flask being kept cool, and then 10 grams of potassium or sodium sulphate (anyhdrous) shall be added, together with either 5 grams of crystalline sodium thiosulphate or 2 grams of zinc dust. The flask shall be heated until the colour of the clear liquid ceases to diminish. and for an hour thereafter. A further quantity of concentrated sulphuric acid may be added if necessary. Copper sulphate or mercury may be used as described. in paragraph (iii) (a) (A).

> (B) The quantity of ammonia shall be determined as prescribed in paragraph (iii) (a) (B).

(c) Nitrogen in form of ammonium salts.

(Note:-In the case of compound fertilisers containing calcium carbonate with small quantities of ammonium

salts, the portion taken for analysis must be dissolved in hydrochloric acid and the solution used for distillation with alkali.)

(1) In absence of organic matter.—A weighed portion of the sample shall be dissolved in water and made up to a definite bulk. An aliquot part of the solution shall be transferred to a distillation flask and the quantity of ammonia shall be determined as above prescribed in paragraph (iii) (a) (B).

(2) In presence of organic matter.—A weighed portion of the sample shall be transferred to a distillation flask with about 200 millilitres of water and 5 grams of magnesium oxide, free from carbonates, and the quantity of ammonia determined by distillation into standard acid.

(d) Nitrogen in nitrates.

(1) In absence of organic matter.

(A) A weighed portion of the sample shall be dissolved in water and made up to a definite bulk. An aliquot part of the solution shall be transferred to a flask and a quantity of finely powdered Devarda metal added. The quantity of Devarda metal shall be not less than six times the weight of the sample present in the aliquot part taken. An excess of concentrated alkali shall then be added and the flask at once connected with a distillation apparatus. After standing for 30 minutes to allow the reaction to proceed, heating gently if necessary, the ammonia shall be distilled over into standard acid.

(B) Alternative method.—10 grams of the sample shall be dissolved in water and the solution made up to 500 millilitres. 50 millilitres of the solution shall be placed in a half-litre Erlenmeyer flask and 10 grams of reduced iron and 20 millilitres of sulphuric acid of 1.35 specific gravity shall be added. The flask shall be closed with a rubber stopper provided with a thistle tube, the head of which shall be half filled with glass beads, and allowed to stand until effervescence ceases. The liquid shall then be boiled for five minutes, the flask removed from the flame and any liquid that may have accumulated among the beads rinsed back with water into the flask. The solution shall be boiled for three minutes more and the beads again washed with a little water. The quantity of ammonia shall then be determined as described in paragraph (iii) (a) (B).

(2) In presence of organic matter.

One gram of the sample shall be placed in a halflitre Erlenmeyer flask with 50 millilitres of water. 10 grams of reduced iron and 20 millilitres of sulphuric acid of 1.35 specific gravity shall be added and the procedure prescribed in paragraph (iii) (d) (1) (B) above shall be followed, except that the quantity of ammonia contained in the liquid, after the treatment with reduced iron, shall be determined by distillation after addition of magnesium oxide as in paragraph (iii) (c) (2). In cases in which the proportion of nitrates is small, a larger quantity of the sample shall be taken.

(e) Control experiment in determination of nitrogen.—The materials used in any of the methods described in paragraph (iii) shall be examined as to their freedom from nitrogen by means of a control experiment carried out under similar conditions with the same quantities of the re-agents which have been employed in the actual analysis in the case of (a) one gram of pure sugar being used in place of the weighed portion of the sample. The quantity of standard acid found to have been neutralised in the control experiments shall be deducted from the total quantity of acid neutralised in the distillation of the sample.

(iv) Determination of phosphoric acid.

(a) Soluble phosphoric acid.—20 grams of the sample shall be continuously agitated for 30 minutes in a litre flask with 800 millilitres of water at room temperature. The flask shall then be filled to the mark and shaken and the contents shall be filtered.

> (A) 50 millilitres of the filtrate shall be boiled with 20 millilitres of concentrated nitric acid and the phosphoric acid shall be determined by the molybdate method prescribed in paragraph (iv) (d). In the case of fertilisers in which the proportion of phosphoric acid soluble in water is small, a larger quantity of the filtrate prepared as above shall be taken.

> method.—50 millilitres (B) Alternative of thefiltrate shall be boiled with 20 millilitres of concentrated nitric acid, cooled and the excess of acid \mathbf{with} neutralised ammonia, 50millilitres of ammonium citrate solution, prepared as described below, shall be added and the mixture raised to boiling point. Magnesia mixture shall then be added in the manner prescribed in paragraph (iv) (d).

(b) Insoluble phosphoric acid.—The quantity of soluble phosphoric acid as determined in paragraph (iv) (a) shall be deducted from the quantity of phosphoric acid as determined in paragraph (iv) (c) and the difference, if any, shall be taken as the quantity of insoluble phosphoric acid.

(c) Total phosphoric acid.

(A) A weighed portion of the sample shall be heated with concentrated sulphuric acid until all organic matter is destroyed and the phosphoric acid is completely in solution. After dilution the solution shall be filtered, the insoluble matter thoroughly washed and the filtrate made up to a definite bulk. The phosphoric acid shall be determined, by the method prescribed in paragraph (iv) (d), in an aliquot part of the solution, which shall first be nearly neutralised and then acidified with nitric acid. The insoluble matter is to be washed from the filter, re-extracted with acid and any phosphoric acid present in the solution added to the main quantity.

(B) Alternative method.—A weighed portion of the sample shall be incinerated or otherwise treated to destroy organic matter, if present. When direct incineration is employed, the weighed portion of the sample shall be treated, before being heated, with a nitrate or other oxidising material to prevent loss of phosphoric acid during heating or subsequent treatment. The residue (or the weighed portion taken, if no organic matter is present) shall be dissolved in hydrochloric acid, with the addition, if necessary, of nitric acid, and the solution shall be evaporated to dryness or, if much calcium is present, to a syrupy consistency to fix silica. The residue shall be boiled with nitric acid and, when much iron is present, with hydrochloric acid also. After dilution the solution shall be filtered, the insoluble matter thoroughly washed and the filtrate made up to a definite bulk. The phosphoric acid shall be determined in an aliquot part of the solution by the method prescribed in paragraph (iv) (d). The insoluble matter is to be washed from the filter, reextracted with acid and any phosphoric acid present in the solution added to the main quantity.

(d) Molybdate method.—To the solution, which should contain not more than 0.4 gram of phosphoric acid (P_2O_5) , and preferably from 0.1 to 0.3 gram, obtained as above described in paragraphs (iv) (a) (A) or (c), 100 to 150 millilitres of molybdic acid solution pre-

pared as described below, or an excess of such solution, i.e., more than is sufficient to precipitate all the phosphoric acid present in the solution, shall be added and the vessel containing the solution shall be placed in a water bath maintained at 70°C, for 15 minutes or until the solution has reached 70°C. It shall then be taken out of the bath and allowed to cool and the solution shall be filtered, the phosphomolybdate precipitate being washed several times by decantation and finally on the paper with one per cent. nitric acid solution. The filtrate and washings shall be mixed with more molybdic acid solution and allowed to stand for some hours in a warm place in order to ascertain that the whole of the phosphoric acid has been precipitated. The phospho-molbydate precipitate shall be dissolved in cold 2 per cent. ammonia solution, prepared as described below, and about 100 millilitres of the ammonia solution shall be used for the solution and washings. The solution shall be raised to the boiling point, the beaker removed from the burner and 15 to 20 millilitres of magnesia mixture, prepared as described below, or an excess of such mixture, i.e., more than sufficient to precipitate all the phosphoric acid present, shall then be added drop by drop, with constant stirring. The stirring shall be continued at intervals so long as the liquid remains very warm. After standing at least 4 hours with occasional stirring, the precipitate shall be filtered off, washed with 2 per cent. ammonia solution until free from chloride, dried and finally weighed as magnesium pyrophosphate. The filtrate and washings should not exceed 200 millilitres, and are to be tested by the addition of more magnesia mixture.

(e) Preparation of molybdic acid solution.-The molybdic

> 125 grams of molybdic acid and 100 millilitres of water shall be placed in a litre flask and the molybdic acid shall be dissolved by the addition, while the flask is shaken, of 300 millilitres of 8 per cent. ammonia solution, prepared as described below. 400 grams of ammonium nitrate shall be added, the solution shall be made up to the mark with water and the whole added to 1 litre of nitric acid (specific gravity 1.19). The solution shall be maintained at about 35°C. for 24 hours and then filtered.

Preparation of magnesia mixture.—The magnesia mixture shall be prepared as follows :-

110 grams of crystallised magnesium chloride and 140 grams of ammonium chloride shall be dissolved.

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(f)

in 1,300 millilitres of water. This solution shall be mixed with 700 millilitres of 8 per cent. ammonia solution and the whole shall be allowed to stand for not less than three days and shall then be filtered.

(g) Preparation of the ammonia solutions.—The 8 per cent. ammonia solution shall be prepared as follows :—

> One volume of ammonia solution of specific gravity 0.880 shall be mixed with three volumes of water. This solution shall then be adjusted by the addition thereto of more strong ammonia solution or water as required until the specific gravity of the solution is 0.967.

> The 2 per cent. ammonia solution shall be prepared as follows :---

One volume of 8 per cent. ammonia solution shall be mixed with three volumes of water.

(h) Preparation of ammonium citrate solution.—110 grams of pure citric acid shall be dissolved in water, the solution treated with 400 millilitres of 24 per cent. ammonia of specific gravity 0.9135 and then diluted to one litre.

(v) Determination of potash.—Potash shall be determined by the perchloric acid method or, alternatively, by the platinum chloride method.

(1) Perchloric acid method.

Salts of potash free from sulphates.—A weighed portion of the sample equivalent in potash content to 1.5 to 2.0 grams of potash (K_2O) shall be dissolved in water. The solution shall be filtered if necessary and made up to 500 millilitres. The potash shall be determined in 50 millilitres of the solution by precipitation with perchloric acid as prescribed in paragraph (v) (1) (d).

(b) Salts of potash containing sulphates.

(A) A weighed portion of the sample equivalent in potash content to 1.5 to 2.0 grams of potash (K_2O) shall be boiled with 300 millilitres of water to which 20 millilitres of hydrochloric acid have been Barium chloride solution shall be cautiously added. added, drop by drop, to the boiling solution until the sulphuric acid is completely precipitated. The liquid shall be cooled, made up to 500 millilitres and filtered. 50 millilitres of the filtrate shall be taken and evaporated to dryness and shall then be moistened with concentrated hydrochloric acid, again evaporated to dryness, treated with a little dilute hydrochloric acid and filtered if necessary. The potash shall be determined by precipitation with perchloric acid as prescribed in paragraph (v) (1) (d).

If the solution contains phosphates, iron, manganese or other substances that would interfere with the determination of potash, the method prescribed in paragraph (v) (1) (c) is to be used instead of the method prescribed in paragraph (v) (1) (b).

(B) Alternative method.—A weighed portion of the sample equivalent in potash content to 1.5 to 2.0 grams of potash $(K_2^{\prime}O)$ shall be boiled with 300 millilitres of water, cooled, made up to 500 millilitres and filtered. To 50 millilitres of the filtrate, 30 millilitres of a solution of sodium cobaltinitrite shall be added, the mixture stirred and allowed to stand for not less than two hours. It shall then be filtered and washed with water containing a small amount of the cobaltinitrite solution. The precipitate shall be dissolved in hot dilute hydrochloric acid and the solution filtered into a small porcelain dish and evaporated to dryness. The residue shall be dissolved in water and the potash determined by precipitation with perchloric acid as prescribed in paragraph (v) (1) (d).

(c) Potash in guanos and mixed fertilisers.

(A) 10 grams of the sample shall be gently incinerated in order to char organic matter, if present, and shall then be heated for 10 minutes with 10 millilitres of concentrated hydrochloric acid and finally boiled with 300 millilitres of water. The liquid shall be filtered, raised to the boiling point and powdered barium hydroxide shall be added until slightly alkaline. It shall then be cooled, made up to 500 millilitres and filtered. Of the filtrate, 250 millilitres shall be treated with ammonia solution and excess of ammonium carbonate and then while boiling, with a little powdered ammonium oxalate, cooled, made up to 500 millilitres and filtered. Of the filtrate, 100 millilitres are to be evaporated in a porcelain dish to dryness. If desired, nitric acid may be added during the evaporation after free ammonia has been driven off. The residue is to be heated gently over a low flame till all ammonium salts are expelled, the temperature being carefully kept below that of low red-The residue shall be moistened with concenness. trated hydrochloric acid, evaporated to dryness, treated with dilute hydrochloric acid and filtered. The potash shall be determined in the filtrate by precipitation with perchloric acid as prescribed in paragraph (v) (1) (d).

(B) Alternative method.—10 grams of the sample shall be gently incinerated in order to char organic matter, if present, and shall then be heated for 10

minutes with 10 millilitres of concentrated hydrochloric acid and finally boiled with 300 millilitres of The liquid shall be filtered into a half-litre water. flask and the residue washed. The solution shall be made up to 500 millilitres and 50 millilitres taken, boiled with solution of sodium nitrite to expel ammonium salts, if present, and evaporated to dryness. The residue shall be dissolved in water containing a little hydrochloric acid and sufficient sodium citrate added to prevent precipitation of phos-It shall then be mixed with 30 millilitres phates. of cobaltinitrite solution, in the manner described in . paragraph (v) (1) (b) (B) and the precipitate treated as therein directed.

Precipitation c_1^* potash as potassium perchlorate.—To the solution obtained as above described in paragraphs (v) (1) (a), (b) or (c) and placed in a small glass or porcelain basin, about 7 millilitres of a 20 per cent. solution of perchloric acid (specific gravity 1.125), free from chloric acid, shall be added. The basin shall be placed on a hot plate or sand bath and the contents evaporated until white fumes are copiously evolved.

The precipitate shall be re-dissolved in hot water, a few drops of perchloric acid solution added and the whole concentrated again to the fuming stage. After cooling, the residue in the basin shall be thoroughly stirred with 20 millilitres of alcohol of specific gravity 0.816 to 0.812 (95 to 96 per cent. of alcohol by volume). The precipitate shall be allowed to settle and the clear liquid shall be poured through a weighed or counterpoised filter paper, or through a gooch crucible, draining the precipitate as completely as possible from the liquid before adding the washing The precipitate shall be washed by decansolution. tation with alcohol (as above) saturated with potassium perchlorate at the temperature at which it is used, pouring the washings through the paper or gooch crucible on which the whole of the precipitate is finally collected, dried at 100° C. and weighed. The precipitate is to be regarded as $KC10_4$, and is to be calculated to its equivalent as K_2O .

(e) Preparation of the cobaltinitrite solution.—The cobaltinitrite solution shall be prepared as follows :—50 grams of cobalt nitrate and 300 grams of sodium nitrite shall be dissolved in water, acidified with 25 millilitres of glacial acetic acid and diluted to a litre. The solution shall be filtered after standing 24 hours and is then ready for use. It must be kept in the dark.

(d)

(2) Platinum chloride method.

- (a) Salts of potash free from sulphates.—A weighed portion of the sample equivalent in potash content to 1.5 to 2.0 grams of potash (K₂O) shall be dissolved in water : the solution shall be filtered if necessary and made up to 500 millilitres. The potash shall be determined in 50 millilitres of the solution by the platinum chloride method prescribed in paragraph (v) (2) (d).
 (b) Salts of potash containing sulphates.—A weighed portion
 - of the sample equivalent in potash content to 1.5 to 2.0 grams of potash ($K_{2}O$) shall be boiled with 300 millilitres of water to which 20 millilitres of hydrochloric acid have been added. Barium chloride solution shall be cautiously added, drop by drop, to the boiling solution until the sulphuric acid is completely precipitated. Any slight excess of barium shall be removed by the addition of the least possible excess of dilute sulphuric acid. The liquid shall be cooled, made up to 500 millilitres and filtered. 50° millilitres of the filtrate shall be taken and evaporated to dryness and shall then be moistened with concentrated hydrochloric acid, again evaporated to dryness, treated with a little dilute hydrochloric acid and filtered if necessary. The potash shall be determined in the filtrate by the platinum chloride method, prescribed in paragraph (v) (2) (d).

If the solution contains phosphates, iron, manganese, magnesium or other substances that would interfere with the determination of potash, the method prescribed in paragraph (v) (2) (c) is to be used instead of the method prescribed in paragraph (v) (2) (b).

(c) Potash in guanos and mixed fertilisers.—10 grams of the sample shall be gently incinerated in order to char organic matter, if present, and shall then be heated for 10 minutes with 10 millilitres of concentrated hydrochloric acid and finally boiled with 300 millilitres of water. The liquid shall be filtered, raised to the boiling point and powdered barium hydroxide shall It shall then be be added until slightly alkaline. cooled, made up to 500 millilitres and filtered. Of the filtrate. 250 millilitres shall be treated with ammonia solution and excess of ammonium carbonate and then, while boiling, with a little powdered ammonium oxalate, cooled, made up to 500 millilitres and filtered. Of the filtrate, 100 millilitres are to be evaporated in a porcelain dish to dryness. If desired, nitric acid may be added during the evaporation after free ammonia has been driven off. The residue is to be heated gently over a low flame till all ammonium

salts are expelled, the temperature being carefully kept below that of low redness. The residue shall be moistened with concentrated hydrochloric acid, evaporated to dryness, treated with dilute hydrochloric acid and filtered. The potash shall be determined in the filtrate by the platinum chloride method prescribed in paragraph (v) (2) (d).

(d) Precipitation of potash as potassium chloroplatinate.---To the solution obtained as above described in paragraphs (v) (2) (a), (b) or (c), a few drops of hydrochloric acid shall be added, if none is present, and also 10 millilitres or an excess of solution of platinum chloride containing 10 grams of platinum per 100 After evaporation to a syrupy consistency millilitres. on a water-bath, the contents of the basin shall be allowed to cool and shall then be treated with alcohol of specific gravity 0.864, being washed by decantation until the alcohol is colourless. The washings shall be passed through a weighed or counterpoised filter paper, on which the precipitate shall be finally collected, washed with alcohol as above, dried at 100° C. and weighed. The precipitate is to be regarded as K_2 Pt Cl₆ and is to be calculated to its equivalent as K.0.

(vi) Determination of free acid in sulphate of ammonia.—20 grams of the sample shall be dissolved in about 50 millilitres of neutral distilled water and the solution filtered. The filtrate shall be made up to about 250 millilitres and then titrated with decinormal sodium hydroxide solution, using two or three drops of methyl orange solution as indicator. The methyl orange solution shall contain 0.5 gram of methyl orange in a litre of water. The result shall be expressed as percentage by weight of sulphuric acid (H_2SO_4).

(vii) Determination of lime in burnt lime and in calcium hydroxide (hydrated lime).—A portion of the sample shall be rapidly ground and passed through a sieve having apertures about 0.2 millimetre square and from this specially prepared portion the quantities for determination of lime shall be weighed.

A quantity of the sample about 5 grams in weight, accurately weighed, shall be transferred to a stoppered bottle of about 1 litre capacity and moistened with 10 millilitres of alcohol neutral to phenol phthalein, to lessen the possibility of caking. 490 millilitres of a 10 per cent. solution of cane sugar (made neutral to phenol phthalein) shall be added and the bottle at once fitted into a shaking apparatus and agitated for a period of not less than four hours. The solution shall then be filtered through a dry paper into a dry vessel and 50 millilitres of the

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filtrate shall be titrated with seminormal hydrochloric acid (HC1) using phenol phthalein as indicator.

The method gives the total amount of lime present in the sample in the form of caustic lime (CaO) and of calcium hydroxide (Ca $(HO)_2$) and the result may be calculated to CaO, or Ca $(HO)_2$ as desired.

(viii) Determination of calcium carbonate in ground chalk, ground limestone and dried carbonate of lime.

- (a) A weighed quantity of the finely ground sample shall be treated with dilute hydrochloric acid until effervescence ceases, the solution filtered and the insoluble matter washed. The calcium shall be precipitated from the filtrate as oxalate and weighed as oxide. Steps shall be taken to exclude from the oxalate precipitate iron, alumina and other interfering substances.
- (b) The amount of carbon dioxide evolved on treatment of a weighed quantity of the finely ground sample with dilute acid shall be determined in a suitable apparatus.
- (c) The amount of calcium oxide determined under (a) shall be calculated to calcium carbonate, provided that the necessary equivalent of carbon dioxide is present in the sample. If less than the equivalent of carbon dioxide is present in the sample, the quantity of carbon dioxide determined under (b) shall be calculated to calcium carbonate.
- (ix) The prescribed sieve.
 - (a) The sieve* to be used for the purpose of the statement as to fineness of grinding of basic slag, ground limestone and raw phosphate or phosphate rock shall be of the following dimensions :—The standard diameter of the wire and the standard length of the side of the aperture shall be each 0.141 millimetres; no wire shall anywhere be less than 0.137 millimetres in diameter, and no aperture shall be greater than 0.155 millimetres in length of side.
 - (b) The sieving of a sample shall be carried out as follows :— 20 grams of the sample shall be dried at 100°C. and then be transferred to the sieve with the lower receiver attached. The sieve shall then be shaken for 10 minutes with occasional tapping of the sides of the sieve. At the end of 10 minutes, the material which has passed through into the lower box shall be carefully brushed out into a suitable vessel and weighed. The receiver shall be replaced and the shaking re-

^{*}The apertures of the prescribed sieve are practically identical with those of the 90 I.M.M. (Institute of Mining and Metallurgy) sieve and also of the Amandus Kahl 100 E sieve.

peated for another 10 minutes, when the sifted matter shall again be removed, mixed with the first portion and weighed. The process shall be repeated until not more than 0.2 per cent. is sifted during 10 minutes.

Soft lumps which can be caused to crumble by application of the fibres of a bristle brush shall be broken down after each shaking period, but in such manner that the hard parts of the bursh do not come into contact with the sieve. The brush shall not be used in any way to brush particles through the sieve.

Methods of Analysis of Feeding Stuffs.

12. The methods of analysis of a feeding stuff for the purposes of the Act shall be as follows :---

- (i) Preparation of the sample.
 - (a) If the sample is in a fine condition and passes through a sieve having apertures about one millimetre square, it shall be thoroughly mixed and a portion not less than 100 grams in weight shall be placed in a stoppered bottle. From this portion the quantities for analysis shall be taken.
 - (b) If the sample does not wholly pass through a sieve having apertures about one millimetre square and wholly passes through a sieve having apertures from two to three millimetres square, it shall be thoroughly mixed and a portion for the determination of the moisture shall be at once taken.
 - (c) If the sample is in a coarse condition, as, for example, pieces of broken cake, it shall be carefully pulverised antil the whole passes through a sieve having apertures from two to three millimetres square. It shall then be thoroughly mixed and a portion for the determination of the moisture shall be at once taken.
 - (d) From the mixed sample as under (b) above, or from the coarsely crushed sample as prepared under (c) above, a portion not less than 100 grams in weight shall be taken and further powdered and passed through a sieve having apertures about one millimetre square. The portion of the sample so prepared shall be placed in a stoppered bottle and from it the quantities for analysis shall be weighed.
 - (e) If the original sample is appreciably moist, or if for any reason the operations of pulverisation and mixing are likely to result in loss or gain of moisture, the moisture shall be determined in this prepared portion, as well as in the sample prepared as in paragraph (i) (b) or (c) in order that the results of the analysis may be corrected to correspond with the sample in its original condition as regards moisture.

(f) Materials which cannot be conveniently pulverised or passed through a sieve shall be thoroughly mixed by the most suitable means.

(ii) Determination of moisture (loss on drying).—A weighed quantity of the sample shall be dried at 100°C.

- (iii) Determination of oil.
 - (a) A weighed quantity of the sample shall be placed in an extraction thimble, which shall then be placed in an extraction apparatus and extracted with petroleum spirit 40-60°C. b.pt. At the end of three to four hours the thimble shall be removed from the apparatus, dried and its contents finely ground, preferably with sand, in a small mortar previously rinsed with petroleum spirit. The substance shall then be returned to the thimble, the mortar being washed out with petroleum spirit, and the extraction continued for another hour. The extract should be free from suspended matter. After evaporation of the solvent, the oil shall be dried at 100°C. and weighed.
 - (b) In the case of samples containing saccharine matter, the weighed portion in the thimble shall be washed with water and then dried, previous to the extraction.

(iv) Determination of albuminoids (protein).—The percentage of albuminoids (protein) shall be ascertained by multiplying the percentage of nitrogen, other than nitrogen present as ammoniacal or nitric nitrogen, by 6.25. The presence of nitrogen in these latter forms shall be tested for and the quantity so present, if any, shall be determined and deducted from the total nitrogen. (See methods for determination of ammoniacal nitrogen and nitric nitrogen in presence of organic matter under Methods of Analysis of Fertilisers, paragraph (iii).)

The determination of total nitrogen in the absence of nitrates shall be as follows :----

A weighed portion of the sample shall be transferred to a Kjeldahl digestion flask, 25 millilitres of concentrated sulphuric acid (or more if necessary) shall be added and the flask gently heated until frothing ceases. Ten grams of potassium or sodium sulphate (anhydrous) shall then be added and the flask further heated until the colour of the clear liquid ceases to diminish. The heating shall be continued for an hour thereafter to ensure complete oxidation of the organic matter. The operation may be accelerated by the addition of a small crystal of copper sulphate or a globule of mercury to the liquid in the digestion flask.

The quantity of ammonia present in the liquid shall be determined by distillation into standard acid after liberation

with alkali and, where mercury has been used, with the addition also of sodium or potassium sulphide solution.

The materials used shall be examined as to their freedom from nitrogen by means of a control experiment carried out under similar conditions with the same quantities of the re-agents which have been employed in the actual analysis, one gram of pure sugar being used in place of the weighed portion of the sample. The quantity of standard acid found to have been neutralised in this control experiment shall be deducted from the total quantity of acid neutralised in the distillation of the sample.

If nitrates are present, the digestion and subsequent distillation must be carried out as in Methods of Analysis of Fertilisers, paragraph (iii) (b).

(v) Determination of phosphoric acid.—A weighed portion of the sample shall be heated with concentrated sulphuric acid until all organic matter is destroyed and the phosphoric acid is completely in solution. After dilution, the solution shall be filtered, the insoluble matter thoroughly washed and the filtrate made up to a definite bulk. The phosphoric acid shall be determined by the method prescribed in Methods of Analysis of Fertilisers, paragraph (iv) (d), in an aliquot part of the solution, which shall first be nearly neutralised and then acidified with nitric acid.

(vi) Determination of fibre.-Two or three grams, accurately weighed, shall be extracted with petroleum spirit b.pt. 40-60°C. in an extraction apparatus, or at least three times by stirring, settling and decantation, and the dry residue transferred to a conical 1,000 millilitre flask. The material must not be further ground during extraction. A volume of 200 millilitres of a solution containing 1.25 grams of sulphuric acid $(H_{0}SO_{4})$ per 100 millilitres measured at ordinary temperature and brought to boiling point, shall be added to the flask and heated. The contents of the flask must come to boiling within 1 minute and the boiling throughout must be gentle and continuous for exactly 30 minutes, the original volume being maintained. The flask shall be rotated every few minutes in order to mix the contents and remove particles from the sides. At the end of 30 minutes the flask shall be removed and the contents poured at once into the shallow layer of hot water remaining in a funnel fitted with a pump-plate or alternatively into the similar layer remaining in a Buchner funnel. The funnel shall be prepared by cutting a piece of cotton cloth or filter paper to cover the holes; so as to serve as a support for a disc of ordinary filter paper; boiling water shall be poured into the funnel and allowed to remain until the funnel is hot, whereupon suction is applied. The experiment shall be discarded if the time of filtration of the bulk of the 200 millilitres exceeds 10 minutes. The residue shall be washed with boiling water until the washings are free from

acid. The residue shall then be washed from the filter paper back into the flask with a volume of 200 millilitres of a solution of sodium hydroxide, containing 1.25 grams of sodium hydroxide (NaOH) per 100 millilitres free or nearly free from sodium carbonate, measured at ordinary temperature, and brought to boiling The contents of the flask shall be boiled for exactly 30 point. minutes, the precautions given for the treatment with acid being observed. At the end of 30 minutes the flask shall be removed and its contents immediately filtered through an ordinary filter paper. The residue collected on the filter paper shall be washed with boiling water, then with a solution of 1 per cent. hydrochloric acid and again with boiling water until free from acid. The residue shall then be washed twice with 95 per cent. alcohol, and three times with ether. The residue shall then be transferred to a dried weighed ashless filter paper, dried at about 100° C. in an oven and weighed in its weighing bottle until constant in weight. The ash of the paper and contents shall be determined by incineration at a dull red heat. The weight of ash shall be subtracted from the increase of weight found on the paper and the difference shall be reported as fibre.

(vii) Determination of sugar.

- (a) When the substance is in solid form.—About 10 grams of the sample or a larger quantity if the percentage of sugar is low, accurately weighed, shall be ground up with water in a mortar and transferred to a 250 millilitre flask, using in all about 200 millilitres of cold water. The flask shall be shaken at intervals during 30 minutes. It if is necessary to use a clearing agent, basic lead acetate solution followed by sodium sulphate, or alumina cream \mathbf{free} from ammonia shall be employed. The liquid in the flask shall then be made up to 250 millilitres and filtered. The sugar shall be determined in 50 millilitres of the filtrate by the method prescribed in paragraph (vii) (c).
- (b) When the substance is in liquid form.—The prepared portion of the sample shall be thoroughly mixed immediately before weighing out the quantity for sugar determination. About 10 grams of the sample, accurately weighed, shall be washed into a 250 millilitre flask with about 200 millilitres of water and the solution cleared, if necessary, with basic lead acetate solution followed by sodium sulphate, or alumina cream free from ammonia. The liquid in the flask shall then be made up to 250 millilitres and filtered. The sugar shall be determined in 25 millilitres of the filtrate by the method prescribed in paragraph (vii) (c).

(c) The aliquot part of the filtrate obtained as described in paragraph (vii) (a) or (b) shall be measured into a 100 millilitre flask and the sugar inverted as follows: -5 millilitres of hydrochloric acid of 38.3 per cent. strength shall be added and the flask placed in a water bath maintained at 70°C. The solution in the flask should reach a temperature of 67° to 69°C. in $2\frac{1}{2}$ to 3 minutes. It is maintained at 69°C. for 7 to $7\frac{1}{2}$ minutes, the total period of heating being 10 minutes. It is then cooled at once, neutralised, bulked to 100 millilitres and filtered.

The total reducing sugar in the filtrate is determined either by gravimetric or volumetric process, the total copper-reducing power being calculated in terms of cane sugar $(C_{12}H_{22}O_{11})$.

(viii) Determination of salt in fish meal.—5 grams of the sample shall be mixed with pure lime and heated until the organic matter is completely charred. The residue shall be extracted with water, the volume made up to 250 millilitres and the solution filtered. The chlorine shall be determined in an aliquot portion of the filtrate and the result expressed in terms of NaCl.

(ix) Determination of sand, siliceous matter or other insoluble mineral matter :---

- (a) A weighed quantity of the sample, from 2 to 5 grams, shall be incinerated and the weight of the ash shall be taken.
- (b) The ash shall be moistened with hydrochloric acid and evaporated to dryness and shall then be repeatedly extracted with hot dilute hydrochloric acid (one part of concentrated hydrochloric acid to four parts of water). The solution shall be filtered and the insoluble matter washed, incinerated and weighed. The quantity obtained shall be taken as sand and siliceous matter.
- (c) Where the quantity of sand and silica-free ash is so high as to raise a presumption that mineral material has been added, the nature and quantity of such added substances shall, if possible, be determined.

Qualifications to be possessed by Agricultural Analysts and Deputy Agricultural Analysts. (Section 23 (1) (f).)

13. Every person appointed as agricultural analyst or deputy agricultural analyst shall furnish proof to the satisfaction of the Ministry that he has competent knowledge of (a) chemistry, and (b) chemical analysis and microscopy as applied to fertilisers and feeding stuffs. Such proof shall in every case comprise documentary evidence that such person holds a certificate or diploma attesting his possession of the requisite knowledge and given by a recognised competent body. All such documentary evidence

shall be submitted to the local authority making the appointment and shall be forwarded to the Ministry by the local authority when applying for approval of the appointment. The Ministry shall call for further evidence if required in any particular case.

Forms of Certificate of Agricultural Analyst. (Section 23 (1) (f).)

14. The certificate of an agricultural analyst shall be in such one of the forms A and B set forth in the Schedule hereto as may be applicable to the case.

Variations of Schedules. (Section 23 (1) (a).)

15. The Schedules to the Act are hereby varied as follows, and shall have effect accordingly :---

FIRST SCHEDULE.

PART I.

Fertilisers.

The words "Amount of dicyandiamide if in excess of 2 per cent." in the second column, in respect of calcium cyanamide, shall be omitted.

The words "(if any)" shall be inserted after "insoluble phosphoric acid" in the second column, in respect of compound fertilisers.

The words "Sulphate of ammonia (neutral)—Amount of nitrogen" shall be omitted.

FIRST SCHEDULE.

PART II.

Feeding Stuffs.

The word "Cocoanut" in the first column shall be omitted and the word "Coconut" inserted.

The words "Amounts of sugar and fibre respectively" in the second column, in respect of dried sugar beet residue, shall be omitted and the words "Amount of fibre" inserted.

The words "white fish meal" shall be inserted after "Fish meal" in the first column.

After "Maize by-products not otherwise specifically mentioned in this schedule—Amounts of oil, albuminoids (protein) and fibre respectively," there shall be inserted "Maize, flaked —Amounts of oil and albuminoids (protein) respectively."

SECOND SCHEDULE.

PART I.

Fertilisers.

The words "Calcium hydrate; slaked lime—Amount of calcium hydrate and equivalent of calcium oxide" shall be omitted and the words "Calcium hydrate; calcium hydroxide; slaked lime—Amount of calcium hydroxide and equivalent of calcium oxide" inserted.

After "Dried blood for fertilising purposes—Amount of nitrogen" there shall be inserted "Dried carbonate of lime not otherwise specifically mentioned in this schedule —Amount of calcium carbonate and equivalent of calcium oxide."

The words "Calcium hydrate is to be stated in terms of calcium hydrate $(Ca(HO)_2)$ " in the foot-notes shall be omitted and the words "Calcium hydroxide is to be stated in terms of calcium hydroxide $(Ca(HO)_2)$ " inserted.

THIRD SCHEDULE.

The words "whether ground or unground, treated or untreated," shall be inserted after the word "source" in paragraph (a).

Paragraphs (b), (c) and (d) shall be omitted and the following paragraphs inserted :---

"(b) Peat, peat moss, spent hops or sugar cane pith, treated or untreated, ground or otherwise.

(c) Wheat or rye straw, ground or otherwise.

(d) Sawdust or any other form of wood, treated or untreated."

FOURTH SCHEDULE.

PART I.

Fertilisers.

The words "Calcium hydrate; slaked lime—Commercial hydrate of lime" shall be omitted and the following words inserted: "Calcium hydrate; calcium hydroxide; slaked lime—Commercial hydroxide of lime."

After the definition of "Dried blood manure," the following shall be inserted : "Dried carbonate of lime not otherwise specifically mentioned in Part I of the Second Schedule—A product, consisting principally of calcium carbonate, arising as a by-product in manufacturing and other processes."

The following shall be omitted: "Sulphate of ammonia (neutral)—Ammonium sulphate for fertilising purposes, containing not more than 0.025 per cent. of free acid in terms of sulphuric acid."

FOURTH SCHEDULE.

PART II.

Feeding Stuffs.

After the definition of "Fish Meal; fish residue meal" the following shall be inserted : "Flaked maize—The product obtained by cooking and flaking commercially pure maize or Indian corn, either as grown or from which the germ, in whole or in part, has been removed."

The word "cocoanut" where it occurs under the entry "Nut cakes or meals," in the first column shall be omitted and the word "coconut" inserted.

FIFTH SCHEDULE.

The words "in hydrochloric acid" in the last paragraph shall be omitted.

Definitions.

16. In these Regulations-

"The Ministry" means the Ministry of Agriculture, Northern Ireland;

"Purchaser" and "seller" include their respective Agents, other than carrying Agents;

"The Act" means the Fertilisers and Feeding Stuffs Act, 1926;

"Fertiliser" means any article for use as a fertiliser of the soil;

"Cattle" means bulls, cows, oxen, heifers, calves, sheep, goats and swine;

"Feeding Stuff" means any article for use as food for cattle or poultry.

Other expressions have the same respective meanings as in the Act.

Revocation.

17. The Fertilisers and Feeding Stuffs (Ireland) Regulations, 1910, and the Fertilisers and Feeding Stuffs (Methods of Analysis —Ireland) Regulations, 1918, in so far as they apply to Northern Ireland, are hereby revoked as from the time at which these Regulations take effect.

Commencement.

18. These Regulations shall take effect on the first day of July, 1928, and shall remain in force until altered or revoked by the Ministry.

Short Title.

19. These Regulations may be cited as the Fertilisers and Feeding Stuffs (Northern Ireland) Regulations, 1928.

In Witness whereof the Official Seal of the Ministry of Agriculture, Northern Ireland, is hereunto affixed this fifth day of June, nineteen hundred and twenty-eight.

(L.S.)

(Signed)

James S. Gordon,

Secretary.

SCHEDULE.

FORM A.

Certificate for Fertiliser (1).

I, the undersigned, agricultural analyst for the (2) in pursuance of the provisions of the Fertilisers and Feeding Stuffs Act, 1926, hereby certify that I received on the day of 19, from (3) two parts of a sample of (4) for analysis which parts were duly scaled and fastened up and marked (5)

for analysis; which parts were duly sealed and fastened up and marked (5) and were accompanied by the annexed (6)

and also by a signed statement that the sample was taken in the prescribed manner; and that one of the said parts has been analysed by me, or under my direction, and I declare the results of the analysis to be as follows :----

(7)	Nitrogen (N)	••	••	••	••	••	••	per cent.
(8)	Phosphoric acid	1 (P ₂ O ₅)	}	Soluble in v Insoluble in Total	water a water	••• ••	•• •• ••	per cent. per cent. per cent.
(9)	Potash (K_2O)	••	•.•	••	••	••	••	per cent.
(10)	Calcium oxide ((CaO)	·	••	• .• •	. per	cent. d	or ·
	Calcium hydrox	ride (Ca(I	$HO)_2)$	• •	••	per	$cont. \epsilon$	quivalent.
	to calcium oz	cide (ÇaC))	••	••	per	cent. d)r
	Calcium carbon	ate (CaC	O_3)	••	••	· per	cent. e	quivalent.
	to calcium oz	ride (CaC	D)		••	per	cent.	-
·	Free acid, as su	dphúric a	acid (H_2SO_4), in	sulphate	of	• •	
	ammonia	المترقية الم	••	••	••	••		per cent.
	Amount that w	ill pass t	hroug	sh prescribe	eveia bi	· ••	••	per cent.

(11) and I am of opinion that (12)

The analysis was made in accordance with the Fertilisers and Feeding Stuffs (Northern Ireland) Regulations, 1928.

As witness my hand this

day of

19

(Signature and address of analyst.)

(1) Statements made in certificates are to be confined to matters which either are necessary to be stated for the purposes of the Act or are voluntarily stated by the seller.

(2) Here insert the name of the county, county borough, or other district.

(3) Here insert the name of the inspector or official sampler who delivered the sample, and, if so, "by post."

(4) Here insert the name of the article as stated in the statutory statement, warranty or particulars marked in or indicated by a mark applied to the article.

(5) Here insert the distinguishing mark on the sample.

(6) Here insert either "statutory statement," "copy of statutory statement," "warranty," "copy of warranty," "copy of particulars marked on the article" or "copy of particulars indicated by a mark applied to the article" as the case may be. The document annexed should be initialled by the analyst for purposes of identification.

(7) Nitrogen is to be stated in terms of nitrogen (N).

(8) Phosphoric acid is to be stated in terms of phosphoric anhydride (P_2O_5) .

(9) Potash is to be stated in terms of potassium oxide (K_2O) .

(10) Calcium oxide, calcium hydroxide and calcium carbonate are to be stated in terms of calcium oxide (CaO), calcium hydroxide (Ca(HO)₂) and calcium carbonate (CaCO₃) respectively. If any entry is made here, the portions not applicable are to be deleted.

(11) Here insert the names and percentages of other chemical or other ingredients or particulars as to the fineness of grinding, when any such statement is made in any written document (other than the statutory statement) descriptive of the article.

(12) Here state, as the case may require :-

- (a) Whether the composition of the article agrees with the statements. contained in the statutory statement, warranty, or particulars marked on or indicated by a mark applied to the article, having regard to Section 2 (3) of the Act and the definitions contained in the Fourth Schedule; and, if not, in what respects.
- (b) In what respect, if any the statutory statement, warranty or particulars marked on or indicated by a mark applied to the article are false in any material particular to the prejudice of the purchaser and in excess of the limits of variation (if any) prescribed under the Act.

FORM B.

Certificate for Feeding Stuff (1).

I, the undersigned, agricultural analyst for the (2)in pursuance of the provisions of the Fertilisers and Feeding Stuffs Act, 1926, hereby certify that I received on the day of 19, from (3) two parts of a sample of (4) for analysis; which parts were duly sealed and fastened up and marked (5).

and were accompanied by the annexed (6)

and also by a signed statement that the sample was taken in the prescribed. manner; and that one of the said parts has been analysed by me, or under my direction, and I declare the results of the analysis to be as follows :-

	Oil	•• •	•			:	•• •	per cent.
	Albuminoids	(protein) .	•					per cent.
	Fibre		•		••		••	per cent.
	Sugar		•				••	per cent.
(7)	Salt (NaCl)				••		·	per cent.
• •	Sand and oth	ler siliceous	matter		••		••	per cent.
(8)	Phosphoric a	(P_2O_5)			••		••	per cent.
(9)	-							-

and I am of opinion that (10)

The analysis was made in accordance with the Fertilisers and Feeding Stuffs (Northern Ireland) Regulations, 1928.

As witness my hand this

day of

19

(Signature and address of analyst.)

(1) Statements made in certificates are to be confined to matters which either are necessary to be stated for the purposes of the Act or are voluntarily stated by the seller.

(2) Here insert the name of the county, county borough, or other district.

(3) Here insert the name of the inspector or official sampler who delivered thesample and, if so, "by post."

(4) Here insert the name of the article as stated in the statutory statement,. warranty or particulars marked on or indicated by a mark applied to the article.

(5) Here insert the distinguishing mark on the sample.

(6) Here insert either "statutory statement," "copy of statutory statement," "warranty," "copy of warranty," "copy of particulars marked on the article" or "copy of particulars indicated by a mark applied to the article" as the case-may be. The document annexed should be initialled by the analyst for purposes. of identification.

(7) Salt is to be stated in terms of sodium chloride (NaCl).

(8) Phosphoric acid is to be stated in terms of phosphoric anhydride (P_2O_5) .

(9) Here insert the names and percentages of other nutritive or other ingredients, when any such statement is made in any written document (other than the statutory statement) descriptive of the article.

(10) Here state, as the case may require :---

- (a) Whether the composition of the article agrees with the statements contained in the statutory statement, warranty, or particulars marked on or indicated by a mark applied to the article, having regard to Section 2 (3) of the Act and the definitions contained in the Fourth Schedule; and, if not, in what respects.
- (b) In what respect, if any, the statutory statement, warranty or particulars marked on or indicated by a mark applied to the article are false in any material particular to the prejudice of the purchaser and in excess of the limits of variation (if any) prescribed under the Act.
- (c) Whether the article is suitable for feeding purposes for cattle (as defined by the Act) or for poultry, as the case may be; and, if not, in what respects.
- (d) Whether the article contains any ingredient included in the Third Schedule to the Act not expressly stated in the statutory statement; and, if so, the name of such ingredient and, if possible, the estimated percentage.
- (e) Whether the article contains any ingredient deleterious to cattle (as defined in the Act) or poultry having regard to Section 7(2) and the Fifth Schedule to the Act; and, if so, the name of such ingredient and, if possible, the estimated percentage.

FORM C.

Fertilisers and Feeding Stuffs Act, 1926.

Name of County, County Borough, or other District.....

Return to the Ministry of Agriculture, Northern Ireland, for the Quarter ended.....

FERTILISERS.

			*Sub-			Results of Analysis (percentages).											
Serial	Date	Name	mitted by In- spector	In the case of samples taken under Section 12, Nams and Address of the person on whose premises the sample was taken; or, in the case of samples taken under Section 3, Name and Address of the seller.				Ph	osphoric a (P2O5).	ciđ						Tites	
Sample. Rec	of Receipt.	oı Fer- tiliser.	(1.) or by Official Sampler (O.S.).			Mois- ture.	Nitro- gen.	Sol- uble in water.	Insol- uble in in water.	Total.	Potash (K2O),	Lime (CaO).	Calcium hydroxide (Ca(HO)2).	Calcium car- bonate (CaCO ₃).	Acidity as H ₂ SO4.	of grind- ing.	+
<u> </u>			,		Guar- anteed. Found.								*				
							5						-	· .			
		,															
•			-			·										•	
i																	

* Where an officer holds appointments as both inspector and official sampler, "I" should be inserted if the sample was taken in his capacity as inspector and "O.S." if the sample was taken in his capacity as official sampler.

[†] In this column should be given any further information relating to the composition or condition of the sample. *Note.*—If no samples have been analysed, a "nil" return should be rendered.

Signature.....

Date.....

FORM D.

Fertilisers and Feeding Stuffs Act, 1926.

Name of County, County Borough, or other District.....

Return to the Ministry of Agriculture, Northern Ireland, for the Quarter ended.....

FEEDING STUFFS.

	*Sub- mitted In the case of samples taken		*Sub- mitted by In-		Results of Analysis (Percentages).								· ·		
Serial No. of Sample.	Date of Receipt.	Name of Feed- ing Stuff.	by In- spector (I.) or by Official Sampler (O.S.).	under Section 12, Name and Address of the person on whose premises the sample was taken; or. in the case of samples taken under Section 3, Name and Address of the seller.		Mois- ture.	Oil.	Albu- minoids. (pro- tein).	Fibre.	Sugar.	Salt (NaCl).	Sand and other siliceous matter.	Phosphoric acid (P_2O_5) .	ł	Gene
					Guaranteed. Found.										ral
				•	•										Regu
															latio
															ns
•				· · · · · · · · · · · · · · · · · · ·										. <u>.</u>	

* Where an officer holds appointments as both inspector and official sampler, "I" should be inserted if the sample was taken in his capacity as inspector and "O.S." if the sample was taken in his capacity as official sampler. † In this column should be stated whether the sample contained any deleterious ingredient or any substance included in the Third Schedule and any other particulars as to the composition or condition of the sample. If estimated, percentages should be given.

Note.---If no samples have been analysed, a " nil " returned should be rendered.

Signature	
Date	

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