CHAPTER 211

FERTILIZERS AND FEEDING STUFFS REGULATIONS 9/1979.

ARRANGEMENT OF SECTIONS

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CHAPTER 211

9/1979

FERTILIZERS AND FEEDING STUFFS REGULATIONS
(Section 14)

[10th March, 1979]

Short title.

1. These Regulations may be cited as the

FERTILIZERS AND FEEDING STUFFS REGULATIONS.

Definitions.

2. In these Regulations, unless the context otherwise requires-

"Act" means the Fertilizers and Feeding Stuffs Act;

"amount of protein" means-

(a) except in the case of compound feeding stuffs or feed supplements, the amount of nitrogen, other than ammoniacal, nitrate or urea nitrogen, multiplied by 6.25;

(b) in the case of compound feeding stuffs or feed supplements, the amount of nitrogen, including urea and arnmoniacal nitrogen but not nitrate nitrogen multiplied by 6.25;

"antioxidant" means any substance which delays, retards or prevents the development in a feeding stuff or rancidity or other deterioration arising from oxidation;

"binder" means any non-nutritional substance which aids the compaction of feeding stuffs;

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"colourant " means any substance, other than a basic feed ingredient, which is added to a feeding stuff only to import colour to a feeding stuff or to an animal product;

"compound feeding stuffs" means a product, other than a feed supplement, obtained by mixing two or more materials, including at least one of the materials mentioned in the first column of Part II of the First Schedule, to these Regulations for the purpose of this definition the presence of any added substance of a kind referred to in regulation 9 shall be disregarded;

"daily ration" means the total quantity of feeding stuffs, expressed on a 12 percent moisture basis, necessary on average for an animal of a given kind: age group and level of production in order to satisfy its nutritional needs;

"emulsifier" means any substance, other than a basic feed ingredient, which aids the formation of the uniform dispersion of two or more immiscible substances;

"feed supplement" means a product obtained by mixing two or more materials, being a product of kind commonly sold or used to supplement other feeding stuffs to an extent of not more than one-twentieth of the total quantity;

"fibre" means the organic matter calculated as the result of treatment of the feeding stuff according to the method of analysis described in paragraph 7.2 of the Fourth Schedule, to these Regulations;

"oil" means the extract obtained as a result of treatment of a feeding stuff according to the method of analysis described in paragraph 3.21 or 3.22 of the Fourth Schedule, to these Regulations;

"stabiliser" means any substance, other than a basic feed ingredient, which maintains the uniform dispersion of two or more immiscible substances;

"sugar" means total reducing sugars after inversion expressed as sucrose;

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"whole feeding stuff" means a mixture of feeding stuffs which by reason of its composition, is sufficient to ensure a daily ration.

Ingredients of certain articles.

3. (1) For the purposes of section 4 any article sold under a name set out in Column I of the First Schedule, to these Regulations has the meaning and shall contain the main ingredients set out against that name in Column 2 of that Schedule and the Statutory Statement shall contain *inter alia* the percentage of the article mentioned in Column 3 thereof.

(2) For the purposes of section 6 the limits of variations permissible in respect of any article named in Column 3 of the First Schedule to these Regulations shall be that set out against that article in Column 4 of that Schedule.

Form of certificate.

4. The forms of certificate of analysis to be granted by the Analyst shall be as in the Second Schedule to these Regulations.

Deleterious substances.

5. For the purposes of section 12 (1) the following substances are declared to be deleterious substances:-

(a) Salts soluble in water, if present in a feeding stuff in proportions likely to be injurious to the health of animals.

(b) All substances (poisonous) except those naturally present in the material or materials from which the feeding stuff is derived.

(c) Sand, silicious matter or other insoluble matter not naturally associated with ingredients of the feeding stuff which do not fall within the scope of this regulation or which, even if naturally so associated are present in greater proportion than the maximum than maybe expected in average

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commercial samples of the feeding material.

6. (1) The presence of the following in the feeding stuffs shall be disclosed. Disclosure of materials in feeding stuffs.

(a) Husks, chaff, glumes, shudes, hulls, nutshells or skins of nuts, from any source, whether ground or unground, treated or untreated, when used as separate ingredients or artificial mixtures in the manufacture of feeding stuffs.

(b) Peat, peamoss, spent or sugar cane pith.

(c) Wheat or rye straw.

(d) Sawdust or any other form of wood.

(2) When the kernels naturally associated in seeds with one or other of the above mentioned under item (a) of paragraph (1) of this regulation are present in a feeding stuff along with the materials with which they are so associated, regard shall be had to the proportion of the above materials which might reasonably be expected to accompany such kernels when the seed from which they are derived is in its condition (natural), provided that feeding in this condition is regarded as a common practice in the feeding of livestock.

7. The methods by which analysis of fertilizers and feeding stuffs shall be made for the purposes of the Act shall be as set out in the Third and Fourth Schedules respectively to these Regulations. Method of analysis.

8. The use of additives shall be as set out in the Fifth Schedule to these Regulations. Use of additives.

9. (1) No person shall sell or have in his possession with a view to sale for use as a feeding stuff or import into Belize for such any material containing Prohibition.

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any added antioxidant, colourant, emulsifier, stabiliser, binder, vitamin D2 or D3, copper, cobalt, selenium, manganese, zinc, urea or any added substance of a description specified in the first column of Part V of the table in the Fifth Schedule to these Regulations unless the material complies with the provisions of that Schedule as respects content and, where appropriate marking and it shall be an offence if a sampled portion of any such material does not comply with the provisions of that Schedule as regards content.

(2) The provisions of this regulation shall not apply as respects to any antioxidant, colourant, emulsifier, stabiliser, binder, vitamin D2 or D3, copper, cobalt, selenium, manganese, zinc or urea or substance as aforesaid which is-

(a) for use only in accordance with a prescription given by a veterinary surgeon or practitioner for the treatment of a particular animal or herd under his care;

(b) a medicinal product or for use for a medicinal purpose in a feeding stuff;

(c) for use only for the purpose of scientific research or experiment and is not generally for sale, purchase or use in a feeding stuff;

(d) intended for the exportation to a destination outside Belize and is clearly marked or labelled to that effect.

(3) No person shall use as feeding stuff or import into Belize for use any material containing any added substance not being a substance of a name or description specified in any part in the table set out in the Fifth Schedule to these Regulations or in- paragraph 1*(f)* or *(g)* of that Schedule, which is deleterious to poultry, cattle or to human beings, and it shall be an offence if a

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sampled portion of any such material is shown by analysis of the sample taken from it to contain an added substance which is deleterious as aforesaid.

Marking of articles.

10. Articles I shall be marked legibly in writing, printing, or stenciling-

(a) on the article itself or on a label securely attached thereto, or

(b) where the article is packed in a number of separate packages either on the wrapper or on the container, or on a label securely attached to each of the packages, or

(c) where the article is in a bulk container or tanker-

(i) on the bulk container or tanker, or a label securely attached thereto, or

(ii) where the bulk container or the tanker is a road vehicle, on a document which clearly relates to the material which is retained in the vehicle and is readily available for inspection, or

(iii) otherwise in such a manner that the mark shall be readily apparent and unequivocally associated with the material, or

(d) where the article is loose in bags or bales in such a manner that the mark shall be readily apparent and unequivocally associated with the material.

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Posting of samples.

11. Any part of a sample required to be sent to any person shall be sent by registered post.

Analysis of samples.

12. Where a sample of a feeding stuff has been taken by an inspector in the prescribed manner and sent to an agricultural analyst for analysis, any such analysis of the oil content shall be disregarded unless it is carried out before the end of three weeks commencing the date of sampling.

Taking of samples, etc..

13. The manner of taking, dividing, marking, sealing and fastening of samples shall be as described in the Sixth Schedule to these Regulations.

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FIRST SCHEDULE
PART I FERTILIZERS
SECTION 4 (a), (b) AND (C), AND SECTION 6 (I b)

Column I	Column II	Column III	Column IV
Name of article	Meanings/Ingredients of articles	Ingredients the percentage of which are to be declared in the statutory statement	Limits of variation
Compound fertilizer	A mixture of any two or more of the materials mentioned in this table	Amounts, f any of nitrogen, potash phosphoric acid	Nitrogen, potash and phosphoric acid 10%
Ammonium nitrate	Ammonium nitrate for fertilizing purposes	Amount of nitrogen	5% of the amount stated
Ammonium suphate nitrate	A mixture of ammonium sulphate and nitrate	Amount of nitrogen	5% of the amount stated
Calcium cyanamide	Commercial calcium cyanamide	Amount of nitrogen	5% of the amount stated
Calcium nitrate	Calcium nitrate for fertilizing purposes	Amount of nitrogen	5% of the amount stated
Nitrogenous gas liquor, ammoniacal gas liquor, gas liquor	Ammoniacal liquor produced in the carbonsation of coal. free from visible tar	Amount of nitrogen	5% of the amount stated
Ammonium suphate	Ammonium sulphate for fertilizing purposes	Amount of nitrogen	5% of the amount stated
Urea	Commercially pur, urea containing less than 1.5% biuret	Amount of nitrogen	5% of the amount stated
Basic slag	A by-product containing phosphorous, obtained in the manufacture of stgeeal to which no addition had been made	Total amount of phosphoric acid	10% of the amount stated

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FIRST SCHEDULE (cont.)

Column I	Column II	Column III	Column IV
Dicalcium phosphate	Dicalcium phosphate for fertilizing purposes	Amount of phosphoric acid	5% of the amount stated
Phosphate rock, ground or otherwise	The substance obtained from minieral deposits, to which nothing else has been added	Amount of phosphoric acid	10% of the amount stated
Superphosphate	Phosphate rock treated with sulphuric acid (1) Phosphate rock treated with sulphuric acid and phosphoric acid. (2) Phosphate rock treated with phosphoric acid only.	Amount of phosphoric acid	10% of the amount stated
Superphosphate concentrated		(1) Amount of phosphoric acid	10% of the amount stated
Superphosphate, triple		(2) Amount of phosphoric acid	10% of the amount stated
Kainit	A mineral potassium salt with or without magnesium	Amount of potash	5% of the amount stated
Magnesium Kainit	Mineral postassium salt containing at least 3.6% magnesium	Amount of potash	5% of the amount stated
Potassium chloride	Potassium chloride for fertilizing purpose	Amount of potash	5% of the amount stated
Potassium sulphate	Potassium chloride for fertilizing purpose	Amount of potash	5% of the amount stated

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FIRST SCHEDULE (cont.)

Column I	Column II	Column III	Column IV
Potassium nitrate	Potassium nitrate for fertilizing purposes	Amounts of nitrogen and potash	5% of the amount stated
Potassic nitrate of soda	A mixture of sodium and potassium nitrates	Amounts of nitrogen and potash	5% of the amount stated
Chilean potash nitrate	For fertilizing purposes	Amounts of nitrogen and potash	5% of the amount stated
Potassic basic slag	A mixture of basic slag and potassium chloride or sulphate	Amounts of phosphoric acid Amount of potash	10% of the amount stated
Bone meal, obtained by grinding or otherwise treating bone	Commercially pure bone, raw, degreased or steamed, of which 90% passes a sieve of 1/4 inch square aperture	Amounts of nitrogen and phosphoric acid	10% of the amount stated
Dried blood	A product dried and ground to which no other matter has been added.	Amount of nitrogen	10% of the amount stated
Fish residues	A product dried and ground to which no other matter has been added	Amounts of nitrogen and phosphoric acid	10% of the amount stated
Hoofs	A product, crushed and ground to which no other matter has been added	Amount of nitrogen	10% of the amount stated
Hoofs and horns	A mixture of hoof and horn, crushed or ground to which no other matter has been added	Amount of nitrogen	10% of the amount stated
Horns	A product, crushed and ground to which no other matter has been added	Amount of nitrogen	10% of the amount stated
Meat and bone residue	A product of drying and grinding bone, flesh and other slaughterhouse residues	Amount of nitrogen and phosphoric acid	10% of the amount stated

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FIRST SCHEDULE (cont.)

Column I	Column II	Column III	Column IV
Oil seed fertilizers, obtained by removal of oil from seeds	The residue obtained by the removal of oil from commercially pure seed	Amount of nitrogen	10% of the amount stated
PART II FEEDING STUFFS			
Column I	**Column II**	**Column III**	**Column IV**
Name of article	Meaning/Ingredients of articles	Ingredients the percentages of which are to be declared in the statutory statement	Limits of variation
Compound feeding stuff	A mixture of any two or more of the materials mentioned in this table	Amount, if any, of protein (stating as being included therein the amount, if any of protein equivalent of urea and ammonia) and amount, if any of oil and fibre	10% of the amount stated in each case
Artificially dried grass, any other artificially dried green crop or a mixture of any of them	Any product whether ground or otherwise which (a) is obtained be artificially drying grass, green cereal or mixture of them (b) has had no other substance added thereto	Amount of protein	10% of the amount stated in each case
Coconut or copra meal or cake	The residue resulting from the removal of oil from commercially pure nut	Amount of oil and protein respectively	10% of the amount stated in each case

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FIRST SCHEDULE PART II (cont.)

Column I	Column II	Column III	Column IV
Cotton cakes or meals not decorticated	The residue resulting from the removal of oil from commercially pure cotton seed, not decorticated	Amount of oil and protein respectively	10% of the amount stated in each case
Cotton cakes or meals not decorticated or partly decorticated coton seed	The residue resulting from the removal of oil from commercially pure cotton seed from which the cortex in whole or part, has been removed	Amount of oil, protein and fibre respectively	10% of the amount stated in each case
Dried brewery grains	The material produced by drying the residue of matlted and unmalted cereals usec in brewing, to which no other matter has been added	Amount of oil and protein respectively	10% of the amount stated in each case
Dried distillery by-products (other than yeast and malt culms)		Amount of oil and protein respectively, of fibre if present in excess of 2% and calcium if present in excess of 2%	10% of the amount stated in each case
Dried yeast	A material produced by drying yeast residues to which no other matter has been added	Amount of protein	10% of the amount stated

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FIRST SCHEDULE PART II (cont.)

Column I	Column II	Column III	Column IV
Feed supplement		Instructions for mixing with other feeding stuffs or information as to use where supplement is fed direct to animals. Protein equivalent of urea, if any	Protein equivalent of urea 10%, iodine, cobalt, copper, iron, manganese, zinc, molybdenum, selenium, vitamin D2 and D3 30% or amount stated. Other vitamins no upper limit, if less, 30% of the amount stated
Feeding bone flour or any other bone product for feeding purposes	Commercially pur bone, raw or degreased from which nitrogen may have been partially removed by steam	Amounts of phosphoric acid and protein	10% of the amount stated in each case
Feeding dried blood	Blood which has been dried to which no other matter has been added	Amounts of nitrogen	10% of the amount stated
Feeding meat or bone meal	The product containing not less than 40% protein and not more 4% salt obtained by drying and grinding animal carcasses or portions thereof (excluding hoof, horn and feathers) to which no other matter has been added but which may have been preliminarily treated for the removal of fat	Amounts of oil, protein and phosphoric acid	10% of the amount stated in each case

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FIRST SCHEDULE PART 11 (cont.)

Column I	Column II	Column III	Column IV
Feeding meat meal	The product, containing not less than 55% protein and not more 4% salt obtained by drying and grinding animal carcasses or portions thereof (excluding hoof, horn and feathers) to which no other matter has been added but which may have been preliminarily treated for the removal of fat	Amounts of oil, protein and phosphoric acid	10% of the amount stated in each case
Fish meal, white fish meal	A product obtained by drying and grinding or otherwise treating fish or waste of fish, to which no other matter has been added	Amounts of oil, protein phosphoric and salt	10% of the amount stated in each case
Maize, flaked	A product obtained by cooking anf flaking commercially pure maize, either as grown, or froma which the germ in whole or part, has been removed	Amounts of oil and protein	10% of the amount stated in each case
Maize germ cake or meal	A meal or cake resulting from the grinding of maize germs or from which the oil has been removed in whole or in part	Amounts of oil and protein	10% of the amount stated in each case

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FIRST SCHEDULE PART II (cont.)

Column I	Column II	Column III	Column IV
Maize gluten feed	A by-product resulting from the removal of starch and germ from maize, to which no other matter has been added	Amounts of oil and protein	10% of the amount stated in each case
Maize meal	The meal obtained by grinding commercially pure maize, as grown	Amounts of oil and protein	10% of the amount stated in each case
Milk powders, including oil and/or fat fortified milk powders	The product obtained by drying milk from which oil may be have been removed or added	Amounts of oil and protein	10% of the amount stated in each case
Mixture of molasses and urea		Sugar and protein equivalent of urea	10% of the amount stated in each case
Molasses feeds	Any mixture, other than molasses and urea, containing not less than 10% sugar or an absorbent material and treacle or molasses	Amounts of sugar and fibre	10% of the amount stated in each case
Oil cakes or meals which are the product of undecorticated material from which oil has been removed	The product obtained from commercially pure nuts or beans	Amount of oil and protein	10% of the amount stated in each case

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FIRST SCHEDULE PART II (cont.)

Column I	Column II	Column III	Column IV
Oil cakes or meals which are the product of any one decorticated or partly decorticated material from which oil has been removed	The product obtained from commercially pure nuts or beans	Amount of oil and protein	10% of the amount stated in each case
Rice bean or meal	The by-product produced in milling shelled rice to which no other matter has been added	Amount of oil, protein and fibre	10% of the amount stated in each case
Nut cakes or meals	The residue resulting from the removal of oil from commercially pure nut kernels	Amount of oil and protein	10% of the amount stated in each case
Wheat offals or millers offals	A product of wheat separated in the process of milling and containing not more than 4% of vegetable matter, other than wheat, extracted from wheat in the process of cleaning by the maker of the offals in the production of flour	Amount of fibre	Not to exceed 10% of the amount stated

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SECOND SCHEDULE

FORM I *(Section 10)*

FQRM OF CERTIFICATE OF ANALYSIS

CERTIFICATE OF ANALYSIS OF FERTILIZER (1)

I, the undersigned, agricultural analyst for the Belize Government, in pursuance of the provisions of the , hereby certify that I received on the day of 20 , from (2) one part of a sample of (3) for amdysis; which was duly sealed and fastened up and marked (4) and was accompanied by a (5) , as follows: (6) and also by a signed statement that the sample was taken in the prescribed manner; and that the said part has been analysed by me, or under my direction, and I declare the results of analysis to be as follows: (7)

%	ppm
Nitrogen (N).....	Boron (B) .....
Phosphoric acid ($P_2 0_5$) Total	Cobalt (Co) .....
Potash ($K_2 0$)	Copper (Cu) .....
Iron (Fe)	Manganese (Mn) .....
Magnesium (Mg)	Molybdenum (Mo) .....

Neutralising value expressed in terms of calcium oxide ..... %
Names of herbicides and pesticides found % and I am of opinion that (8) .....

The analysis was made in accordance with the Fertilizers and Feeding Stuffs Regulations.
As witness my hand this day of , 20 ,

(Signature and address of analyst)

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SECOND SCHEDULE FORM 1 *(CONT.)*

(1) Statements made in the certificate are to be confined to matters which are necessary to verify compliance with the Act.

(2) Here insert the name of the inspector who submitted the sample for analysis; and also the mode of transit, i.e. "by hand", "by registered post", etc. as the case may be.

(3) Here insert the name or description applied to the material.

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

(5) Here insert either "statutory statement", "copy of statutory statement", "copy of particulars marked on the material", or "copy of particulars indicated by a mark on or indicated by a mark applied to the material", as the case may be.

(6) Here insert the particulars contained in the statutory statement, or particulars marked on or indicated by a mark applied to the material, or as the case may be.

(7) Insert relevant results under the appropriate headings, i.e. percentage or parts per million.

(8) Here enter information as follows:

(a) If the material was sold under a name mentioned in the first column of Part I of the First Schedule, state whether it accords with the meaning given in the second column; and if not, in what respect.

(b) If the composition of the material agrees with or does not differ by more than the limits of variation from the statement of particulars contained in the statutory statement, or the particulars marked on or indicated by a mark associated with the material, state that the particulars are correct within the limits of variation.

(c) If the composition of the material differs by more than the limits of variation from the particulars contained in the statutory statement, or the particulars marked on or indicated by a mark associated with the material, state the difference between the amount found and the amount stated, and that the difference is outside the limits of variation; and that the difference is to the prejudice of the purchaser, if such is believed to be the case.

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(These notes and the numbers referring to them are for guidance only and do not form part of the certificate.)

FORM II

CERTIFICATE OF ANALYSIS OF FEEDING STUFF (1)

I, the undersigned, agricultural analyst for the Belize Government, in pursuance of the provisions of the , hereby certify that I received on the day of , 20 , from (2) one part of a sample of (3) for analysis; which was duly sealed and fastened up and marked (4) and was accompanied by a (5) as follows: (6)
and also by a signed statement that the sample was taken in the prescribed manner; and that the said part has been analysed by me or under my direction, and I declare the results of analysis to be as follows: (7)

%		ppm		units/kg or IU/kg
Oil		Vitamin A		
Protein: Total, including		Vitamin D2		
Protein equivalent of urea		Vitamin D3		
Protein equivalent of urea		Vitamin E		
Fibre		Other vitamins or pro		
Sugar		Vitamins		
Salt (NaCI)		Permitted antioxidant (8)		
Phosphoric acid ($P_2 0_5$)		Permitted colourant (8)		
Calcium (Ca)		(9)		
Copper (Cu)		(10)		
Magnesium (Mg)				

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Molybdenum (Mo) .....
Selenium (Se) .....
Iron (Fe) .....
Iodine (I) .....
Cobalt (Co) .....
Manganese (Mn) .....
Zinc (Zn) .....
(11) Analysis for oil was completed on
and I am of opinion that (12)
The analysis was made in accordance with the Fertilizers and Feeding Stuffs Regulations.

As witness by my hand this day of 20 .

(Signature and address of analyst)

(1) Statements made in the certificate are to be confined to matters which are necessary to verify compliance with the Act.
(2) Here insert the name of the inspector who submitted the sample for analysis; and also the mode of transit, i. e. " by hand by registered post or as the case may be.
(3) Here insert the name or description applied to the material.
(4) Here insert the distinguishing mark on the sample and the date of sampling shown thereon.
(5) Here insert either "statutory statement", "copy of statutory statement", "copy of particulars marked on the material" or "copy of particulars indicated by a mark applied to the material", or as the case may be.
(6) Here insert the particulars contained in the statutory statement, or particulars marked on or indicated by a mark applied to the material, or as the case maybe.
(7) Insert relevant results under the appropriate headings, i.e. percentage, parts per million or units/kg or IU/kg.
(8) Here indicate whether the antioxidant or colourant is an antioxidant listed in Part I *of the table to the Fifth Schedule or a colourant listed in*

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SECOND SCHEDULE FORM 11 *(cont.)*

Part II of the table to the Fifth Schedule.

(9) Here indicate the presence of any emulsifier, stabiliser or binder not listed in *Part* III *of the table to the Fifth Schedule.*

(10) Here insert the name and estimated percentage of any ingredient found in the sample, being an ingredient deleterious to animals of any description or to human beings.

(11) In the case of a sample of any feeding stuff containing oil insert the date of completion of the oil analysis.

(12) Here enter information as follows:

(a) If the material was sold under a name mentioned in the first column of Part II, state whether it accords with meaning given in the second column; and if not, in what respect.

(b) If the composition of the material agrees with or does not differ by more than the limits of variation from the statement of particulars contained in the statutory statement, or the particulars marked on -or indicated by a mark associated with the material, state that the particulars are correct within the limits of variation.

(c) If the composition of t he material differs by more than the limits of variation from the statement of particulars contained in the statutory statement, or the particulars marked on or indicated by a mark associated with the material, or as the case may be, state the difference between the amount found and the amount stated, and that the difference is outside the limits of variation, and that the difference is to the prejudice of the purchaser if such is believed to be the case.

(d) If the material is not suitable for use as a feeding stuff having regard to the Act, state in what respect.

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THIRD SCHEDULE

METHODS OF ANALYSIS OF FERTILIZERS

(In this Schedule a "decimal" system had been adopted for the numbering of divisions and sub-divisions. Main divisions are given numbers which precede a decimal point. Each sub-division into which a main division is first divided is distinguished by a digit immediately following the decimal point. For example, the main division 5 is divided into three sub-divisions numbered 5.1, 5.2, and 5.3 respectively. Succeeding digits indicate further sub-division with the result that, for example, the sub-division numbered 5.1 may itself be divided into sub-divisions numbered 5.11, 5.12, 5.13 etc. and those sub-divisions may be further divided in the same way (thus, 5. 111, 5.112, 5.113 etc.) and so on.

The main Divisions in the Schedule are as follows:

1. Preparation of the sample for analysis.
2. Determination of moisture.
3. Determination of Nitrogen.
4. Determination of Phosphoric acid.
5. Determination of Potash.
6. Determination of neutralising value in liming materials.
7. Determination of magnesium in lime and ground limestone.
8. Determination of boron.
9. Determination of cobolt.
10. Determination of copper.
11. Determination of iron.
12. Determination of magnesium.
13. Determination of manganese,
14. Determination of molybdenum.

NOTE: Reference to "water" mean distilled water. All reagents used should be of analytical quality.

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1. Preparation of the sample for analysis

With some materials, fine grinding may lead to loss or gain of moisture, and allowance for this may be made. Grinding should be as rapid as possible and unnecessary exposure to the atmosphere avoided. Grinding in a laboratory mill is usually quicker than grinding in a mortar although the latter is permissible.

1.1 Procedure

For solid fertilizers, weigh the whole sample and then empty on to a smooth dry surface. Remove, and allow for in the calculation of results, any obvious extraneous matter e.g. metallic particles which may be present in samples of basic slag.

1.11 Dry Powdered and Granulated Fertilizers

Grind the sample as finely as possible. Mix thoroughly and take a representative portion of about 250 g. Transfer to a non-corrodible container provided with an air-tight closure.

1.12 Crystalline Fertilizers. e.g. Sulphate of potash and Nitrate of Soda

Grind the sample as finely as possible. Mix, withdraw a portion for analysis and transfer to a non-corrodible container provided with an airtight closure.

1.13 Hoof Meal

In the case of hard samples of hoof meal which cannot be ground in the "as received" condition, determine the moisture in the sample by the method described in paragraph 2. Then grind the dried portion in a mill and transfer to a non-corrodible container provided with an air-tight closure. Determine the moisture in this prepared sample and calculate the result of analysis of this sample to the "as received" condition.

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1.14 Fertilizers in a Moist Condition

Mix the sample well and withdraw a portion for moisture determination. Determine the moisture in this portion by the method described in paragraph 2. (In the case of fertilizers in which ammonia is lost on heating or of fertilizers containing soluble phosphoric acid, the sample should be dried either by placing it in a desiccator over calcium chloride or silica gel, or alternatively by passing dry air at room temperature over the sample until it is in a suitable condition for grinding and sieving). For subsequent analysis, dry a further portion under similar conditions and grind this dried portion in a mortar or mill. Mix thoroughly and transfer to a non-corrodible container provided with an air-tight closure. Determine the moisture in a portion of this prepared sample. Calculate the results of analysis of the sample to the "as received" condition.

1.13 Liquid Fertilizers

Shake to mix thoroughly, ensuring that any insoluble matter is thoroughly dispersed immediately before drawing a portion of the sample for analysis.

2. Determination of Moisture

Weigh to the nearest mg. about 5 g. of the sample, heat at 1000C for two to three hours, cool in a desiccator and weigh. Reheat for another hour, cool and reweigh. If the difference in weight exceeds 10 mg, continue the heating and cooling procedure until a weight constant within 2 mg is attained. Calculate the total loss of weight as a percentage of the original weight and regard as moisture.

3. Determination of Nitrogen

The relevant methods of analysis are described in the following paragraphs.

3.3 Total nitrogen (organic and ammoniacal) in the absence of nitrates.
3.4 Total nitrogen (organic, ammoniacal and nitrate) in the presence of nitrates.

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3.1 Reagents

Aluminium ammonium sulphate.

Devarda's alloy - finely powdered - not less than 80% to pass through a sieve having apertures of about 0.25 mm square.

Indigo carmine standard solution - Cautiously add 40 ml concentrated sulphuric acid to 1 g indigo carmine (B.P. quality) and stir until dissolved. Pour the solution into 800 ml water, cool and dilute to 1 litre. Adjust the strength of the solution to comply with the following test:

Add 20 ml to a solution of 4 mg potassium nitrate in 20 ml water. Add rapidly 40 ml concentrated sulphuric acid and heat to boiling point; the blue colour is just discharged in one minute.

Boric acid Indicator Solution - Add 5 ml of indicator solution (0.1 % methyl red and 0.2% bromocresol green in alcohol) to 1 litre saturated boric acid solution.
Paraffin Wax.
Sodium sulphate or potassium sulphate - anhydrous.
Sodium hydroxide solution, 5% w/v - Dissolve 50 g sodium hydroxide in water and dilute to 1 litre.

Sodium hydroxide solution, 50% w/v - Dissolve 500 g sodium hydroxide in water and dilute to 1 litre.
Sulphuric acid, concentrated (d = 1.84) - Nitrogen free.
Sulphuric acid, 10% v/v - To 500 ml water cautiously add 100 ml concentrated sulphuric acid. Cool and dilute to 1 litre.
Sulphuric acid, 50% v/v - To 500 ml water cautiously add 500 ml concentrated sulphuric acid. Cool and dilute to 1 litre.
Sulphuric acid (or hydrochloric acid), 0.2 N.

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3.2 Test for absence of Nitrates

Shake 5 g of the sample with 80 ml water in a 100 ml volumetric flask. Add 1 g aluminium ammonium sulphate, dilute to 100 ml. Shake well and filter into a dry beaker. Dilute 1 ml of the filtrate with 8 ml water. Add 1 ml indigo carmine solution and 10 ml concentrated sulphuric acid. Heat to boiling point. If blue colour is not discharged, regard the sample as free from nitrates.

3.3 Total Nitrogen (organic and ammoniacal) in the absence of Nitrates

3.31 Weigh to the nearest mg. about 2 g of the sample (or such an amount as shall contain not more than 250 mg nitrogen) and transfer to a kjeldahl flask. Add 25 ml concentrated sulphuric acid, approximately 0.5 g copper sulphate and 5 mg selenium and 10 g anhydrous sodium sulphate or potassium sulphate. Heat gently over a small flame until frothing ceases and the liquid is practically colourless. Continue to heat for a further two hours. Avoid local overheating. If frothing is excessive, add about 0.5 g paraffin wax.

Dissolve the cooled digest in water, and make up to a total volume of about 250 ml. Taking precautions against loss of ammonia, add sufficient sodium hydroxide solution to neutralise the acid and 10 ml in excess; mix well and connect immediately to a distillation apparatus. Distil into 10 ml of boric acid Indicator solution, diluted with 20 ml water controlling the rate of distillation so that not less than 150 ml distils in thirty minutes. Titrate the distillate against the standard 0.2 N acid solution. Carry out a similar determination using all the reagents omitting only the sample. Calculate the total nitrogen content of the sample. 1 ml 0.2 N acid = 0.0028 g nitrogen.

3.4 Total nitrogen (organic, ammoniacal and nitrate) in the presence of nitrate

Weigh to the nearest mg. about 2 g of the sample (or such an amount as shall

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contain not more than 250 mg nitrogen), transfer to a 500 mi Kjeldahl flask, add 3 g Devarda alloy and wash down the inside wall of the flask with 50 ml water. Close the flask with a rubber stopper provided with funnel and a delivery tube connected with 2 "U"-tubes (with bulbs) in series, each containing 10 ml 10% sulphuric acid. Add 5 ml 50% sodium hydroxide solution through the tap funnel. Allow to stand for thirty minutes and then heat up just below boiling point for sixty minutes. Cool, add 20 ml 50% sulphuric acid through the tap funnel, such that the sides of the flask are washed down by the acid. Remove the rubber stopper, wash the contents of the "U"-tubes into the Kjeldahl flask, add 30 ml concentrated sulphuric acid and heat until all the water has boiled off. Heat gently over a small flame until the solution is clear and then heat for a further two hours. If frothing is excessive add 0.5 g paraffin wax. Cool, carefully dilute with water, cool and transfer quantitatively to 250 mi volumetric flask. Dilute to 250 ml, mixing well and transfer an aliquot of 100 ml to a 500 ml distillation flask. Add 200 mi water and 500/c sodium hydroxide solution, until the solution is neutral, cooling during the addition. Add an additional 10 ml 50% sodium hydroxide, quickly close the distillation flask and distil about 150 ml into 10 ml boric acid/indicator solution diluted with 20 ml water. Titrate the distillate against the standard 0.2 N acid solution. Carry out a similar determination using all the reagents omitting only the sample. Calculate the total nitrogen content of the sample. 1 ml 0.2 N acid - 0.0028 g nitrogen.

4. Determination of Phosphoric acid

For the purposes of the Act, Part IV, "phosphoric acid" means $P_2 0_5$ (molecular weight 142.04).

Phosphoric acid shall be determined by the spectrophotometric, (vanadium phosphomolybdate) method.

The spectrophotometric method compares the amount of light transmitted by the solution to that by a solution of known phosphoric acid content. The determination is carried out differentially in order to increase the accuracy. Preferably an instrument with a monochromator giving a source of light with a

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wavelength of 420 nm is required; alternatively a filter instrument can be used.

4.1 Spectrophotometric (Vanadium Phosphomolybdate) Method

4.11 Reagents

Calcium oxide - finely ground.
Hydrochloric acid, concentrated (d-1.18).
Nitric acid, concentrated (d-1.42).
Potassium dihydrogen phosphate solution (stock phosphate solution) -
Dissolve in water 1.917 g potassium dihydrogen phosphate previously dried at 105^0C for 1 hour and dilute to 1 litre.
Potassium dihydrogen phosphate solution (standard phosphate solution)
Dilute 50 ml stock solution to 250 ml with water. (1 ml - 0.2 mg phosphoric acid, $P_2 0_5$).
Sodium hydroxide, N.
Vanado-molybdate reagent - Dissolve separately 20 g ammonium molybdate and 1 g ammonium vanadate in water, mix, acidify with 140 ml concentrated nitric acid and dilute to 1 litre.

4.12 Total phosphoric acid in fertilizers

4.121 Dissolution of the sample

4.1211 In the absence of organic matter

Weigh to the nearest mg. about 5 g of the sample into a 400 ml beaker, add 100 ml water and stir thoroughly. Boil the mixture, add slowly to the boiling solution 10 ml concentrated hydrochloric acid in a thin stream, and then 10 ml concentrated nitric acid; boil gently for ten minutes, cool, transfer to a 500 ml volumetric flask and dilute to the mark with water. Mix well and filter the solution through a dry filter paper into a dry flask, discarding the first 10 or 20 ml. Retain the rest of the filtrate.

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4.1212 In the presence of organic matter

Weigh to the nearest mg. about 5 g of the sample into a capsule or dish of about 5 cm in diameter; add I g calcium oxide and mix well with a stout platinum wire or thin glass rod. Calcine the mixture at a temperature not exceeding 500°C to destroy the organic matter. Allow the capsule or dish to cool and transfer the contents to a 400 ml beaker; add 100 ml water, stir thoroughly and heat to boiling point. Add slowly to the boiling solution 10 ml concentrated hydrochloric acid, and then 10 ml concentrated nitric acid, and boil gently.

If the solution is clear, continue to boil gently for ten minutes, then cool, transfer to a 500 ml volumetric flask, and dilute to the mark.

If the solution shows the presence of carbonaceous matter, filter the solution, wash the insoluble matter with a little water, and then transfer the filter paper containing the insoluble matter to the capsule or dish and calcine until all the carbon is destroyed. Allow to cool and transfer the contents to the filtrate; heat to boiling point and gently boil for ten minutes. Then cool, transfer to a 500 ml volumetric flask and dilute to the mark. Filter.

4.122 Procedure

4.1221 Standardisation of instrument

From a burette, measure into a series of 100 ml volumetric flasks 25.0, 26.0, 27.0, 28.0, 29.0, 30.0, and 31.0 ml of the standard phosphate solution (i.e. 5.0, 5.2, 5.4, 5.6, 5.8, 6.0 and 6.2 mg phosphoric acid). Add 25 ml of the vanado-molybdate reagent to each flask and dilute to 100 ml with water making sure that the temperature of the reagent and the dilution water is 20°C. Shake and allow to stand for ten minutes.

Set the spectrophotometer to the correct wavelength, circa 420 nm, fill two 1 cm cells with the 5.0 mg solution and check the extinction of the cell. If there is a small difference, select the cell with the smaller reading as the standard

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reference cell.

Determine the apparent extinction at 20°C (correct for cell differences) of the 5.2, 5.4, 5.6, 6.0 and 6.2 mg phosphoric acid solutions referred to the 5.0 mg phosphoric acid solution as standard.

Plot a calibration graph of scale reading against known phosphoric acid content.

4.1222 Analysis of sample

Successively dilute a portion of the solution prepared according to paragraph 4.1211 or paragraph 4.1212 so that the final volume of about 25 ml contains between 5.5 and 6.2 mg phosphoric acid, taking care that the dilution water is at a temperature of 20°C.

Transfer this final volume to a 100 ml volumetric flask, add 25 ml of the vanado-molybdate reagent (at a temperature of 20°C), dilute to the mark, mix, and allow to stand for ten minutes. At the same time transfer 25 ml of the standard phosphate solution (at 20°C) into a second 100 ml volumetric flask. Add 25 ml of the vanado-molybdate reagent (at 20°C), dilute to the mark, mix, and allow to stand for ten minutes.

Measure the difference in extinction at 20°C between the two solutions and estimate the phosphoric acid content of the volume of the unknown solution from the calibration graph.

Calculate the phosphoric acid content of the sample from known dilution factors and the weight of the sample.

NOTE: Prepare a fresh reference standard for each series of readings on the instrument.

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5. Determination of Potash

For the purposes of the Act, "potash" means potassium oxide (K_20) Potash in all kinds of fertilizers may be determined by the flame photometric method.

5.1 Flame Photometric Method

The determination of potash by this method depends on the measurement of the characteristic radiation emitted from a flame into which a solution of the sample is sprayed. The chosen radiation lie in the spectral range 766-770 nm. These radiations may be isolated by either a monochromator or the use of a suitable filter.

5.11 Reagents

Ammonia solution, 30% v/v - Dilute 30 ml concentrated ammonia solution (d-0.88) with water to 100 ml.

Ammonium oxalate solution - saturated aqueous solution.
Hydrochloric acid, concentrated (d-1.18).

Potassium dihydrogen phosphate solution (stock potash solution) - Dissolve in water 5.779 g potassium dihydrogen phosphate previously dried for one hour at 105°C and dilute to 1 litre.

Potassium dihydrogen phosphate solution (standard potash solutions) - Dilute 50 ml stock solution to 1 litre with water. This solution contains 100 ppm potash (K_20).

5. 12 Potassium salts

If the salts contain calcium, iron, aluminium or other interfering substances, the procedure described in paragraph 5.13 should be used instead of the following procedure.

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Weigh to the nearest mg. about 2.5 g of the sample and transfer to a 400 ml beaker. Add 5 ml concentrated hydrochloric acid 50 ml water and bring the contents to the boiling point, breaking down with a stirring rod any crystals or lumps. Dilute the solution with water to about 100 ml and boil gently for a few minutes. Cool the solution to 20°C, transfer to a 250 ml volumetric flask, and dilute to the mark. Mix and filter through a dry filter paper. Successively dilute so that the final solution contains approximately 16 ppm potash and determine the potash in the filtrate by the method described in paragraph 5.15.

5.13 Potash in mixed fertilizers containing little or no organic matter

Weigh to the nearest mg. about 2.5 g of the sample and transfer to a 400 ml beaker. Add 50 ml of water and 5 ml concentrated hydrochloric acid and evaporate to dryness on a water bath. Add 125 ml water and 50 ml ammonium oxalate solution. Boil the contents for thirty minutes. If necessary, a small quantity of a potassium-free anti-foaming agent may be added. Cool the liquid, add a slight excess of ammonia solution and cool to 20°C. Transfer to a 250 ml volumetric flask, and dilute to the mark. Mix the solution and filter through a dry filter paper. Successively dilute so that the final solution contains approximately 16 ppm potash in the filtrate by the method described in paragraph 5.15.

5.14 Potash in mixed fertilizers containing organic matter

Weigh to the nearest centigram about 10 g of the sample and gently incinerate at a temperature not exceeding 500°C in order to destroy the organic matter. Grind the residue to eliminate lumps, add 50 ml of water, 10 ml of concentrated hydrochloric acid, and evaporate to dryness on a water bath. Boil the residue for thirty minutes, with 125 ml water and 50 m ammonium oxalate solution. Cool the solution, add a slight excess of ammonia solution, cool to 20°C, transfer to a 500 ml volumetric flask and dilute to the mark. Mix the solution and filter through a dry filter paper. Successively dilute so that the final solution contains approximately 16 ppm potash and determine the potash in the filtrate by the method described in paragraph 5.15.

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5.15 Determination of Potash by Flame Photometry

5.151 Calibration of instrument

From the standard potash solution, prepare a set of accurate dilutions containing 10, 12, 14, 16, 18 and 20 ppm potash. Set the sensitivity of the flame photometer so that 100 scale divisions (full scale deflection) is equivalent to 20 ppm. potash solution. Spray the 10, 12, 14, 16, and 18 ppm potash solutions three times. Take the median reading (not the mean), and construct a calibration graph. After spraying each different strength solution, again spray the 20 ppm solution to ensure that the sensitivity of the flame photometer has not changed.

5.152 Analysis of sample

Reset the instrument at 100 scale divisions (full scale deflection) with 20 ppm potash solution. Spray the diluted fertilizer solution prepared in accordance with paragraph 5.12, 5.13 or 5.14 and read from the graph the approximate potash content of the solution.

Prepare two further dilutions of the standard potash solution to contain respectively 1 ppm more and 1 ppm less potash than the estimated potash content of the diluted solution of the sample. Successively spray the low standard solution, the diluted solution of the sample, and the high standard solution. Repeat this operation twice more. Take the median result of each set of three readings and calculate the potash content of the sample solution and hence of the fertilizer from the proportionality of the radiation given by the sample solution and that given by the two standard solutions containing respectively 1 ppm more and 1 ppm less potash than the predicted potash content.

NOTE: It is essential that the flame photometer should be set up in a vibration-free position and in a dust-free atmosphere.

Dilute standard solutions should be freshly prepared.

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6. Determination of neutralising value in liming materials

6.1 Reagents

Hydrochloric acid, 0.5 N.
Phenolphthalein indicator solution - Dissolve 0.25 g phenolphthalein in 150 ml industrial methylated spirit and dilute with water to 250 ml.
Sodium hydroxide,0.5 N - carbonate free.

6.2 Prepare the sample as described in paragraph 1.11

6.3 Procedure

Weigh to the nearest mg. about 500 mg of the sample prepared according to paragraph 6.2 and transfer to a 300 ml flask. Add 50 ml 0.5 N hydrochloric acid, cover the flask with a glass and boil the contents gently for five minutes. Cool the mixture, add two or three drops of the phenophthalein indicator solution and titrate with 0.5 N sodium hydroxide solution. Calculate by difference the volume of 0.5 N hydrochloric acid required to neutralise the sample. Express the result as percentage by weight of calcium oxide (CaO). 1 ml 0.5 N hydrochloric acid 0.01402 g calcium oxide (CaO).

7. Determination of magnesium in lime and ground limestone

7.1 Reagents

Ammonia solution, 25% v/v - Dilute 30 ml concentrated ammonia solution (d-0.91) with water to 100 ml.

Ammonium chloride solution - Dissolve 330 g ammonium chloride in water and dilute to 1 litre.

Ammonium persulphate solution - Dissolve 10 g ammonium persulphate in water and dilute to 100 ml. Store in a cool dark place for not more than one week.

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Buffer solution - Dissolve 6.75 g ammonium chloride, 62 mg magnesium sulphate ($MgS0_4.7H_20$) 93 mg disodium ethylenediamine-tetra-acetate dihydrate and 57 ml ammonia solution (d-0.88) in water and dilute to 100 ml.

Calcium standard solution - Dissolve 2.5 g calcium carbonate in 125 ml 0.5 N hydrochloric acid and dilute to 1 litre.

EDTA solution, 0.025 M - Dissolve 10 g disodium ethylenediaminetetra-acetate dihydrate in 800 ml water containing 55 ml N sodium hydroxide solution. Dilute 20 ml standard calcium solution with 30 ml water. Add 1 ml buffer solution and 200 mg Mordant Black 11; titrate with the EDTA solution to a blue end point and adjust the strength of this solution so that 1 ml is equivalent to 2.5 mg calcium carbonate ($CaC0_3$).

Hydrochloric acid, 0.5 N.
Hydrogen peroxide, 6% v/v (20 volume).

Mordant Black 11 indicator (colour index No. 14645) - Mix 200 mg Mordant Black 11 and 50 g sodium chloride uniformly and together grind to pass through a sieve having apertures of about 0.3 min square.

Murexide indicator - Mix 200 mg Murexide and 100 g sodium chloride uniformly together and grind to pass through a sieve having apertures of about 0.3 mm square. Protect this mixture from light.
Sodium hydroxide, N.

7.2 Procedure

Weigh to the nearest mg. about 1 g finely ground sample and add 50 ml 0. 5 N hydrochloric acid. Transfer to a conical flask, cover with a glass and boil for 3 minutes. Add 2 ml hydrogen peroxide solution, reboil, cool, add 1 ml ammonium chloride solution, a slight excess of 25 % ammonia solution and 1 ml ammonium persulphate solution. Remove the excess ammonia by boiling and filter the precipitate, if any, on a small paper and wash with two portions each of 10 ml

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hot water. Wash the precipitate off the paper with not more than 50 ml water, and boil with 50 ml 0.5 N hydrochloric acid. Cool the solution, add 1 ml ammonium chloride solution, a slight excess of dilute ammonia and 1 ml ammonium persulphate solution and remove the excess of ammonia by boiling. Filter and wash with hot water. Add the filtrate and washings to the filtrate and washing from the first precipitation, cool and dilute the whole to 200 ml.

If no precipitate forms on the addition of the ammonia and persulphate solutions, remove the excess of ammonia by boiling, add 6 ml ammonium chloride solution, cool and dilute to 200 ml.

If the amount of the precipitate is small, omit the second precipitation but add 6 ml ammonium chloride solution to the filtrate and washings before cooling and diluting to 200 ml.

Dilute 20 ml of the solution to 50 ml and add 3 ml 25% ammonia solution. Then add 200 mg Mordant Black 11 indicator and titrate with EDTA solution to a blue end point.

Dilute a further 20 ml of the solution to 50 ml and add 7 ml N sodium hydroxide. Then add 200 mg Murexide indicator and titrate with EDTA solution to a violet end point.

Calculate the magnesium content from the difference between the two titrations. 1 ml EDTA solution = 0.608 mg magnesium.

8. Determination of Boron

For levels above 1,000 ppm, boron is determined by titration as boric acid and for levels up to 1,000 ppm by the carmine spectrophotometric method.

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8.1 Titrimetric Method

8.11 Reagents

Calcium oxide.
Hydrochloric acid, 50% v/v - Dilute 50 ml concentrated hydrochloric acid (d = 1. 18) with water to 100 ml.
Hydrochloric acid, 0.5 N.
Lead nitrate solution - Dissolve 10 g lead nitrate in water and dilute to 100 ml.
Mannitol.
Methylated indicator solution - Dissoive 0.025 g methyl red in 5 ml 90% industrial methylated spirit with the aid of 0.5 ml 0.1 N sodium hydroxide. Dilute to 250 ml with 50% industrial methylated spirit.
Sodium carbonate.
Sodium hydroxide, 0.5 N.
Sodium hydroxide, 0.05 N - Prepare from a 50% solution which has been allowed to settle. Use boiled and cooled water for dilution. Store in a polythene bottle protected from the atmosphere by a guard tube and fitted with a syphon for withdrawing the solution.

8.12 Dissolution of the sample

8.121 In the absence of organic matter

Weigh to the nearest mg. about 2 g of the sample, if it contains 0.5% or less of boron, and 1 g if it contains from 0.5 - 1.0% of boron. Transfer to a 400 ml beaker. Add 100 ml water and some phenolphthalein indicator. Add sodium carbonate to make the solution slightly alkaline and boil gently. Keep the boiling solution just alkaline by further additions of sodium carbonate until all the ammonia which may be present has been evolved. Cool the solution, add 12 ml 50% hydrochloric acid.

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8.122 In the presence of organic matter

Weigh to the nearest mg. about 2 g of the sample, if it contains 0.5% or less of boron, and 1 g if it contains from 0.5 - 1.0% of boron. Place in a silica dish, add 0.2 g calcium oxide each 1 g of the sample, for moisten with water, mix thoroughly, evaporate the mixture to dryness and ignite gently in a muffle furnace at 450°C. Allow the washing to proceed for about three hours. Cool. Moisten with 10 ml 50% hydrochloric acid, warm on a water bath for fifteen minutes, covering the dish with a watch glass. Transfer to a 400 ml beaker, add a few drops of phenolphthalein indicator and dilute to about 120 ml with water.

8.13 Procedure

To the solution prepared in accordance with paragraph 8.121 or 8.122, add 20 ml lead nitrate solution for each 12% P_2O_5 in the sample if 2 g of the sample have been used and 10 ml lead solution for each 12% P_2O_5 in the sample if 1 g of the sample has been used. Heat just to boiling, remove from source of heat and make just alkaline by adding solid sodium carbonate. Stand on a water bath for five minutes. Cool, transfer to a 200 ml volumetric flask and dilute to the mark with water. Mix and filter through a 24 cm Whatman No. 42 (or equivalent) filter paper, rejecting the first 10 - 20 ml of the filtrate. Transfer 100 ml of the filtrate to a 250 ml beaker. Add a few drops of methyl red indicator and acidify the solution with 0.5 N hydrochloric acid. Heat almost to boiling and stir vigorously to remove carbon dioxide, adding a little more 0.5 N hydrochloric acid if the colour changes to orange or to yellow. Neutralise to methyl red with 0.5 N sodium hydroxide and make just acid with 0.5 N hydrochloric acid. Cover with a watch glass and boil gently for five minutes to expel any remaining carbon dioxide. Cool rapidly.

Place the electrodes of potentiometric titration apparatus in the beaker and adjust the pH to 6.3 by adding 0.05 N sodium hydroxide solution. Add 10 g mannitol and titrate with 0.005 N sodium hydroxide solution to final pH of 6.3. Add a further quantity of mannitol and continue the titration to a pH of 6.3. Further additions of mannitol should not alter the pH. Let x ml of 0.05 N sodium

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hydroxide be used for the titration after the addition of the mannitol.

Allow a standard value of 0. 1 ml 0.05 N sodium hydroxide solution as "blank" value.

Calculate Boron: % Boron in sample $\frac{0.1082 (x - 0.1)}{\text{Weight of sample taken}}$

8.2 Spectrophotometric (Carmine) Method

9.2 Reagents

Boric acid (stock boron solution - Dissolve 1.905 g boric acid in water and dilute to 1,000 ml at 20^0C. 1 ml = 0.333 mg boron.

Boric acid (standard boron solution) - Dilute 10 ml stock solution with water to 100 ml at 20^0C. Transfer 5, 10, 15, 20 and 25 ml of this dilute solution to 100 ml volumetric flasks and dilute to the marks with water.
These standards will contain 5, 10, 15, 20, 25 ug of boron per 3 ml.

Calcium oxide.

Establish the calibration graph as follows:

Measure amounts of standard cobalt solution corresponding to 0, 3, 6, 9, 12, 15 ug of cobalt into a series of 100 ml beakers and proceed as described above commencing at "Add 15 ml sodium citrate solution." Measure the extinctions to the solutions, and construct a graph relating the extinctions to the number of micrograms of cobalt.

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10. Determination of Copper

Copper may be determined by the diethyldithiocarbam ate spectrophotometric method or, alternatively, by the atomic absorption spectrophotometric method.

10.1 Diethyldithiocarbamate spectrophotometric method

10.11 Reagents

Ammonia solution, approximately 6 N - This may be prepared by passing gaseous ammonia into distilled water, or by purifying ammonia solution as described for EDTA-citrate solution below.

Carbon tetrachloride, redistilled.

Copper sulphate, stock solution - Dissolve 0.393 g copper sulphate, $CuS0._4.5H_20$, in 100 ml 2 N sulphuric acid and dilute to 1 litre at 20^0C with distilled water.

Copper sulphate standard solution - Dilute 5 ml stock solution to 250 ml with 2 N sulphuric acid at 20^0C immediately before use. 1 ml = 2 ug copper.

EDTA-citrate solution - Dissolve 20 g ammonium citrate and 5 of the disodium salt of ethylenediamine-tetra-acetic acid (EDTA) in distilled water and dilute to 100 ml. To purify, add 0.1 ml sodium diethyldithiocarbamate solution and extract with carbon tetrachloride. Add a further quantity of sodium diethyl dithiocarbamate solution to ensure that it is in excess.

Sodium diethyldithiocarbariiate solution - Dissolve 1 g sodium diethyl dithiocarbamate in distilled water and dilute to 100 ml; Filter the solution if it is not clear. Store the solution in the dark in a refrigerator and discard after seven days.

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Sodium hydroxide, 0. 1 N.
Sulphuric acid, 2 N.

Thymol blue indicator solution - Dissolve 0.1 g thymol blue in 2.15 ml 0.1 N sodium hydroxide and dilute to 100 ml with water.

10.12 Dissolution of the sample

Prepare a solution of the sample as described in paragraph 9.2.

10.13 Procedure

Transfer to a separating funnel a suitable aliquot (containing not more than 50 ug of copper) of the solution prepared in accordance with paragraph 9.2. Add 10 ml EDTA-citrate solution, 2 drops of thymol blue indicator solution and ammonia solution until the mixture is coloured green or bluish-green.

Cool the mixture, add 1 ml sodium diethyldithiocarbamate solution and, from a burette, 15 ml carbon tetrachloride. Stopper the funnel, shake vigorously for two minutes and allow the layers to separate. Place a piece of cotton-wool in the stem of the funnel and run off the carbon tetrachloride layer into a dry spectrophotometer cell. Avoid undue exposure of the solution to light.

Simultaneously with the test determination, carry out a blank determination on all the reagents used.

Measure immediately the extinctions of the test and blank solutions at a wavelength of 436 nm, using carbon tetrachloride in the comparison cell. Read from a previously prepared calibration graph the number of micrograms of copper corresponding to the observed extinctions of the test and blank solutions, and so obtain by difference the net measure of copper in the sample.

Establish the calibration graph as follows:

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To a series of separating funnels transfer 10 ml EDTA-citrate solution and the following amounts of standard copper solution and 2 N sulphuric acid:

Copper solution... ..0	1	2.5	5	10	15	20	25 ml
2 N sulphuric acid 25	24	22.5	20	15	10	5	0 ml

Proceed as for the test solution, as described above, commencing at "2 drops of thymol blue . . .". Measure the extinctions of the solutions and construct a graph relating the extinctions to the number of micrograms of copper.

10.2 Atomic absorption spectrophotometric method

10.21 Apparatus

Atomic absorption spectrophotometer.
Copper hollow-cathode lamp.

10.22 Reagents

Copper sulphate standard solution - Dissolve 0.393 g copper sulphate $CuS0_4.5H_20$, in 0.5 N hydrochloric acid and dilute to 100 ml with 0.5 N hydrochloric acid, 1 ml = 1 mg copper. Dilute this solution as required.
Hydrochloric acid, 0.5 N.

10.23 Procedure

Set up the instrument using the line at 324.7 nm. Prepare from the standard copper solution a series of solutions in 0.5 N hydrochloric acid, containing between 0 and 10 ppm copper. Dilute a suitable aliquot of the sample solution, prepared in accordance with paragraph 9.2, with 0.5 N hydrochloric acid to produce a standard volume of solution containing between 0 and 10 ppm copper. Prepare a blank solution from which only the sample has been omitted. Spray distilled water into the flame and zero the instrument. Spray successively, in

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triplicate, the standard solutions, sample and blank, washing the instrument through with distilled water between each spraying. Record the absorbance reading or the peak high on the recorder if a recording instrument is used. Plot the mean reading obtained for each standard solution against its copper content. Determine the copper content of the sample and blank solutions from the graph and from the difference between them calculate the copper content of the sample.

If a large number of samples is being examined one or more standard solutions must be resprayed at intervals during the course of the analyses.

11. Determination of Iron

For levels up to 1% iron is determined by the o-phenanthroline spectrophotometric method and for levels above 1% by the titrimetric method with potassium dichromate.

11. 1 o-Phenanthroline Method

11.11 Reagents

Ammonium ferric sulphate solution (stock iron solution) - Dissolve 0.863 g ammonium ferric sulphate, $Fe_2(S0_4)_3$. $(NH_4)_2SO_4.24H_20$, in water containing 2 ml perchloric acid, and dilute to 100 ml at 20^0C.

Ammonium ferric sulphate solution (standard iron solution) - Dilute 10 ml stock solution to 100 ml with water at 20^0C immediately before use. 1 ml = 100 mg iron.

Bromophenol blue indicator solution - Dissolve 0.4 g bromophenol blue in 95% ethanol and dilute to 100 ml.

Hydrochloric acid, 50% v/v - Dilute 50 ml concentrated hydrochloric acid (d = 1.18) with water to 100 ml.

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Hydrochloric acid, 20% v/v - Dilute 20 ml concentrated hydrochloric acid (d =. 1. 18) with water to 100 ml.

Nitric acid, 3096 v/v - Dilute 30 ml concentrated nitric acid (d 1.42) with water to 100 ml.

o-Phenanthroline solution - Dissolve 0.25 g o-phenanthroline in 25% ethanol and dilute to 100 ml.

Quinol solution - Dissolve 1 g quinol in water and dilute to 100 ml.

Sodium citrate solution - Dissolve 25 sodium citrate in water and dilute to 100 ml.

11.12 Dissolution of the sample

Prepare a solution of the sample as described in paragraph 9.2.

11.13 Procedure

Transfer a suitable aliquot of the solution, prepared in accordance with paragraph 9.2, to a small flask, add a few drops of the bromophenol blue indicator solution, and titrate with sodium citrate solution until the colour changes from yellow to blue. Transfer another aliquot to a 25 ml volumetric flask, add 1 ml solution, 3 ml o-phenanthroline solution and an amount of sodium citrate solution equal to the above titration, and then dilute with water to 25 ml. Allow the solution to stand for 1 hour.

Carry out a blank determination on all the reagents used.

Measure the extinctions of the test and blank solutions at a wavelength of 510 nrn using 4 cm or 1 cm cells according to the depth of colour with water in the comparison cell. Read the number of micrograms of iron equivalent to the observed extinctions of the test and blank solutions from a previously prepared

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calibration graph, and so obtain the net measure of iron in the sample.

Establish the calibration graph as follows:

Measure amounts of standard iron solution corresponding to 0, 200, 300, 400, 500, 600 ug of iron into a series of 100 ml volumetric flasks. To each add 50 ml 20% v/v hydrochloric acid, and dilute to 100 ml with water. Using 5 ml aliquots, proceed as for the test solution, as described above commencing at "Transfer a suitable aliquot of the solution .....".

Measure the extinctions of the solutions and construct a graph relating the extinctions to the number of micrograms of iron.

11.2 Titrimetric Method

11.21 Reagents

Hydrochloric acid, concentrated (d = 1.18).

Hydrochloric acid, 30% v/v - Dilute 30 ml concentrated hydrochloric acid (d = 1.18) with water to 100 ml.

Mercuric chloride solution - Dissolve 5 g mercuric chloride in water and dilute to 100 ml.

Orthophosphoric acid, concentrated (d = 1.75).

Potassium dichromate, 0.1 N.

Sodium diphenylamine-4-sulphonate indicator solution - Dissolve 0.2 g sodium diphenylamine-4-sulphonate in water and dilute to 100 ml.

Stannous chloride solution - Dissolve 15 g stannous chloride dihydrate in 30% v/v hydrochloric acid and dilute to 100 ml with 30% v/v hydrochloric acid.

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This solution should be prepared immediately before use.

Stannous chloride, dilute - Dilute 5 ml stannous chloride solution with 30% v/v hydrochloric acid to 50 ml.

Sulphuric acid, 16% v/v - To 50 ml water cautiously add 16 ml concentrated sulphuric acid (d = 1.84). Cool and dilute to 100 ml.

11.22 Dissolution of the sample

Prepare a solution of the sample as described in paragraph 9.2.

11.23 Procedure

Transfer a suitable aliquot of the solution, prepared in accordance with paragraph 9.2, to a 500 ml flask and dilute or concentrate the solution to about 20 ml. Add concentrated hydrochloric acid so that the total amount of acid present is equivalent to about 5 ml concentrated hydrochloric acid.

Heat the solution to 70 - 90°C add the - stannous chloride solution drop wise until the yellow colour has almost disappeared. Continue the addition using diluted stannous chloride solution until the solution becomes colourless or slightly green and add one or two drops more. Cool the solution rapidly to room temperature, and add 10 ml mercuric chloride solution. A small, white 'silky' looking precipitate should form. (If no precipitate forms, insufficient stannous chloride has been added; on the other hand if the precipitate is grey or black to much stannous chloride has been added. In either case the solution must be discarded). Add 200 ml water, 10 ml 16% v/v sulphuric acid, 5 ml orthophosphoric acid and 6-8 drops of indicator. Titrate with 0.1 N potassium dichromate until the indicator changes from green to violet-blue. Calculate the amount of iron in the sample using the factor 1 ml 0.1 N potassium dichromate = 0.00559 g iron.

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12. Determination of Magnesium

Magnesium may be determined by the pyrophosphate method or, alternatively, by the atomic absorption spectrophotometric method.

12.1 Pyrophosphate Method

12.11 Reagents

Ammonia solution, (d = 0.9 1).

Ammonia solution, 5 % v/v - Dilute 5 ml concentrated ammonia solution (d = 0.91) with water to 100 ml.

Ammonium phosphate solution - Dissolve 20 g diammonium hydrogen phosphate, $(NH_4)_2HP0_4$, in water and dilute to 100 ml.

Ammonium oxalate solution - saturated aqueous solution.

Calcium wash solution - Dissolve 1 g oxalic acid, $(COOH)_2.2H_20$ and 2 g ammonium oxalate, in water and dilute to 1,000 ml.

Citric acid, monohydrate.

Hydrochloric acid, concentrated (d = 1.18).

Hydrochloric acid, 20% v/v - Dilute 20 ml concentrated hydrochloric acid (d = 1.18) with water to 100 ml.

Methyl red indicator solution - Dissolve 0.025 g methyl red in 5 ml 90% industrial methylated spirit with the aid of 0.5 ml 0.1 N sodium hydroxide. Dilute to 250 ml with 50% industrial methylated spirit.

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Oxalic acid solution - Dissolve 10 g oxalic acid, $(COOH)_2.2H_20$, in water and dilute to 100 ml.

12.12 Dissolution of the sample

Prepare a soltition of the sample as described in paragraph 9.2.

12.13 Procedure

Transfer a suitable aliquot (containing approximately 50 mg magnesium) of the solution, prepared in accordance with paragraph 9.2, to a 500 ml beaker, and add 5% v/v ammonia solution until a slight precipitate is formed. Add citric acid, in small portions, until the precipitate just dissolves, and then 1 g in excess. Heat the solution to 40°C, add 0.2 ml (4 drops) of methyl red indicator solution, Neutralise with 5% v/v ammonia solution, and I ml in excess. Add oxalic acid solution until the mixture is just acid, and then 10 ml in excess. Boil the solution for one to two minutes, add 50 ml saturated ammonium oxalate solution, dilute if necessary, to 200 ml with distilled water, boil for a further minute and heat on a water bath for at least an hour. Filter through a Whatman No. 40 (or equivalent) filter paper; wash the residue thoroughly with calcium wash solution. Combine the filtrate and washings, measure the volume, transfer to a beaker, and add, while stirring with a glass rod (avoid touching the sides of the beaker with the rod), 20 ml of ammonium phosphate solution. While stirring continuously throughout, neutralise the solution with ammonia solution, added drop by drop from a burette, and add 20 ml in excess, together with a further 10 ml of ammonia solution for each 100 ml of solution in the beaker, Set the beaker aside for at least four hours or, preferably, overnight. Filter through a No. 4 sintered-silica crucible, and wash the residue with cold 5% v/v ammonia solution, ensuring that any precipitate adhering to the sides of the beaker and the glass rod is transferred to the crucible. Dry the crucible and residue, transfer to a cool muffle furnace, slowly raise the temperature to 950°C, and heat at this temperature for a half to one hour. Allow the crucible to cool in a desiccator, and weigh. Calculate the weight of the precipitate to its equivalent of magnesium

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by multiplying its weight by 0.2184.

12.2 Atomic absorption spectrophotometric method

12.21 Apparatus

Atomic absorption spectrophotometer.

Magnesium hollow-cathode lamp.

12.22 Reagents

Hydrochloric acid, 0.5 N.

Magnesium sulphate standard solution - Dissolve 1.013 g magnesium sulphate, $MgSO_4.7H_20$, in 0.5 N hydrochloric acid and dilute to 100 ml with 0.5 N hydrochloric acid, 1 ml = 1 mg magnesium. Dilute this solution as required.

Strontium chloride solution - Dissolve 15 g strontium chloride, $SrC1_6.6H_20$, in 0.5 N hydrochloric acid and dilute to 100 ml with 0.5 N hydrochloric acid.

12.23 Procedure

Set up the instrument using the line at 285.2 nm. Prepare from the standard magnesium solution a series. of solutions, in 0.5 N hydrochloric acid, containing between 0 and 3 ppm - magnesium (see Note). Dilute a suitable aliquot of the sample solution, prepared in acordance with paragraph 9.2 with 0.5 N hydrochloric acid to produce a standard volume of solution containing 0 and 3 ppm magnesium (see Note). Prepare a blank solution from which only the sample has been omitted (see Note).

Spray distilled water into the flame and zero the instrument.
Spray successively, in triplicate, the standard solution, sample and blank, washing the instrument with distilled water between each spraying. Record the

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absorbance reading, or the peak height on the recorder if a recording instrument is used. Plot the mean reading obtained for each standard solution against its magnesium content. Determine the magnesium content of the sample and blank solutions from the graph and from the difference between them calculate the magnesium content of the sample. If a large number of samples is being examined one or more standard solutions must be resprayed at intervals during the course of the analyses.

NOTE: If the sample contains phosphate add strontium chloride solution at the rate of 5 ml for each 50 ml of diluted sample solution, before adjusting to standard volume.

13. Determination of Manganese

13.1 Reagents

Orthophosphoric acid, concentrated (d = 1.75).

Potassium periodate.

Potassium permanganate (stock manganese solution) - Dissolve 0.288 g potassium permanganate in 100 to 200 ml water, add 5 ml 25% v/v sulphuric acid and dilute with water to 1 litre at 20^0C.

Potassium permanganate (standard manganese solution - Dilute 10 ml stock solution to 100 ml with water at 20^0C immediately before use. 1. ml = 10 ug manganese.

Sulphuric acid, concentrated (d = 1.84).

Sulphuric acid, 25% v/v - To 50 ml water cautiously add 25 centrated sulphuric acid (d = 1.84). Cool and dilute to 100 ml.

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13.2 Dissolution of the sample

Prepare a solution of the sample as described in paragraph 9.2.

13.3 Procedure

Transfer to a small beaker a suitable aliquot (containing not more than 70 ug of manganese) of the solution prepared in accordance with paragraph 9.2. Evaporate just to dryness at a low heat on a hot-plate, cool, add 10 ml water, 1.5 ml orthophosphoric acid and 1.5 ml concentrated sulphuric acid. Warm until the residue is dissolved and evaporate on the hot-plate at a low heat until the solution just fumes. Cool, add 3 ml water, warm again and transfer the solution to a glass-stoppered tube calibrated 10 ml. Wash the beaker with two further 3 ml quantities of water, adding these to the contents of the tube. (If there is a precipitate, allow the solution to stand and withdraw an aliquot of the clear supernatent liquid). Add 0.5 g potassium periodate, adjust the volume of the solution to just above the 10 ml mark with water and heat the loosely stoppered tube in a boiling water bath for thirty minutes. Cool, and adjust the volume to the mark with water. Carry out a blank determination on all the reagents used.

Measure the extinction of the test and blank solutions at a wavelength of 526 nm, using 1 cm cells with water, in the comparison cell. Read from a previously prepared calibration graph the number of micrograms of manganese corresponding to the observed extinctions of the test and blank solutions, and so obtain by difference the net measure of manganese in the sample.

Establish the calibration graph as follows:

Measure the amounts of the standard manganese solution corresponding to 0, 10, 20, 30, 40, 50, 60, 70 ug manganese into a series of glass-stoppered tubes calibrated at 10 ml. To each add 1.5 ml orthophosphoric acid and 1.5 ml concentrated sulphuric acid, and proceed as described above for the test solution, commencing at "Add 0.5 g potassium periodate .....". Measure the

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extinctions of the solution, and construct a graph relating the extinctions to the number of micrograms of manganese.

14. Determination of Molybdenum

14.1 Reagents

Ammonium molybdate (stock molybdenum solution) - Dissolve 1.840 g ammonium molybdate, $(NH_4)_6$ $M0_70_{24}.4H_20$ in water and dilute 1,000 ml at 20^0C.

Ammonium molybdate standard molbydenum solution) - Dilute 1 ml stock solution to 1,000 ml with water at 20^0C immediately before use. 1 ml - 1 ug molybdenum.

Ammonium ferrous sulphate solution - Dissolve 4 g ammonium ferrous sulphate in water and dilute to 100 ml.

Hydrochloric acid, N.

Hydrochloric acid, 2 N.

Potassium thiocynate solution - Dissolve 40 g potassium thiocyanate in water and dilute to 100 ml.

Sodium sulphate, anhydrous.

Solvent mixture - Mix equal volumes of carbon tetrachloride and 3-methylbutan-1-01.

Stannous chloride solution - Suspend 40 g stannous chloride dihydrate in 20 ml 6.5 N hydrochloric acid, add water to dissolve and dilute to 100 ml. Filter if turbid.

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14.2 Dissolution of the sampie

Prepare a solution of the sample as described in paragraph 9.2

14.3 Procedure

Transfer a suitable aliquot of the solution prepared in accordance with paragraph 9.2 to a 125 ml separating funnel, add I ml ammonium ferrous sulphate solution and sufficient N hydrochloric acid to bring the volume to 50 ml (see note), then add 1 ml potassium thiocynate solution and mix.

Add 1 ml stannous chloride solution, and mix again. Add exactly 7 ml solvent mixture, shake vigorously for 2 minutes and allow to separate for fifteen minutes. Filter the lower layer through a 7 cm paper into a small stoppered tube.

If the lower layer is not bright or if filtration is difficult, filter through a small suitable column packed with anhydrous sodium sulphate, solid stannous chloride and plugged with cotton wool.

Carry out a blank determination on all the reagents used.

Measure the extinction of the test and blank solutions at a wavelength of 470 nm, using 1 cm cells with water in the comparison cell.

Read the number of micrograms of molybdenum equivalent to the observed extinctions of the test and blank solutions from a previously prepared calibration graph, and so obtain the net measure of molybdenum in the sample.

Establish the calibration graph as follows:

Measure amounts of the standard molybdenum solution corresponding to 0, 5, 10, 15, 20, 25 ug molybdenum into a series of 125 ml separating funnels. Add to each funnel 1 ml ammonium ferrous sulphate and 25 ml 2 N hydrochloric acid, dilute to 50 ml and proceed as for the test solution, as described above

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beginning at "Then add 1 ml potassium thiocyanate solution and mix". Measure the extinctions of the solutions at a wavelength of 470 nm and construct a graph relating extinction to the number of micrograms of molybdenum.

NOTE: The acidity of the final solution must not exceed 1.5 N with respect to hydrochloric acid; with more strongly acid conditions, fading of the colour will occur.

FOURTH SCHEDULE

METHODS OF ANALYSIS OF FEEDING STUFFS

(A "decimal" system has been adopted for the numbering of divisions and sub-divisions in this Schedule. It is explained at the beginning of the Schedule to these Regulations).

The main divisions in this Schedule are as follows:

1. Preparation of the sample for analysis.
2. Determination of moisture.
3. Determination of oil.
4. Determination of protein.
5. Determination of ammoniacal and urea nitrogen.
6. Determination of phosphoric acid.
7. Determination of fibre.
8. Determination of sugar.
9. Determination of salt.
10. Determination of ash.
11. Determination of calcium.
12. Determination of copper.
13. Determination of magnesium.

NOTE: References to "water" means purified water as defined in the British Pharmacopoeia. All reagents used should be of analytical quality.

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1. Preparation of sample for analysis

With some materials, fine grinding may lead to loss or gain of moisture, and allowance for this must be made. Grinding should be rapid as possible and unnecessary exposure to the atmosphere avoided. Grinding in a laboratory mill is usually quicker than grinding in a mortar although the latter is permissible.

1.1 If the sample is in a fine condition, mix thoroughly and transfer a portion of not less than 100 g to a non-corrodible container provided with an airtight closure.

1.2 If the sample is not finely ground, mix thoroughly and further grind a portion of not less than 100 g. Transfer the portion so prepared to a non-corrodible container provided with an airtight closure.

1.3 If the sample is appreciably moist or if for any reason the process of grinding and mixing are likely to result in loss or gain of moisture, take a sample immediately after the preliminary mixing procedure described in paragraph 1.2 or the preliminary grinding and mixing procedure described in paragraph 1.3 for the determination of moisture by the method described in paragraph 2. Determine also the moisture content in the finally prepared sample so that the results of the analysis may be corrected to correspond with the sample in its original condition as regards moisture.

2. Determination of Moisture

Weigh to the nearest mg about 5 g of the sample, heat at 100'C for 2 to 3 hours, cool in a desiccator and weigh. Reheat for another hour, cool and reweigh. If the difference in weight exceeds 10 mg, continue the heating and cooling procedure until a weight constant within 2 mg is attained. Calculate the total loss of weight as a percentage of the original weight and regard as moisture.

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3. Determination of Oil

3.1 Reagent

Light petroleum boiling 40-60^{0}C.

3.2 Procedure

3.21 For feeding stuffs not containing milk powder and/or oil or fat fortified milk powder.

Weigh to the nearest mg about 3 - 5 g of the sample; transfer to an extraction apparatus and extract with light petroleum for a period of at least four hours. Transfer the residue of the feeding stuff from the extraction apparatus to a small mortar, grind lightly and return it to the extraction apparatus. Wash out the mortar with a small quanitity of light petroleum and add the washings to the contents of the extraction flask. Continue the extraction for at least another hour. The extract should be clear but if seen to include insoluble matter pour it through a filter paper or cotton wool plug into another weighed flask; wash the extraction flask and filter twice with light petroleum and add the washings to the contents of the second weighed flask. Remove the bulk of the solvent from the flask, dry at 100^{0}C for two hours, cool and weight. Reheat at 100^{0}C for thirty minutes, cool and again weigh. The second weight should not differ by more than 1 or 2 mg from the first weight. Regard this light petroleum extract as oil.

Where a sample is presumed to have an oil content in excess of ten percent or where there is reason to believe that the whole of the oil will not be removed from the feeding stuff in a five hour extraction, place a fresh flask on the extraction apparatus and continue the extraction with a fresh quantity of light petroleum for at least a further hour. Filter and wash into a second weighed flask; dry and weigh as described in the preceding paragraph.

3.22 For milk powders, including oil or fat fortified milk powders, and feeding stuffs containing milk powder and/or oil or fat fortified milk powder.

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3.221 Reagents

Ammonia solution (d = 0.9 1).
Diethyl ether, peroxide free.
Ethanol, 95% v/v.
Light petroleum, boiling range 40 - 60°C.

3.222 Procedure

Weigh, to the nearest 0.2 mg, 1 - 1.1 g of the feeding stuff and transfer to a fat extraction tube provided with a glass stopper and siphon tube.

Add 9 ml water, temperature 60 - 70°C, stopper the tube and shake vigorously until the sample is uniformly suspended. Cool to room temperature, add 1.5 ml ammonia solution, stopper and shake thoroughly. Add 10 ml ethanol, using some to rinse the stopper and collect the washings in the extraction tube.

Stopper the tube and shake thoroughly. Add 25 ml diethyl ether, using some to wash the stopper as before, stopper the tube and shake vigorously for ninety seconds. Cool the tube and remove the stopper cautiously so as to avoid loss of contents. Add 25 ml light petroleum, washing the stopper as before, stopper the tube and shake vigorously for ninety seconds. Allow to stand for fifteen minutes, or until the solvent layer separates cleanly. Remove the stopper, insert a tube and transfer the ethereal layer to a flask. Raise the siphon and, before removing it from the tube, wash it down with 15 ml of disthyl ether. Remove the siphon tube and rinse the tip with ether, collecting the rinsings in the flask. Add 1 ml ethanol to the tube, stopper, shake vigorously for ninety seconds, cool, remove the stopper, add 15 ml light petroleum and again shake for ninety seconds. Allow to stand for fifteen minutes or until the layer separates cleanly, fit the siphon tube and remove the solvent layer to the flask as before.

Carry out a third extraction with 15 ml diethyl ether followedby 15 ml light petroleum in the same way, collecting the solvent in the flask. Remove the

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solvent from the flask by evaporating and dry the flask lying on its side at 100°C for two hours; cool in a desiccator and weight. Reheat at 100°C for thirty minutes, cool and weigh. Add about 20 ml light petroleum to the flask and swirl gently to dissolve the oil, warming if necessary. Allow any residue to settle, then decant the supernatant solution taking care to retain any insoluble residue. Add another 20 ml light petroleum, swirl cautiously and decant as before. Repeat with further small quantities of light petroleum until all the oil has been removed from the flask. Reheat the flask, lying on its side, at 100°C for one hour, allow to cool and weigh. Record the difference in weights as the weight of oil.

4. Determination of Protein

Ascertain the percentage of nitrogen by the method described in paragraph 4.3, and calculate the percentage of protein by multiplying the result by 6.25.

4.1 Nitrogen

4.2 Reagents

Boric acid/indicator solution - add 5 ml of indicator solution (0.1% methyl red and 0.2% bromocresol green in alcohol) to 1 litre saturated boric acid solution.

Sodium hydroxide solution, 50% w/v - dissolve 500 g sodium hydroxide in water and dilute to 1 litre.

Sodium sulphate or potassium sulphate - anhydrous.
Sulphuric acid, concentrated (d = 1.84) - nitrogen free.
Sulphuric acid, (or hydrochloric acid), 0.2 N.
Paraffin wax.
Copper sulphate.

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4.3 Procedure

Weigh to the nearest mg about 2 g of the sample (or such an amount as shall contain not more than 250 mg nitrogen) and transfer to a kjeldahl flask. Add 25 ml concentrated sulphuric acid, approximately 0.5 g copper sulphate, and 10 g anhydrous sodium sulphate or potassium sulphate. Heat gently over a small flame until ffothing ceases and the liquid is practically colourless. Continue to heat for a further two hours. Avoid local overheating. If frothing is excessive, add about 0.5 g paraffin wax.

Dissolve the cooled digest in water, and make up to a total volume of about 250 ml. Taking precautions against loss of ammonia, add sufficient 50% sodium hydroxide solution to neutralise the acid and 10 ml in excess; mix well and connect immediately to a distillation apparatus. Distil into 10 ml of boric acid indicator solution, diluted with 20 ml water, controlling the rate of distillation so that not less than 150 ml distil in thirty minutes. Titrate the distillate against the standard 0.2 N acid solution. Carry out a blank test on the reagents. Express the result in terms of nitrogen. 1 ml 0.2 N = 0.0028 g nitrogen.

NOTE: Where there is reason to suspect that the sample contains nitrogen in the form of ammoniacal, nitrate or urea nitrogen, the appropriate determination should be made as described in paragraph 3.2, 3.4 of the Third Schedule or paragraphs 5.1, 5.2 of this Schedule and the amount so obtained deducted from the total nitrogen content. In the case of compound feeds containing urea, the deduction of the nitrogen content is unnecessary for the calculation of the protein content.

5.1 Determination of ammoniacal nitrogen

Weigh to the nearest mg about 5 g of the sample, transfer to a 250 ml volumetric flask, add 200 ml water and shake well to ensure solution of all water soluble matter. Dilute to 250 ml, filter and transfer 50 ml of the filtrate to a distillation flask. Add about 300 ml water and 20 ml 50% sodium hydroxide solution.

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Distil into 10 ml boric acid indicator solution, diluted with 20 ml water, controlling rate of distillation so that not less than 150 mi distil in thirty minutes. Titrate the distillate against the standard 0.2N acid solution. Express the result in terms of ammonia nitrogen. 1 ml 0.2N acid - 0.0028 g nitrogen.

5.2 Determination of urea nitrogen

5.21 Reagents

Activated charcoal.

Carrez solution 1 - dissolve 21.9 g zinc acetate dihydrate in water and 3 ml glacial acetic acid and dilute to 100 ml with water.

Carrez solution 2 - dissolve 10.6 g potassium ferrocyanide in water and dilute to 100 ml.

4-Dimethylaminobenzaldehyde solution - dissolve a 2g 4-dimethylaminobenzaldehyde in 10 ml concentrated hydrochloric acid and dilute to 100 ml with propan-2-ol.
Hydrochloric acid, 0.01 N.

Sodium acetate solution - dissolve 136 g sodium acetate ($CH_3COONa .3H_20$) in water and dilute to 1 litre.
Urea standard solution - dissolve 1 g urea in water and dilute to 100 ml.

5.22 Procedure

Weigh to the nearest mg about 5 g of the sample (or such an amount as shall contain not more than 250 mg urea) and transfer to a 250 ml volumetric flask. Add 150 ml 0.02 N hydrochloric acid, shake for thirty minutes then add 10 ml sodium acetate solution and mix well. Add 1 g activated charcoal (see note) to the flask and shake well, and stand for a further fifteen minutes. Add 5 ml Carrez solution 1, followed by 5 ml Carrez solution 2, mixing well between

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additions. Dilute the volume with water and mix well. Filter a portion through a suitable dry filter paper into a clean dry 250 ml beaker. Transfer a 10 ml aliquot of the filtrate to a 50 ml flask, add 10.0 ml 4-dimethylaminobenzaldehyde solution, dilute to 50 ml with water, mix well and allow to stand for ten minutes. Determine the extinction of the solution at 435 nm using a 1 cm cell against a blank of 10 ml 4-dimethylaminobenzaldehyde reagent diluted to 50 ml with water. Calculate the urea content of the sample by reference to a calibration graph prepared at the same time as the test sample. (mg urea x 0.4665 = mg urea nitrogen).

Establish the calibration graph as follows:

Measure amounts of standard urea solution corresponding to 50, 100, 150, 200 and 250 mg of urea into a series of 250 ml volumetric flasks and proceed as described above commencing at "Add 150 ml 0.02 N hydrochloric acid ....". Measure the extinctions of the solution, and construct a graph relating the extinctions to the milligrams of urea.

NOTE: If the sample is highly coloured due to the presence of molasses the proportion of activated charcoal must be increased to 5 g. The final solution after filtering should be colourless.

6. Determination of Phosphoric Acid

For the purposes of Act Part IV "phosphoric acid" means $P_2 0_5$ (molecular weight 142.04). Phosphoric acid shall be determined by the spectrophotometric (vanadium phosphomolybdate) method.

6.1 Spectrophotometric (Vanadium Phosphomolybdate Method)

6.11 Reagents

Calcium oxide - finely ground.

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Hydrochloric acid, concentrated (d = 1.18)
Nitric acid, contentrated (d = 1.42).

Potassium dihydrogen phosphate solution (stock phosphate solution) -dissolve in water 1.917 g potassium dihydrogen phosphate previously dried at 105°C for one hour and dilute to 1 litre.

Potassium dihydrogen phosphate (standard phospate solution) - dilute 50 ml stock solution to 250 ml with water 1 ml = 0.2 mg phosphoric acid ($P_2 0_5$).

Vanado-molybdate reagent - dissolve separately 20 g ammonium molybdate and 1 g ammonium vanadate in water, mix, acidify with 140 ml concentrated nitric acid and dilute to 1 litre.

6.12 Dissolution of the sample

Weigh to the nearest mg about 5 g of the sample into a capsule or dish; and 1 g calcium oxide, mix well and thoroughly wet with a little water. Dry the mixture and incinerate at a temperature not exceeding 500°C until completely charred. Cool, transfer the contents of the capsule or dish to a 250 ml beaker and add 10 ml water; then add slowly 12 ml concentrated hydrochloric acid, taking suitable precautions to avoid loss by effervescence, and finally 5 ml of concentrated nitric acid. Heat to incipient boiling and keep at this temperature for ten minutes. Dilute with about 10 ml of water, filter, transfer the insoluble matter to the filter paper with a minimum amount of water and wash twice with small volumes of water. Then transfer the filter paper and insoluble matter to the original capsule or dish and incinerate until all the carbon is destroyed. Combine the ash with the filtrate and heat to boiling point. Cool, transfer to 250 ml volumetric flask, dilute to the mark, mix well and filter. Discard the first 10 or 20 ml of the filtrate.

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6.13 Procedure

6.131 Standardisation of instrument

From a burette measure into a series of 100 ml volumetric flask 25.0, 26.0, 27.0, 28.0, 29.0, 30.0 and 31.0 ml of the standard phosphate solution (i.e. 5.0, 5.2, 5.4, 5.6, 5.8, 6.0, and 6.2 ing phosphoric acid). Add 25 ml of the vanado-molybdate reagent to each flask and dilute to 100 ml with water making sure that the temperature of the reagent and the dilution water is 20°C. Shake and allow to stand for ten minutes.

Set the spectrophotometer to the correct wavelength, circa 420 nm, fill two 1 cm cell with the 5.0 mg solution and check the extinction of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent extinction at 20°C (corrected for cell differences) of the 5.2, 5.4, 5.6, 5.8, 6.0 and 6.2 mg phosphoric acid solution referred to the 5.0, mg. phosphoric acid solution as standard.

Plot a calibration graph of scale readings against known phosphoric acid content.

6.132 Analysis of sample

Successively dilute a portion of the solution prepared according to paragraph 6.12 so that the final volume of about 25 ml contains between 5.5 and 6.2 mg phosphoric acid, taking care that the dilution water is at a temperature of 20°C.

Transfer this final volume to a 100 ml volumetric flask, add 25 ml of the vanado-molybdate reagent (at a temperature of 20°C), dilute to the mark, mix, and allow to stand for ten minutes. At the same time transfer 25 ml of the standard phosphate solution (at 20°C) into a second 100 ml volumetric flask. Add 25 ml of the vanado-molybdate reagent (at 20°C), dilute to the mark, mix

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and allow to stand for ten minutes.

Measure the difference in extinction at 20^{0}C between the two solutions and estimate the phosphoric acid content of the volume of the unknown solution from the calibration graph.

Calculate the phosphoric acid content of the sample from known dilution factors and the weight of the sample.

NOTE: Prepare a fresh reference standard for each series of readings on the instrument.

7. Determination of Fibre

7. 1 Reagents

Alcohol - industrial methylated spirit.
Diethyl ether.
Hydrochloric acid, 1% v/v - dilute 10 ml concentrated hydrochloric acid with water to 1 litre.
Light petroleum - boiling point 40 - 60^{0}C.
Sodium hydroxide, 0.313 N - this solution must be free from sodium carbonate.
Sulphuric acid, 0.255 N.

7.2 Procedure

Weigh to the nearest mg about 2.7 to 3.0 g of the sample, transfer to an extraction apparatus and extract with light petroleum. Alternatively, extract with light petroleum stirring, settling and decanting three times. Air dry the extracted sample and transfer to a dry 1,000 ml conical flask (see note). Add 200 ml 0.2555 N sulphuric acid measured at ordinary temperature and brought to boiling point, the first 30 or 40 ml being used to disperse the sample, and heat to boiling point within 1 minute. An appropriate amount of anti-foaming agent may be added if necessary. Boil gently for exactly thirty minutes, maintaining a constant volume

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and rotating the flask every few minutes in order to mix the contents and remove particles from the sides.

Meantime, prepare a Buchner fpnnel fitted with a perforated plate by adjusting a piece of cut cotton cloth or filter paper to cover the holes in the plate so as to serve as a support for a circular piece of suitable filter paper. Pour boiling water into the funnel, allow to remain until the funnel is hot and then drain by applying suction. Care should be taken to ensure that the filter paper used is of such quality that it does not release any paper fibre during this and subsequent washings.

At the end of the thirty minutes boiling period, allow the acid mixture to stand for one minute and then pour immediately into a shallow layer of hot water under gentle suction on the prepared funnel. Adjust the suction so that the filtration of the bulk of the 200 ml is completed within ten minutes. Repeat the determination if this time is exceeded.

Wash the insoluble matter with boiling water until the washings are free from acid; then wash back into the original flask by means of a wash bottle containing 200 ml 0.313 N sodium hydroxide solution measured at ordinary temperature and brought to boiling point. Boil for thirty minutes with the same precautions as those used in the earlier boiling and treatment. Allow to stand for one minute and then filter immediately through a suitable filter paper. Transfer the whole of the insoluble material to the filter paper by means of boiling water, wash first with boiling water then with 1% hydrochloric acid, and finally with boiling water until free from acid. Then wash twice with alcohol and three times with ether. Transfer the insoluble matter to a dried weighed ashless filter paper and dry at 100^0C to a constant weight. Incinerate the paper and contents to an ash at a dull red heat. Subtract the weight of the ash from the increase of weight on a paper due to the insoluble material, and report the difference as fibre.

NOTE: In the event of the sample containing 3% or more of calcium carbonate (chalk or limestone flour), it will be necessary to remove the calcium carbonate before digesting the sample with acid. This can be done at the stage in the

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procedure when the portion taken for analysis has been extracted with light petroleum. The original weight taken for the determination should be such that the actual amount of feeding stuff free from calcium carbonate is between 2.7 and 3.0.

Transfer the air-dried extracted sample to a 1,000 ml conical flask, add a quantity of 196 hydrochloric acid more than sufficient to neutralise the calcium carbonate present and stir well. Allow to settle, decant off the supernatant liquid through a filter and wash the residue twice by decantation with water, passing the washings through the filter. Allow the residue and the filter to drain thoroughly. Bring 200 ml 0.255 N sulphuric acid (measured at ordinary temperature) to boiling point and use a portion of this to wash any particles on the filter back into the flask. Add the remainder of the acid to the flask and heat to boiling point within one minute. Continue the determination as described in paragraph 7.2.

8. Determination of Sugar

For the purposes of the Act, Part IV, "sugar" means total reducing sugars after inversion expressed as sucrose.

Declarations of sugar are required only in respect of molasses, treacle molasses feeds and molassed beet pulp. It is necessary, therefore, as the first procedure, to "clean" the sugar from impurities, or from its absorbent body. The total reducing sugar content is then determined after inversion of the sucrose.

8.1 Reagents

Fehling's solution - mix equal volumes of a solution of copper sulphate and a solution of sodium potassium tartrate prepared as follows:

Copper sulphate solution - dissolve 69.28 g copper sulphate ($CuS0_4 .5H_20$) in water and dilute to 1 litre.

Sodium potassium tartrate solution - dissolve 346 g sodium potassium tartrate

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and 100 g sodium hydroxide in water and dilute to 1 litre.

NOTE: The strength of the Fehling's solution should be such that 10 ml is equivalent to 0.0525 g invert sugar. It should be checked by titrating with a solution of pure sucrose (inverted by the procedure described in the Note following paragraph 8.223) using the procedure described in paragraph 8.223.

Hydrochloric acid, N.

Methylene blue solution - dissolve 2.5 g methylene blue in water and dilute to 250 ml.

Phenolphthalein indicator solution - dissolve 0.25 g phenolpthalein in 150 ml industrial methylated spirit and dilute with water to 250 ml.

Potassium ferrocyanide solution - dissolve 106 g potassium ferrocyanide in water and dilute to 1 litre.

Potassium oxalate solution - dissolve 50 g potassium oxalate in water and dilute to 1 litre.

Sodium hydroxide, 10% w/v - dissolve 100 g sodium hydroxide in water and dilute to 1 litre.

Zinc acetate solution - dissolve 219 g zinc acetate and 30 ml glacial acetic acid in water and dilute to 1 litre.

8.2 Procedure

8.21 Preparation of the sample

8.211 When the substance is in solid form

Weigh to the nearest centigram about 10 g of the sample or a sufficient quantity

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to contain about 2 g sugar. Grind in a mortar with hot water (temperature not to exceed 60°C) and transfer to a 500 ml volumetric flask using in all about 400 ml water. Shake the flask at intervals during thirty minutes. Add 5 ml potassium Qxalate solution to the contents of the flask, followed by 5 ml zinc acetate solution; mix well and then add 5 ml potassium ferrocyanide solution, make up with water to 500 ml at the correct temperature, mix well and filter. Determine the sugar in 100 ml of the filtrate by the method described in paragraph 8.22.

8.212 When the substance is in liquid form

Weigh to the nearest mg about 5 g of the sample and wash with water into a 250 ml volumetric flask using about 200 ml water. To clear the solution add 5 ml zinc acetate solution. Mix, then add 5 ml potassium ferrocyanide solution, again mix, dilute to 250 ml, mix and filter. Determine the sugar in 25 ml of the filtrate by the method described in paragraph 8.22.

8.22 Determination of the sugar content

8.221. Transfer the measured volume of filtrate obtained as described in paragraph 8.211 or paragraph 8.212 to a 300 ml beaker, add 15 ml N hydrochloric acid, dilute to 150 ml with water, cover with a watch glass and heat to boiling point. Continue to boil for two minutes, cool, add two or three drops of phenolphthalein indicator solution, just neutralise with 10% sodium hydroxide solution, transfer to a 200 ml volumetric flask and dilute to 200 ml. Filter if necessary.

8.222 Preliminary Estimation

(This estimation is usually necessary where the percentage of sugar is unknown.) - Transfer exactly 10 ml Fehling's solution to a 250 ml conical flask and add 20 ml water. Add from a burette approximately 10 ml of the filtrate prepared as described in paragraph 8.221, heat to boiling point and boil briskly for one minute. Add three drops of methylene blue solution and titrate from the burette at the rate of 1 ml per fifteen seconds until the blue colour is discharged, the

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contents of the flask being kept boiling throughout the titration. Note the total number of ml required and call this X ml. This titration should not be outside the range of 15 - 40 ml otherwise the determination should be repeated using a more appropriate volume of the filtrate.

8.223 Exact Determination

To a 10 ml Fehling's solution in a 250 ml conical flask add from a burette (X - 1) ml of the filtrate prepared as described in paragraph 8.221, together with sufficient water to make a total volume of 60 ml. Heat to boiling point, boil briskly for one and a half minutes and add three drops of methylene blue solution. Titrate from the burette at the rate of approximately 0.25 ml per fifteen seconds until the blue colour is discharged, the contents of the flask being kept boiling briskly throughout the titration which must not take more than one and a half minutes. Then the total number of ml used in the determination equals the sugar equivalent of 10 ml Feliling's solution.

10 ml Fehling's solution = 0.0525 g invert sugar.

Not more than 1 ml of filtrate should be required for the completion of the titration. If more than 1 ml is required, then the determination should be repeated using a more closely calculated volume of filtrate for the original addition. The time taken from the initial boiling point until the end of the titration should be about three minutes. If this time is exceeded by more than about twenty seconds, the titration should be repeated.

The total copper reducing power should be calculated as invert sugar and multiplied by 0.905 give sucrose.

NOTE: The Fehling's solution may be standardised as follows:

Dissolve 2.375 g sucrose (dried at 100^{0}C) in about 100 ml water in a 300 ml beaker, add 15 ml N hydrochloric acid and sufficient water to give a volume of 150 ml. Heat to boiling point, boil for two minutes, cool, add two or three

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drops of phenolphthalein solution, just neutralise with 10% soldium hydroxide solution, transfer to a 500 ml volumetric flask and dilute to 500 ml. Then follow the procedure described in paragraph 8.223.

1 ml of this solution - 0.00475 g sucrose = 0.005 g invert sugar, i.e., 10 ml Fehling's solution = 10.5 ml of this standard invert sugar solution.

9. Determination of Salt

9.1 Reagent

Calcium oxide - finely ground - this reagent must be free from chloride.

9.2 Procedure

Weigh to the nearest mg about 5 g of the sample, mix with 1 g calcium oxide and wet with water to a thick paste. Dry the mixture, grind to a fine powder and heat to a termperature not exceeding 500^{0}C until all the organic matter has been thorough charred. Extract the residue with repeated portions of hot water, filter, cool the filtrate and dilute to 250 ml in a volumetric flask. Determine the chloride in an aliquot part of the filtrate and express the result in terms of soldium chloride (NaCl).

10. Determination of Ash

Weigh to the nearest mg from 2 to 5 g of the sample, incinerate at a temperature not exceeding 500^{0}C until the carbon has been destroyed. Cool, weigh and regard as ash.

11. Determination of Calcium

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

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11.1 Oxalate Method

11.11 Reagents

Ammonia solution, 2% v/v dilute 25 ml concentrated ammonia solution (d = 0.91) 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% v/v - dilute 50 ml concentrated hydrochloric acid (d - 1.18) with water to 100 ml.

Potassium permanganate, 0.1 N.

Sulphuric acid, 20% 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.

11.12 Dissolution of the sample

Weigh to the nearest mg, about 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% 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

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to dryness. Continue heating for at least one hour to dehydratd any silica which may be present. Cool, add 20 ml water and 10 ml 50% v/v hydrochloric acid, bring to the boil and filter into a 250 ml volumetric flask. Wash the basin the filter with hot water collecting the washings in the flask. Cool, make up to volume and mix.

11.13 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 fifteen minutes, allow to cool and stand for at least four 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% v/v ammonia solution and wash the beaker and crucible with 2% 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% 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 thirty seconds, transferring the crucible to the beaker towards the end of the titration.
1 ml 0. 1 N $KMn0_4$ = 2.0 mg calcium

11.2 Atomic Absorption Spectrophotometric Method

11.21 Apparatus

Atomic absorption spectrophotometer.

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Calcium hollow-cathode lamp.

11.22 Reagents

Calcium stock solution - dry calcium carbonate at 105°C for one 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 = 1 mg calcium.

Calcium dilute solution - dilute 20 ml calcium stock solution to 200 ml. 1 ml = 100 ug 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 = 100 ug calcium) into the flasks and dilute to 100 ml with water. The flasks contain 0, 3, 6, 9, 12, 15 ug Ca per ml respectively.

Lanthanum oxide solution (releasing agent) - wet 117.3 g lanthanurn oxide, La_2O_3, low in calcium with water. Add 350 ml concentrated hyhdrochloric acid (d - 1.18) slowly, and shake until all the lanthanum oxide is dissolved. Allow to cool and dilute to 1 litre with water.

11.23 Procedure

Set up the instrument using the line at 422.7 nm. Use a fuel rich flame. Add releasing agent and wafer to a suitable aliquot of the sample solution, prepared in accordance with paragraph 11.12 to produce a standard volume of solution to contain between 5 and 10 ug of calcium per ml and 10% v/v releasing agent. Prepare a blank solution from which only the sample has been omitted. Spray water into the frame 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

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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.

12. Determination of Copper

Copper may be determined by the diethyldithiocarbamate spectrophotometric method or alternatively by the atomic absorption spectrophotometric method.

12.1 Diethyldithiocarbamate Spectrophotometric Method

12.11 Reagents

Ammonia solution, approximately 6 N - this may be prepared by passing gaseous ammonia into distilled water, or by purifying ammonia solution as described for EDTA-citrate solution below.

Carbon trtrachoride, redistilled.

Copper sulphate, stock solution - dissolve 0.393 g sulphate, $CuS0_4.5H_20$, in 100 ml 2 N sulphuric acid and dilute to 1 litre at 20^0C with distilled water.

Copper sulphate standard solution - dilute 5 ml stock solution to 250 ml with 2 N sulphuric acid at 20^0C immediately before use. 1 ml = 2 mg copper.

EDTA-citrate solution - dissolve 20 g ammonium citrate and 5 g of the disodium salt of ethylenediamine-tetra-acetic acid (EDTA) in distilled water and dilute to 100 ml. To purify, add 0.1 ml sodium diethyldithiocarbamate solution and extract with carbon tetrachloride. Add a further quantity of sodium diethyldithiocarbamate solution to ensure that it is in excess.

Sodium dithyldithiocarbamate solution - dissolve 1g sodium diethyldithiocarbamate in distilled water and dilute to 100 ml. Filter the solution if it is not clear. Store the solution in the dark in a refrigerator and discard after

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seven days.

Sodium hydoxide, 0.1 N.

Sulphuric acid, 2 N.

Thymol blue indicator solution - dissolve 0.1 g thymol blue in 2.15 ml 0.1 N sodium hydroxide and dilute to 100 ml with water.

12.12 Preparation of sample

Grind the sample to pass a stainless steel sieve having apertures about 1 mm square. With some materials, fine grinding may lead to loss or gain of moisture, and allowance for this must be made. Grinding should be as rapid as possible and unnecessary exposure to the atmosphere avoided.

A moisture determination should be carried out on the sample "as received" and again on the sample after grinding, before analysis.

12.13 Dissolution of the sample

Weigh to the nearest mg about 10 g of the sample into a silica basin, cover with a silica clock glass, and place in a cool muffle furnace. Raise the temperature to 450 + 10°C, and allow to wash overnight; a slow movement of air through the furnace during the initial stages of ashing is desirable. In the case of high-fat materials, care must be taken to avoid ignition of the sample.

When all the organic matter has been destroyed, cool, add 10 ml 50% v/v hydrochloric acid, and evaporate to dryness on a water bath. Extract the soluble salts from the residue with two successive 10 ml portions of boiling 2 N hydrochloric acid, decanting the solution each time through the same Whatman No. 451 (or equivalent) filter paper into a 50 ml volumetric flask. Then add 5 ml 50% hydrochloric acid and about 5 ml 30% v/v nitric acid to the residue in the basin, and take the mixture to dryness on a hot-plate at low heat. Finally,

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add a further 10 ml boiling 2 N hydrochloric acid to the residue and filter solution through the same paper into the flask. Dilute the combined extracts to the mark with distilled water, washing the filter paper in the process.

12.14 Procedure

Transfer to a separating funnel a suitable aliquot (containing not more than 50 ug of copper) of the solution prepared in accordance with paragraph 12.13. Add 10 ml EDTA-citrate solution, two drops thymol blue indicator solution and ammonia solution until the mixture is coloured green or bluishgreen. Cool the mixture, add 1 ml sodium diethyldithiocarbamate solution and, from a burette, 15 ml carbon tetrachloride. Stopper the funnel, shake vigorously for two minutes and allow the layers to separate. Place a piece of cotton-wool in the stem of the funnel and run off the carbon tetrachloride layer into a dry spectrophotometer cell. Avoid undue exposure of the solution to light.

Simultaneously with the test determination, carry out a blank determination on all the reagents used.

Measure immediately the extinctions of the test and blank solutions at a wavelength of 436 nm, using carbon tetrachloride in the comparison cell. Read from a previously prepared calibration graph the number of the micrograms of copper corresponding to the observed extinctions of the test and blank solutions, and so obtain by difference the net measure of copper in the sample.

Establish the calibration graph as follows:

To a series of separating funnels transfer 10 ml EDTA-citrate solution and the following amounts of standard copper solution and 2 N sulphuric acid-

Copper solution ..... 0 1 2.5 5 10 15 20 25 ml
2 N sulphuric acid ... 25 24 22.5 20 15 10 5 0 ml

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Proceed as for the test solution, as described above, commencing at "two drops thymol blue . . ...". Measure the extinctions of the solutions and construct a graph relating the extinctions to the number of micrograms of copper.

12.2 Atomic Absorption Spectrophotometric Method

12.21 Apparatus

Atomic absorption spectrophotometer.

Copper hollow-cathode lamp.

12.22 Reagents

Copper sulphate standard solution - dissolve 0.393 g copper sulphate, $CuSO_4.5H_20$, in 0.5 N hydrochloric acid and dilute to 100 ml with 0.5 N hydrochloric acid. 1 ml - 1 mg copper. Dilute this solution as required.

Hydrochloric acid, 0.5 N.

12.23 Procedure

Set up the instrument using the line at 324.7 nm. Prepare from the standard copper solution a series of solutions, in 0.5 N hydrochloric acid, containing between 0 and 10 ppm copper. Dilute a suitable aliquot of the sample solution, prepared in accordance with paragraph 12.13, with 0.5 N hydrochloric acid to produce a standard volume of solution containing between 0 and 10 ppm copper. Prepare a blank solution from which only the sample has been omitted. Spray distilled water into the flame and zero the instrument. Spray successively, in triplicate, the standard solutions, sample and blank, washing the instrument through with distilled water between each spraying. Record the galvanometer deflection, or the peak height on the recorder if a recording instrument is used. Plot the mean reading obtained for each standard solution against its copper content. Determine the copper content of the sample and blank solutions from

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the graph and from the difference between them calculate the copper content of the sample.

If a large number of samples is being examined one or more standard solutions must be resprayed at intervals during the course of the analyses.

13. Determination of Magnesium

Magnesium may be determined by the pyrophosphate method or, alternatively, by the atomic absorption spectrophotometric method.

13.1 Pyrophosphate Method

13.11 Reagents

Ammonia solution, (d-0.91).

Ammonia solution, 5% v/v - dilute 5 ml concentrated ammonia solution (d - 0.91) with water to 100 ml.

Ammonium oxalate solution - saturated aqueous solution.
Ammonium phosphate solution - dissolve 20 g diammonium hydrogen phosphate, $(NH_4)_2HP0_4$, in water and dilute to 100 ml.

Calcium wash solution - dissolve 1 g oxalic acid, $(COOH)_2.H_20$ and 2 g ammonium oxalate in water and dilute to 1,000 ml.

Citric acid - monohydrate.
Hydrochloric acid, concentrated (d = 1.18).

Hydrochloric acid, 20% v/v - dilute 20 ml concentrated hydrochloric acid (d - 1.18) with water to 100 ml.

Methyl red indicator solution - dissolve 0.025 g methyl red in 5 ml 90% industrial

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methylated spirit with the aid of 0.5 ml 0.1 N sodium hydroxide. Dilute to 250 ml with 50% industrial methylated spirit.

Oxalic acid solution - dissolve 10 g oxalic acid, $(COOH)_2.2H_20$, in water and dilute to 100 ml.

13.12 Dissolution of the sample

Prepare a solution of the sample as described in paragraph 12.13.

13.13 Procedure

Transfer a suitable aliquot (containing approximately 50 mg magnesium) of the solution, prepared in accordance with paragraph 12.13, to a 500 ml beaker, and add 5% v/v ammonia solution until a slight precipitate is formed. Add citric acid, in small portions, until the precipitate just dissolves, and then 1 g in excess. Heat the solution to 50°C, add 0.2 ml (four drops) methyl red indicator solution. Neutralise with 5% v/v ammonia solution, and add 1 ml in excess. Add oxalic acid solution until the mixture is just acid, and then 10 ml in excess. Boil the solution for one to two minutes, add 50 ml saturated ammonium oxalate solution, dilute if necessary, to about 200 ml with distilled water, boil for a further minute, and heat on a water bath for at least one hour. Filter through a Whatman No. 40 (or equivalent) filter paper, wash the residue thoroughly with calcium wash solution.

Combine the filtrate and washings, measure the volume, transfer to a beaker and add, while stirring with a glass rod (avoid touching the sides of the beaker with the rod), 20 ml ammonium phosphate solution. While stirring continuously throughout, neutralise the solution with ammonia solution added drop by drop from a burette and add 20 ml in excess, together with a further 10 ml ammonia solution for each 100 ml of solution in the beaker. Set the beaker aside for at least four hours or, preferably, overnight.

Filter through a No. 4 sintered silica crucible and wash the residue with cold

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5% v/v ammonia solution, ensuring that any precipitate adhering to the sides of the beaker and the glass rod is transferred to the crucible. Dry the crucible and residue, transfer to a cool maffle furnace, slowly raise the temperature to 950°C, and heat at this temperature for a half to one hour. Allow the crucible to cool in a dessicator and weigh. Calculate the weight of the precipitate to its equivalent of magnesium by multiplying its weight by 0.2184.

13.2 Atomic Absorption Spectrophotometric Method

13.21 Apparatus

Atomic absorption spectrophotometer.

Magnesium hollow-cathode lamp.

13.22 Reagents

Hydrochloric acid, 0.5 N.

Magnesium sulphate standard solution - dissolve 1.013 g magnesium sulphate, $MgS0_4.7H_20$, in 0.5 N hydrochloric acid. 1 ml = 1 mg magnesium. Dilute this solution as required.

Strontium chloride solution - dissolve 15 g strontium chloride, $SrCl_2 6H_20$, in 0.5 N hydrochloric acid and dilute to 100 ml with 0.5 N hydrochloric acid.

13.23 Procedure

Set up the instrument using the line at 285.2 nm. Prepare from the standard magnesium solution a series of solution, in 0.5 N hydrochloric acid, containing between 0 and 3 ppm magnesium (see note). Dilute a suitable aliquot of the sample solution, prepared in accordance with paragraph 12.13, with 0.5 N hydrochloric acid to produce a standard volume of solution containing between

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0 and 3 ppm magnesium (see note). Prepare a blank solution from which only the sample has been omitted (see note). Spray distilled water into the flame and zero the instrument. Spray successively, in triplicate, the standard solutions, sample and blank, washing the instrument through with distilled water between each spraying. Record the galvanometer deflection, or the peak height on the recorder if a recording instrument is used. Plot the mean reading obtained for each standard solutions against its magnesium content. Determine the magnesium content of the sample and blank solutions from the graph and from the difference between them calculate the magnesium content of the sample. If a large number of samples is being examined one or more standard solutions must be resprayed at intervals during the course of the analyses.

NOTE: If the sample contains phosphate add strontium chloride solution, at the rate of 5 ml for each 50 ml diluted sample solution, before adjusting to standard.

FIFTH SCHEDULE

PERMITTED ADDITIVES AND PROVISIONS RELATING TO THEIR USE

1. Subject to the following provisions of this Schedule, no material intended for use as a feeding stuff shall contain-

(a) any other antioxidant other than an antioxidant of a name and description specified in Part I of the Table in this Schedule nor any added antioxidant of name and description so specified in proportions which, taking account of any such antioxidant which is normally present, exceed 150 parts per million in whole feeding stuffs either separately or in combination with other antioxidants so specified;

(b) any added colourant other than a colourant of a name specified in Part II of the Table to this Schedule nor in the case of material intended for use as a feeding stuff for poultry any added colourant of a name specified in section A of the said Part II of the Table to this Schedule in propor-

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tions, which, taking account of any such colourant which is naturally present, exceed eighty parts per million in the whole feeding stuff either separately or in combination with other colourants so specified;

(c) any added emulsifier, stabiliser or binder other than an emulsifier, stabiliser or binder of a name or description specified in Part III of the Table to this Schedule;

(d) any added vitamin D2 or vitamin D3, save that material intended for use as a feeding stuff for any animal of a kind specified in the second column of.

(i) Section A Part IV of the Table to this Schedule may contain vitamin D2 or D3 (but not both added) in proportions which taking account of any such vitamin which is naturally present, do not exceed those specified in the third column thereof in relation to kind of animal;

(ii) Section B of the said Part IV may contain vitamin D3 in proportions which taking account of any vitamin D3 which is naturally present, do not exceed those specified in relation thereto in the third column thereof in relation to the kind of animal.

(e) any added substance of a description specified in the first column of Part V of the Table to this Schedule in proportions which, taking account of any such substance which is naturally present, exceed those specified in relation thereto in the second column of the Table;

(f) any added copper save that intended for use as a feeding stuff-

(i) for pigs may contain copper (whether naturally present or added) in proportions not exceeding one hundred parts per million;

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(ii) for any other kind of animal other than pigs or sheep may contain copper (whether naturally present or added) in proportions not exceeding fifty parts per million;

(iii) for sheep (whether natural or added) not exceeding twenty-five parts per million;

(g) any added urea save that intended for use as a feeding stuff for bulls, cows, steers, heifers, calves, sheep or goats may contain urea.

2. If the material is intended for mixing with other materials before use as a feeding stuff and it contains any added substance mentioned in Part I, II, IV or V of the Table to the Schedule or added copper in proportions which, taking account of any substance or copper which is naturally present, do not exceed in each case five times the maximum content specified in relation to the substance in paragraph 2 above or in the Table below, that material may be imported into Belize or sold or processed with a view to sale for use as a feeding stuff if it is marked with the following statement "Feeding Stuffs Regulations. This feeding stuff may only be used for (X) up to a quantity of (Y) grammes per kilogramme." The statement shall be completed by inserting at (X) the kind and, if appropriate the age group of the animal for which the material is intended and at (Y) by inserting such a figure that if the statement is put into effect, the material used as a feeding stuff will comply with the preceding provisions of this Schedule. In this statement they may be substituted for the words "grammes per kilogramme; the symbol "lb per cwt" or "lb per ton".

3. If the material containing any added iron, iodine, cobalt, manganese, selenium, zinc, vitamins (other than vitamins A, D or E) or pro-vitamins, in conformity with the provisions of this Schedule is marked with a statement, additional to the statutory statement required by section 4, containing the name and statement of the total amount present (whether naturally present or added) if any iron, iodine, cobalt, manganese, selenium, zinc, vitamins (other than vitamins A, D or E) or pro-vitamins respectively it shall be marked in a manner specified in section 4.

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4. The drugs mentioned in Part VI to this Schedule shall be permitted drugs for the purposes of this Schedule.

TABLE

PART I

PERMITTED ANTIOXIDANTS

Name	*Chemical Description*
Octyl gallate	Octyl 3, 4, 5-trihydroxybenzoate
Dodecyl gallate	Dodecyl 3, 4, 5-trihydroxybenzoate
N-propyl gallate	N-propyl 3, 4, 5-trihydroxybenzoate
BHA	Mixture of 3- and 2-tertbutyl 4-hydroxyanisole
BHT	2, 6-di (tert-butyl)-4-methylphenol
Ethoxyquin	6-ethyoxy-1,2-dihydroxy-2, 2-4-trimethylquinoline

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PART II

PERMITTED COLOURANTS

Section A	*Section B*
Capasanthen	Patent Blue V
Lycopen	Curcumin
Beta-8,-apo-carotenal	Amaranth
Ethyl ester of beta-8-apocarotenoic acid	Tartrazine
Lutein	Orange G
Cryptoxanthin	Green S
Violaxanthin	Indigo carmine
Canthaxanthin	Brilliant Black (Black PN)
Zeaxanthin	Carmoisine
	Ponceau 4R
	Sunset Yellow FCF
	Brown KF
	Red 6B

PART III

PERMITTED EMULSIFIERS, STABILIZERS AND BINDERS

Lecithin, alginic acid, sodium alginate, potassium alginate, ammonium alginate, calcium alginate, 1,2-dihydroxypropyl alginate, agar-agar, carragen, carob seed flour, tamarind seed flour, guar, guar gum, guar seed flour, gum tragacanth, gum arabic, sorbitol, mannitol, glycerol, pectin, methyl cellulose, carboxymethyl cellulose, hydroxy-propyl-methyl cellulose, ethyl methyl cellulose, sodium,

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potassium or calcium salts of food fatty acids derived from edible oils and fats, or from distilled food fatty acids, mono and di-glycerides of food fatty acids, mono- and di-glycerides of food fatty acids esterified with the following acids:

acetic, lactic, citric, tartaric and mono-acetyltartaric and dicetyl-tartaric, sucrose esters of food fatty acids, sucro-glycerides of mono- and diglycerides of food fatty acids, polyglycerol esters of the nonpolymerised fatty acids, propylene glycolesters of the food fatty acids, sodium stearoyl-2-lactylate, calcium stearoyl-2-lactylate, lignosulphonates, koalin, bentonites and other montmorillonite c lays, silica, silicates, gelatine, sodium hexametaphosphate, sorbitan esters, polyoxethylene sorbitan esters, disodium ethylenediamine tetraacetate, vermiculite and esters of polyethylene glycol.

PART IV

VITAMINS D

Vitamin	*Kind of Animal*	*Maximum content (International Units per kilogramme in the whole feeding stuff)*
	SECTION A	
	Pigs	2,000
	Piglets	10,000 in milk replacer feeds only
Vitamin D2	Cattle	4,000
or	Calves	10,000 in milk replacer feeds only
Vitamin D3	Sheep	4,000
	Lambs	10,000 in milk replacer feeds only
	Horses	4,000
	Other kinds except poultry	2,000

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SECTION B

Vitamin D3	Laying hens	3,000
	Poultry other than laying hens	2,000

Note: In this part of the table "milk replacer feed" means a manufactured feed used as a substitute for natural milk.

PART V

TRACE ELEMENTS

Elements	*Maximum Contents (Part per million in whole feeding stuff)*
Iron	1,250
Iodine	40
Cobalt	10
Manganese	250
Zinc	250
Molybdenum	2.5
Selenium	0.5

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PART VI

PERMITTED DRUGS

Amprolium
Sulphaquinoxaline

NOTE 1: In the case of material of any description specified in the first column of Part II of this Schedule, the Statutory Statement shall contain the particulars, or in the case of any feed supplement the instructions as to handling or use, specified in relation to that material in the second column hereof and also, where there has been added in the course of manufacture or preparation for sale-

(a) any copper or magnesium, a statement of the total present (whether naturally present or added) or any copper (if present in excess of fifty parts per million) or magnesium (if present in excess of 0.5 %);

(b) any antioxidant or colourant, either the words "contains permitted antioxidant" or "contains permitted colourant" as appropriate, or the name of the antioxidant or colourant;

(c) any vitamin A, D or E, the name of the vitamin and a statement of the total amount present (whether naturally present or added) and an indication of the period during which that amount will remain present;

(d) any trace element named in Part V of the Fifth Schedule, a statement of the total amount of trace elements present (whether naturally present or added);

any amount referred to-

(i) in sub-paragraph (a) above shall be expressed as a percentage by weight (unless the amount present is less than 0.1% by weight in which case it shall be expressed in parts per million);

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(ii) in sub-paragraph (c) above shall be expressed in international units per kilogramme or units per kilogramme;

(iii) in sub-paragraph (d) above shall be expressed in parts per million.

SIXTH SCHEDULE

MANNER OF TAKING, DIVIDING, MARKING, SEALING AND FASTENING OF SAMPLES

Sections 9 (1) and (2)

PART I

PROVISIONS APPLICABLE TO BOTH FERTILIZERS AND FEEDING STUFFS

A. General Provisions

1. In the case of material in any form of package, only unopened packages which appear to the inspector to be the original packages of the material shall be selected for the purpose of the sample.

2. Samples shall not be drawn from any part of the sampled portion which appears to have been damaged.

3. An inspector who proposes to take a sample shall satisfy himself that the conditions in which the material is stored are not such as may cause deterioration of the material and that the material appears not to have been

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contaminated by any other material.

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

B. Provisions applicable where the fertilizer or feeding stuff is in solid condition.

5. It shall be assumed that the sampled portion is composed of separate approximately equal parts and 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 sampled portion is in bulk, to be taken in accordance with paragraph 1 (b) 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. Where material in packages which an inspector has reasonable cause to believe has been purchased, not for resale in the course of trade but for the purpose of use as a fertilizer or feeding stuff, as the case may be, has been delivered to the purchaser and is to be sampled but some of the consignment is no longer present the number of packages to be selected shall be calculated as if not less than the whole consignment were still present.

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

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Provisions applicable where the fertilizer or feeding stuff is in a liquid or semi-liquid condition

8.

(a) In bottles or containers each not containing more than one quart. The number of bottles or containers to be selected shall be taken at random in accordance with the appropriate scale for solid fertilizers in paragraph 1 (a) of Part II of this Schedule. The entire contents of the selected containers shall be emptied into a clean dry vessel and well mixed. From this mixture a sample of between one and two litres shall be drawn.

(b) In containers of more than one litre and not more than 200 litres. The number of containers to be selected shall be taken at random in accordance with the appropriate scale for solid fertilizers in paragraph 1 (a) of Part II of this Schedule. The selected containers shall be well shaken. An approximately equal portion of liquid shall then be taken immediately from each of the selected containers and emptied into a clean dry vessel. From this mixture a sample of between one and two litres shall be taken immediately after the mixture has been thoroughly mixed.

(c) In a bulk container or containers containing more than 200 litres.

(i) If the liquid is homogenous, about one litre shall be drawn from a convenient outlet in the container, into a clean dry vessel, after running off sufficient of the liquid to ensure removal of any residues in the outlet.

(ii) If the liquid is not homogenous the contents must be stirred thoroughly and the sampling proceed as in sub-paragraph (i).

(iii) When the sampled portion consists of two or more containers,

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a sample from each, drawn in the manner described in subparagraph (i) or (ii), as appropriate, shall be placed in a clean dry vessel. After thorough mixing of a sample of about one litre shall be transferred to a clean dry vessel.

TABLE

Quantities of liquid or semi-liquid fertilizers or semi-liquid fertilizers and feeding stuffs to be withdrawn in accordance with sub-paragraph (c) (iii)

Where the sampled portion	*Quantity to be withdrawn*
does not exceed 5,000 litres	not less than one litre
does not exceed 50,000 litres	not less than two litres
exceeds 50,000 litres	not less than ten litres

PART II

PROVISIONS APPLICABLE TO SOLID FERTILIZERS

1.

(a) Where the fertilizer is in packages, a number of packages shall be selected in accordance with the following table:

	No. of packages to be selected for sampling each package
Where the sampled portion consists of up to three packages	3
Where the sampled portion consists of more than three but not more the twenty packages	4
Where the sampled portion consists of more than twenty but not more than sixty packages	6

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	No. of packages to be selected for sampling each package
Where the sampled portion consists of more than sixty but not more than one hundred packages...	8
Where the sampled portion consists of more than one hundred but not more than four hundred packages	10
Where the sampled portion consists of more than four hundred packages	20

Where the number of packages has been selected n accordance with the sub-paragraph either-

(i) the selected packages shall be emptied separately on a clean surface and well mixed with a shovel and one shovel full taken from each and the shovelsful so taken then be thoroughly mixed;

(ii) when the material is of suitable nature, a portion shall be taken from each selected package by means of a closed sampling spear and the separate portions so taken shall be thoroughly mixed.

From the mixture so obtained, the sample shall be drawn in the following manner-

Heap the material to form a "cone", flatten the cone and quarter it. Reject two diagonally opposite quarters, mix the remainder and continue quartering and rejection until the remainder is from 500 g to 1,000 g in weight. Alternatively the reduction in size of the gross sample by the quartering method may be effected by the use of a mechanical device known as a sample divider or riffle.

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(b) In bulk-

Where the fertilizer is in bulk, a number of portions shall be taken by a shovel or a closed sampling spear as follows:

Where the sampled portion does not exceed 100 kg	not less than 1 per 25 kg or part thereof
Where the sampled portion exceeds 100 kg but does not exceed 1,000 kg	not less than 6
Where the sampled portion exceeds 1,000 kg but does not exceed 10,000 kg	not less than 20
Where the sampled portion exceeds 25,000 kg	not less than 40

The portions, according to whether they have been taken by a shovel or spear, shall be treated in the manner described in paragraph 1(a) and the sample drawn in the manner described in that paragraph.

Where the fertilizer is in a coarse condition the shovelsful shall be crushed immediately and the final sample after quartering amount of about 1,000 to 1,500 g. Where the fertilizer is bulky or uneven in character special attention must be made to ensure representative sampling.

PART III

PROVISIONS APPLICABLE TO SOLID FEEDING STUFFS

1. The sample shall be taken in the manner prescribed for a fertilizer in paragraphs 1 (a) or 1 (b) of Part II in this Schedule, where the feeding stuff is in the state of small lumps or meal.

2. Where the feeding stuff is in the form of cake, whether in bags or bulk.

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A number of cakes shall be selected from the different parts of the sampled portion equivalent to the number of portions taken in accordance with paragraph 1 (b) of Part II of this Schedule. The selected cakes shall be broken by a cake breaker or in some other manner so that the whole will pass through a sieve with meshes one and a quarter inch square and then shall be thoroughly mixed. From the mixture so obtained, a sample of not less than 3 kg shall be drawn in the manner described in paragraph 1 (a) of Part II of this Schedule.

3. Where the feeding stuff is in the form of feed blocks or mineral blocks. One block shall be selected irrespective of the size of the sampled portion. From this block a sample of 500 g to 1,000 g shall be taken in any manner.

4. Where the feeding stuff consists of particles grossly differing sizes before the final sample is taken any lumps shall be crushed (and for this purpose may be separated from other materials) and then the whole thoroughly remixed. From the mixture a sample of 500 g to 1,000 g shall be drawn.

5. Where a portion of the feeding stuff is unsuitable for feeding purposes. Where any appreciable portion of the feeding stuff appears to be mouldy, or is otherwise apparently unsuitable for feeding purposes, separate samples shall be drawn 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.

PART IV

DIVISION, MARKING, SEALING AND FASTENING OF SAMPLE

1. Where the sample has been taken in the prescribed manner the person taking the sample shall divide it into three parts, as nearly equal as possible, in the following manner:

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(a) *In the case of dry or powered substances:*

The samples, drawn as prescribed in the foregoing paragraphs, shall be thoroughly mixed on a sheet which will adequately protect the sample from accidental contamination and divided into three approximately equal parts. Each of these parts shall be placed in a clean dry glass or plastic bottle with a close fitting lid so that the original composition of the fertilizer or feeding stuff may be preserved. In the case of materials which undergo change or pick up moisture on exposure to air, the bottle shall have an airtight closure, Each of the said parts shall be so secured and sealed that the bottle containing it cannot be opened without breaking the seal, or alternatively the bottle containing the part of the sample cannot be removed without breaking the seal or the envelope.

(b) *In the case of substance in a liquid or semi-liquid condition.*

The sample, drawn as described in the foregoing paragraphs, shall be thoroughly mixed and at once divided into three similar and approximately equal parts by pouring successive portions into three glass or plastic bottles with airtight stoppers.

2. Each of the 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 was taken, or his representative. Each part shall be marked with the name of the material the sampling and some distinguishing reference in such a manner that the particulars so marked can be seen without breaking the seal or seals.

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