STATUTORY INSTRUMENTS

1973 No. 1521

AGRICULTURE

The Fertilisers and Feeding Stuffs Regulations 1973

Made - - -	*29th August* 1973
Laid before Parliament	*18th September* 1973
Coming into Operation	*1st January* 1974

ARRANGEMENT OF REGULATIONS

Regulation

1. Citation, commencement and interpretation.
2. Descriptions of animals for the purposes of the definition of feeding stuff.
3. Prescribed amount for the purposes of the definition of sampled portion.
4. Manner of taking, dividing, marking, sealing and fastening of samples.
5. Qualifications of agricultural analysts and deputy agricultural analysts.
6. Prescribed descriptions of material and particulars and information to be contained in the statutory statement.
7. Control of feeding stuffs containing added substances.
8. Manner of marking particulars on sales of small quantities.
9. Time by which a statutory statement relating to certain material must be given.
10. Manner of marking material.
11. Modification of section 69(1) and (2) for certain imported material.
12. Register of marks.
13. Meanings of names of material.
14. Limits of variation.
15. Methods of analysis of fertilisers and feeding stuffs.
16. Forms of certificate of analysis.
17. Methods of sending part of a sample.
18. Period within which analysis of the oil content of a feeding stuff must be carried out.

SCHEDULES

Schedule 1. Manner of taking, dividing, marking, sealing and fastening of samples.
Part I Provisions applicable to both fertilisers and feeding stuffs.
Part II Provisions applicable to solid fertilisers.
Part III Provisions applicable to solid feeding stuffs.
Part IV Division, marking, sealing and fastening of sample.

Schedule 2. Prescribed descriptions of material and particulars and information to be contained in the statutory statement.
Part I Fertilisers.
Part II Feeding stuffs.

Schedule 3. Permitted additives and provisions relating to their use and marking.

Schedule 4. Meanings of names of material.
Part I Fertilisers.
Part II Feeding stuffs.

Schedule 5. Limits of variation.
Part I Fertilisers.
Part II Feeding stuffs.

Schedule 6. Methods of analysis of fertilisers.

Schedule 7. Methods of analysis of feeding stuffs.

Schedule 8. Forms of certificate of analysis.
Part I Certificate of analysis of fertiliser.
Part II Certificate of analysis of feeding stuff.

The Minister of Agriculture, Fisheries and Food and the Secretary of State, acting jointly, in exercise of the powers conferred on them by sections 66(1), 67(5), 68(1), (2) and (3), 69(1), (3), (6) and (7), 70(1), 74(1), 74A (inserted by section 4(1) of, and paragraph 6 of Schedule 4 to, the European Communities Act 1972 (a)),75(1), 76(1), 77, 78(2), (4) and (6), 79(1), (2) and (9) and 84 of the Agriculture Act 1970(b) and of all other powers enabling them in that behalf, hereby make the following regulations after consultation with such persons or organisations as appear to them to represent the interests concerned:—

Citation, commencement and interpretation

1.—(1) These regulations may be cited as the Fertilisers and Feeding Stuffs Regulations 1973, and shall come into operation on 1st January 1974.

(2) In these regulations, unless the context otherwise requires—

"the Act" means the Agriculture Act 1970, as amended by section 4(1) of, and paragraph 6 of Schedule 4 to, the European Communities Act 1972;

"compound feeding stuff" and "feed supplement" have the meanings assigned to them respectively by paragraph 4 of Part II of Schedule 2:

AND other expressions have the same meaning as in the Act.

(a) 1972 c. 68. (b) 1970 c. 40.

(3) The Interpretation Act 1889(a) shall apply to the interpretation of these regulations as it applies to the interpretation of an Act of Parliament.

(4) Any reference in these regulations to a numbered regulation or schedule shall be construed as a reference to the regulation or schedule bearing that number in these regulations.

(5) Any reference in these regulations to a numbered section shall, unless the reference is to a section of a specified Act, be construed as a reference to the section bearing that number in the Act.

Descriptions of animals for the purposes of the definition of feeding stuff

2. The following descriptions of animals are hereby prescribed for the purposes of the definition of feeding stuff in section 66(1), that is to say, bulls, cows, steers, heifers, calves, sheep, goats, swine, horses, rabbits, mink, partridges, pheasants, poultry, bees, trout and salmon.

Prescribed amount for the purposes of the definition of sampled portion

3.—(1) The prescribed amount of material for the purposes of the definition of sampled portion in section 66(1) shall be determined in accordance with the provisions of this regulation.

(2) In relation to solid material which is packed in bags, sacks or packages, the prescribed amount shall be the quantity of material present or 5 tons, whichever is the less.

(3) In relation to solid material which is packed in bulk containers—

(*a*) if any of those containers holds not less than 5 tons of material, the prescribed amount shall be the contents of any such container;

(*b*) if all the containers together hold not less than 5 tons of material and every container holds less than 5 tons, the prescribed amount shall be the contents of the lowest number of containers which together hold not less than 5 tons;

(*c*) if all the containers together hold less than 5 tons of material or if all the material is in one container, the prescribed amount shall be the quantity of material present.

(4) In relation to solid material which is loose in heaps or bays—

(*a*) if the material is in more than one heap or bay, any of which contains not less than 5 tons of material, the prescribed amount shall be the contents of any heap or bay containing not less than 5 tons;

(*b*) if all the heaps or bays together contain not less than 5 tons of material and every heap or bay contains less than 5 tons, the prescribed amount shall be the contents of the lowest number of heaps or bays which together contain not less than 5 tons;

(*c*) if all the heaps or bays together contain less than 5 tons or if all the material is in one heap or bay, the prescribed amount shall be the quantity of material present.

(a) 1889 c. 63.

(5) In relation to liquid material in containers—

(*a*) if any of those containers holds not less than 1000 gallons of material, the prescribed amount shall be the contents of any such container;

(*b*) if all the containers together hold not less than 1000 gallons of material and every container holds less than 1000 gallons, the prescribed amount shall be the contents of the lowest number of containers which together hold not less than 1000 gallons;

(*c*) if all the containers together hold less than 1000 gallons of material, the prescribed amount shall be the quantity of material present.

Manner of taking, dividing, marking, sealing and fastening of samples

4. The manner in which samples are to be taken, divided, marked, sealed and fastened in cases where under Part IV of the Act they are taken in the prescribed manner shall be as set out in Schedule 1.

Qualifications of agricultural analysts and deputy agricultural analysts

5. The prescribed qualifications for an agricultural analyst or a deputy agricultural analyst for the purposes of section 67(5) shall be that he shall be an associate member or a fellow of the Royal Institute of Chemistry and that his practical experience of chemical analysis and microscopy, as applied to fertilisers and feeding stuffs, shall be attested by another agricultural analyst or deputy agricultural analyst appointed under section 67(3) of the Act or in accordance with section 11 of the Fertilisers and Feeding Stuffs Act 1926(**a**).

Prescribed descriptions of material and particulars and information to be contained in the statutory statement

6. The descriptions of material prescribed for the purposes of sections 68(1) and 69(1) shall be those set out in the first column of the tables in Parts I and II of Schedule 2 and the particulars or information required by the said section 68(1) to be contained in a statutory statement relating to any such material shall be the particulars or information specified in relation thereto in paragraph 1 of, and the second column of the table in, the said Part I or Part II as the case may be, subject to the provisions of the said Part I or Part II.

Control of feeding stuffs containing added substances

7.—(1) No person shall sell or have in possession with a view to sale for use as a feeding stuff or use as a feeding stuff or import into Great Britain for such use any material containing any added antioxidant, colourant, emulsifier, stabiliser, binder, vitamin D_2 or D_3, copper or urea or any added substance of a description specified in the first column of Part V of the table in Schedule 3 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 any antioxidant, colourant, emulsifier, stabiliser, binder, vitamin D_2 or D_3, copper or urea or substance as aforesaid which is—

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

(**a**) 1926 c. 45.

(*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 available for sale, purchase or use in a feeding stuff;

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

In this regulation the expressions "a medicinal product" and "a medicinal purpose" have the meanings assigned to them by section 130(1) and (2) respectively of the Medicines Act 1968**(a)**.

(3) No person shall use as a feeding stuff or import into Great Britain for such use any material containing any added substance, not being a substance of a name or description specified in the table in Schedule 3 or in paragraph 2(*f*) or (*g*) of that Schedule, which is deleterious to animals of any description prescribed in regulation 2 or to human beings, and it shall be an offence if a sampled portion of any such material is shown by an analysis of the sample taken from it to contain an added substance which is deleterious as aforesaid.

(4) In relation to any material to which this regulation or Schedule 3 applies the operation of the provisions of sections 66(2), 73(1), 80(2) and 82 shall be modified as follows:—

(*a*) Section 66(2) shall have effect as if—

(i) the words "imported or" were inserted immediately before the word "sold" in both places where that word appears, and

(ii) the words "or as so used" were inserted immediately after the words "feeding stuff", and

(iii) the words "or is so used" were inserted immediately after the words "to be so used".

(*b*) Section 73(1) shall have effect as if there were added at the end of that subsection the words "or to human beings".

(*c*) Sections 80(2) and 82 shall apply in relation to proceedings for an offence under this regulation and section 74A(3) as they apply respectively to proceedings for an offence under any of the provisions mentioned in them.

Manner of marking particulars on sales of small quantities

8. The label of a parcel to which paragraph (*b*) of subsection (2) of section 68 relates shall bear, in block capital letters and figures of not less than half an inch in height, the particulars which would, apart from that subsection, be required to be contained in a statutory statement on the sale of the material.

Time by which a statutory statement relating to certain material must be given

9. For the purposes of section 68(3), any statutory statement required to be given on the sale of—

(*a*) any feeding stuff (not being a compound feeding stuff or a feed supplement) or

(*b*) any fertiliser of a description specified in Group G in the first column of the table in Part I of Schedule 2, or

(a) 1968 c. 67.

(*c*) any solid fertiliser, not packed in bags, sacks or packages, of a description specified in any of Groups A to F and H in the first column of the table in Part I of Schedule 2, or

(*d*) any liquid or semi-liquid fertiliser in a container of a capacity in excess of 40 gallons

may be given as soon as practicable after delivery of the material to the purchaser.

Manner of marking material

10. Material required by section 69(1) to be marked shall be marked legibly in writing, printing or stencilling or in any other appropriate manner—

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

(*b*) where the material is packed in a single package, on the wrapper or container of, or on a label securely attached to, the package, or

(*c*) where the material is packed in a number of separate packages, on the wrapper or container of, or on a label securely attached to, each of the packages, or

(*d*) where the material is packed in a number of packages which are themselves enclosed in a larger package or packages, on the wrapper or container of, or on a label securely attached to—

(i) each of the packages, or

(ii) the larger package, or

(iii) each of the larger packages, or

(*e*) where the material is in a bulk container or tanker—

(i) on the bulk container or tanker, or on 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 which 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

(*f*) where the material is loose in heaps or bays in such a manner that the mark shall be readily apparent and unequivocally associated with the material.

Modification of section 69(1) *and* (2) *for certain imported material*

11. In the case of—

(*a*) any feeding stuff (not being a compound feeding stuff or a feed supplement) or

(*b*) any fertiliser of a description specified in Group G in the first column of the table in Part I of Schedule 2, or

(*c*) any solid fertiliser, not packed in bags, sacks or packages, of a description specified in any of Groups A to F and H in the first column of the table in Part I of Schedule 2, or

(*d*) any liquid or semi-liquid fertiliser in a container of a capacity in excess of 40 gallons

which has been imported and is of a description prescribed for the purposes of section 69(1) by regulation 6, subsections (1) and (2) of section 69 shall have effect as if—

(i) the words "and in either case before it is removed from the premises" were omitted from the said subsection (1), and

(ii) the words "any material which has been marked in accordance with this subsection" were substituted for the words "the material" in the said subsection (1).

Register of marks

12.—(1) As respects any material of a description prescribed for the purposes of section 69(1) by regulation 6 which comprises—

(*a*) any feeding stuff (not being a compound feeding stuff or a feed supplement) or

(*b*) any fertiliser of a description specified in Group G in the first column of the table in Part I of Schedule 2, or

(*c*) any solid fertiliser, not packed in bags, sacks or packages, of a description specified in any of Groups A to F and H in the first column of the table in Part I of Schedule 2, or

(*d*) any liquid or semi-liquid fertiliser in a container of a capacity in excess of 40 gallons, or

(*e*) any material, not being of a standard formulation on general sale by the seller concerned, which is specially manufactured or mixed to the order of a particular purchaser,

the matters required by section 69 to be marked on that material may be denoted by a mark whose meaning can be ascertained by reference to a register kept in accordance with this regulation.

(2) The register shall show those matters to which the mark relates, being matters required to be contained in a statutory statement relating to the material to which the mark relates, and the date of entry of those particulars in the register, and entries relating to material of a kind mentioned in paragraph (1)(*e*) of this regulation shall include the name and address of the purchaser, the date of the order and the amount ordered. The register shall be kept as a separate record in book form marked on the outside "Register of marks under section 69(6) of the Agriculture Act 1970" and shall be kept on the premises where the material is held for the purpose of selling it in the course of trade for use as a fertiliser or feeding stuff, save that if the material is in a public store the register shall be kept on the premises of the person who has the material for sale.

(3) As respects any material of a description prescribed for the purposes of section 69(1) by regulation 6 other than material to which paragraph (1) of this regulation applies, the foregoing provisions of this regulation shall apply to any such material which a person has on his premises after the commencement of these regulations for the purpose of selling it in the course of trade for use as a fertiliser or feeding stuff and which is removed from his premises not later than 1st September 1974 as they apply to material to which the said paragraph (1) applies.

(4) The period for which the register is to be preserved in accordance with section 69(7) shall be a period of six months commencing with the first day on which none of the materials referred to in the register remains on the premises for sale as aforesaid.

Meanings of names of material

13. For the purposes of section 70, any name of a material set out in the first column of Schedule 4 shall have the meaning assigned thereto in the second column of that Schedule, subject to the provisions of that Schedule.

Limits of variation

14. For the purposes of section 74, the limits of variation in relation to any mis-statement as to the nature, substance or quality of any material mentioned in the first column of Schedule 5 shall be as set out in relation to that material in the second column of that Schedule.

Methods of analysis of fertilisers and feeding stuffs

15. The methods by which analyses of fertilisers and feeding stuffs shall be made for the purposes of the Act shall be as set out in Schedules 6 and 7 respectively.

Forms of certificate of analysis

16. The certificates of an agricultural analyst of the analysis of a fertiliser and of a feeding stuff shall be in the forms set out in Part I and Part II of Schedule 8 respectively.

Methods of sending part of a sample

17. Any part of a sample required to be sent to any person in pursuance of subsection (1)(*b*) or (2) of section 77 shall be sent by registered post or by the recorded delivery service.

Period within which analysis of the oil content of a feeding stuff must be carried out

18. 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 of that feeding stuff shall be disregarded unless it is carried out before the end of 3 weeks commencing with the date of sampling.

In Witness whereof the Official Seal of the Minister of Agriculture, Fisheries and Food is hereunto affixed on 17th August 1973.

(L.S.)

Joseph Godber,
Minister of Agriculture, Fisheries and Food.

29th August 1973.

Gordon Campbell,
Secretary of State for Scotland.

SCHEDULE 1

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

(Sections 66(1), 73(1), 75(1), 76(1), 77(1), (2) & (3) and 78(2) & (4) and 79(1) & (2) and Regulation 4)

PART I

PROVISIONS APPLICABLE TO BOTH FERTILISERS AND FEEDING STUFFS

A. *General provisions*

1. In the case of material in packages, bottles, drums or kegs, only unopened containers which appear to the inspector proposing to take the sample to be the original containers 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 bears the appearance of having received damage.

3. An inspector who proposes to take a sample under section 76 on premises (not being premises used only as a dwelling) on which he has reasonable cause to believe that there is any fertiliser or feeding stuff which the occupier of the premises has purchased, not for the purpose of re-sale in the course of trade but for the purpose of use as a fertiliser or feeding stuff, shall satisfy himself that the conditions in which the material is stored are not such as might cause deterioration of the material and that the material appears not to have been contaminated by any other material.

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

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

5. It shall be assumed that the sampled portion is composed of separate approximately equal parts and that the number of such parts is equivalent to—

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

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

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

6. Where material in packages which an inspector has reasonable cause to believe has been purchased, not for the purpose of resale in the course of trade but for the purpose of use as a fertiliser or feeding stuff, as the case may be, has been delivered to the purchaser and is to be sampled but some part 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 the taking of a sample, on the ground that the material is unsuitable.

C. *Provisions applicable where the fertiliser or feeding stuff is in a liquid or semi-liquid condition*

8. (*a*) *In bottles or containers each containing not 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 fertilisers in paragraph 1(*a*) of Part II of this Schedule. The entire contents of the selected bottles or containers shall be emptied into a clean dry vessel of glass or other suitable material and well mixed by

stirring or shaking. From this mixture a sample of between about one quart and about half a gallon shall be drawn, the mixture being stirred or shaken immediately before the sample is drawn.

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

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

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

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

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

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

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

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

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

TABLE

Quantities of liquid or semi-liquid fertilisers and feeding stuffs to be withdrawn in accordance with sub-paragraph (*c*)(i) *or* (iv) *above*

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

PART II

PROVISIONS APPLICABLE TO SOLID FERTILISERS

1. *Where the fertiliser is in a state of fine division*

(*a*) *In packages*

Where the fertiliser is in packages, a number of packages shall be selected in accordance with the following table:—

	Number of packages to be selected for sampling
Where the sampled portion consists of one package	1
Where the sampled portion consists of two packages	2
Where the sampled portion consists of three packages	3
Where the sampled portion consists of more than three packages but not more than 20 packages	4
Where the sampled portion consists of more than 20 packages but not more than 60 packages	6
Where the sampled portion consists of more than 60 packages but not more than 100 packages	8
Where the sampled portion consists of more than 100 packages but not more than 400 packages	10
Where the sampled portion consists of more than 400 packages	20

When the number of packages has been selected in accordance with this sub-paragraph either—

(i) the selected packages shall be emptied separately on a clean dry surface and worked up with a shovel and one shovelful taken from each and the shovelfuls so taken shall then be thoroughly mixed together and any lumps broken up, or

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

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 the quartering and rejection until the remainder is from about 2 lb. to 4 lb. in weight. Alternatively the reduction of the gross sample by the quartering method may be effected by the use of a mechanical quartering device known as a sample divider or riffle.

(*b*) *In bulk*

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

	Portions
Where the sampled portion does not exceed 2 cwt.	not less than 1 per ½ cwt. or part thereof
Where the sampled portion exceeds 2 cwt. and does not exceed 1 ton	not less than 6
Where the sampled portion exceeds 1 ton and does not exceed 3 tons	not less than 10
Where the sampled portion exceeds 3 tons and does not exceed 5 tons	not less than 12
Where the sampled portion exceeds 5 tons and does not exceed 25 tons	not less than 20
Where the quantity exceeds 25 tons	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 also described in that paragraph.

2. *Where the fertiliser is in a coarse or lump condition (as in the case of burnt lime not ground)*

(*a*) *In packages*

The packages, selected according to the appropriate scale in paragraph 1(*a*), shall be emptied separately on a clean dry surface and worked up with a shovel and one shovelful taken from each. The shovelfuls so taken shall be crushed immediately and the whole passed through a sieve with meshes one and a quarter inch square. It shall be mixed thoroughly and rapidly and a sample of about 4 lb. to 6 lb. in weight drawn in the manner described in paragraph 1(*a*).

(*b*) *In bulk*

Shovelfuls shall be taken according to the appropriate scale in paragraph 1(*b*). The shovelfuls so taken shall be treated, and a sample shall be drawn, in the manner described in paragraph 1 (a).

3. *Where the fertiliser consists of bulky material, uneven in character and likely to get matted together*

(*a*) *In packages*

The packages, selected according to the appropriate scale in paragraph 1(*a*), shall be emptied separately on a clean dry surface and the matted portions torn up.

One shovelful shall be taken from each and the shovelfuls so taken shall be thoroughly mixed together. The sample shall be drawn from the mixture and shall be from about 2 lb. to 4 lb. in weight. If the material separates into a fibrous part and a powdery part, the sample drawn shall consist of these two parts in approximately their relative proportions as they exist in the material.

(*b*) *In bulk*

Shovelfuls shall be taken according to the appropriate scale prescribed in paragraph 1(*b*). The shovelfuls thus taken shall be treated, and a sample shall be drawn, in the manner described in paragraph 3(*a*).

4. When the fertiliser consists of materials such as burnt lime or slaked lime (calcium hydroxide) which are liable to undergo change on exposure to air and moisture or when the fertiliser consists of materials such as calcium nitrate or ammonium nitrate, which are liable to absorb moisture, or when the material is sulphate of ammonia, the sampling shall be carried out rapidly in a dry place and the sample divided into parts and packed immediately.

5. When stones are naturally present in a fertiliser, they shall, if possible, be broken up and mixed with the quantity from which a sample is to be drawn. If they cannot be broken up they shall be removed from the mixture from which a sample is to be drawn and the weight of the residue of that mixture and the weight of the stones shall be ascertained and reported to the analyst.

PART III

PROVISIONS APPLICABLE TO SOLID FEEDING STUFFS

1. *Where the feeding stuff is in the state of small lumps or meal*

The sample shall be taken in the manner prescribed for a fertiliser in paragraphs 1(*a*) or 1(*b*) of Part II of this Schedule.

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

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 cakebreaker 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 6lb. in weight 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 2 lb. to 4 lb. shall be taken in any manner.

4. *Where the feeding stuff consists of particles of grossly differing sizes*

(*a*) *In packages*

The packages shall be selected according to the appropriate scale in paragraph 1(*a*) of Part II of this Schedule. The selected packages shall be emptied separately on a clean surface, worked up with a shovel and one shovelful from each set aside. The shovelfuls so set aside shall then be thoroughly mixed together and reduced if necessary by the cone and quartering method described in paragraph 1(*a*) of Part II of this Schedule to a quantity of not less than 15 lb. Any lumps in the said quantity shall be crushed (and for this purpose may be separated from other material) and the whole then thoroughly remixed. From the mixture a sample of 2 lb. to 4 lb. weight shall be drawn.

(*b*) *In bulk*

Shovelfuls shall be taken according to the appropriate scale in paragraph 1(*b*) of Part II of this Schedule. The shovelfuls thus taken shall be treated, and a sample drawn, in the manner described in paragraph (*a*) above.

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, or, in the circumstances set out in section 77(2), four parts, as nearly as possible equal, in the following manner:—

(*a*) *In the case of dry or powdered substances*

The samples, drawn as described in the foregoing paragraphs, shall be thoroughly mixed on a floor covering which will adequately protect the sample from accidental contamination, and divided into three or, as the case may be, four similar and approximately equal parts. Each of these parts shall be placed in a clean dry bottle or jar with a close-fitting stopper or lid or (except in the case of a fertiliser) a clean dry tin with a close-fitting lid (such as a lever lid), so that the original composition of the fertiliser or feeding stuff may be preserved. In the case of burnt lime, slaked lime (calcium hydroxide), calcium nitrate, ammonium sulphate and other substances likely to undergo change if not kept in an air-tight receptacle, the bottle or jar used shall have a ground-in or rubber stopper or a metal cap with inner pad or a closure of the kind used on preserving jars. Each of the said parts shall be so secured and sealed that the bottle, jar or tin containing it cannot be opened without breaking the seal; or alternatively, the bottle, jar or tin containing the part may be placed in a stout envelope or in a linen, cotton or plastic bag and the envelope or bag then secured and sealed in such a manner that the part of the sample cannot be removed without breaking the seal or the envelope or the bag.

(*b*) *In the case of substances 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 similar and approximately equal parts by pouring successive portions into each of three or, as the case may be, four clear glass bottles or jars, preferably with wide mouths. The bottles or jars used shall be provided with air-tight stoppers or with lids which shall be so fastened that spillage or evaporation of the contents is prevented.

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

SCHEDULE 2

PRESCRIBED DESCRIPTIONS OF MATERIAL AND PARTICULARS AND INFORMATION TO BE CONTAINED IN THE STATUTORY STATEMENT

(Sections 68(1) and 69(1) and Regulation 6)

PART I

FERTILISERS

1. In the case of material of any description specified in the first column of the table in this Part of this Schedule, the statutory statement shall contain the particulars specified in relation to that material in the second column thereof and also the name of any pesticide or herbicide or of any of the substances boron, cobalt, copper, iron, magnesium, manganese and molybdenum, not being such a substance which has been added with or without other substances in order to improve the handling qualities of the material, which has been added as an ingredient in the course of manufacture or preparation for sale. When any boron, cobalt, copper, iron, magnesium, manganese or molybdenum has been so added, there shall be stated the total amount thereof present expressed as a percentage by weight unless the amount present is less than 0·1 per cent. by weight in which case it shall be expressed in parts per million.

2. The provisions of this Part of this Schedule shall apply to material of any description specified therein under whatever name it may be sold or offered for sale and notwithstanding that it contains a substance not mentioned in this Part of this Schedule.

3. In the said particulars—

(*a*) the amount shall in each case be expressed as a definite percentage of the weight of the material and not as a range of percentages;

(*b*) neutralising value shall be expressed in terms of calcium oxide (Ca0);

(*c*) nitrogen shall be expressed in terms of nitrogen (N);

(*d*) phosphoric acid, soluble phosphoric acid and insoluble phosphoric acid shall be expressed in terms of phosphoric anhydride ($P_2 0_5$);

(*e*) potash shall be expressed in terms of potassium oxide ($K_2 0$).

4. In this Part of this Schedule—

"compound fertiliser" means a product obtained by mixing two or more materials including at least one of the materials mentioned in the first column of the table in this Part of this Schedule but excluding materials used for improving soil structure or as growing media and which contain less than 1 % each of nitrogen, total phosphoric acid and potash: for the purposes of this definition the presence of any of the substances boron, cobalt, copper, iron, magnesium, manganese or molybdenum or any substance added to improve the handling qualities of the product shall be disregarded.

"herbicide" means a substance calculated to destroy or control any unwanted plant.

"pesticide" means a substance calculated to destroy or control any insect, mite, mollusc, nematode, fungus or any other pest capable of destroying, damaging or retarding the growth of any form of plant life.

TABLE

	Description of material	*Particulars to be contained in statutory statement*
Group A	Compound fertiliser	Amounts, if any, of nitrogen, potash, phosphoric acid soluble in water, and phosphoric acid insoluble in water respectively.
Group B	Ammonium nitrate and mixtures of ammonium nitrate with any material mentioned in Group H of this table	Amount of nitrogen.
	Ammonium sulphate nitrate	Amount of nitrogen.
	Calcium cyanamide	Amount of nitrogen.
	Nitrate of lime	Amount of nitrogen.
	Nitrate of soda	Amount of nitrogen.
	Nitrogenous gas liquor; ammoniacal gas liquor; gas liquor.	Amount of nitrogen.
	Sulphate of ammonia	Amount of nitrogen.
	Urea	Amount of nitrogen.
Group C	Basic slag	Total amount of phosphoric acid. Amount of phosphoric acid soluble in 2% citric acid. Amount of the material that will pass through a 0·5 mm sieve.
	Dicalcium phosphate	Amount of phosphoric acid soluble in citric acid.
	Phosphate rock, ground or otherwise.	Amount of phosphoric acid. Amount of the material that will pass through a British Standard Test Sieve Mesh No. 100.
	Superphosphate	Amount of phosphoric acid soluble in water.
	Superphosphate, concentrated.	Amount of phosphoric acid, soluble in water.
	Superphosphate, triple.	Amount of phosphoric acid soluble in water.
Group D	Potassium salts not otherwise mentioned in this table used as fertilisers, including kainit, sylvinite, potash manure salt, muriate of potash, sulphate of potash and sulphate of potash-magnesia.	Amount of potash.
Group E	Nitrate of potash	Amounts of nitrogen and potash respectively.
	Potassic nitrate of soda	Amounts of nitrogen and potash respectively.
Group F	Potassic basic slag	Total amount of phosphoric acid. Amount of phosphoric acid soluble in 2% citric acid. Amount of potash. Amount of slag which will pass through a 0·5 mm sieve.
Group G	Bone meal, or other product not otherwise mentioned in this table, obtained by grinding or otherwise treating bone, used for fertilising purposes.	Amounts of nitrogen and phosphoric acid respectively.

	Description of material	*Particulars to be contained in statutory statement*
	Bone phosphate, precipitated; dicalcium bone phosphate.	Amount of phosphoric acid soluble in citric acid.
	Dissolved or vitriolised bone	Amounts of nitrogen, phosphoric acid soluble in water, and phosphoric acid insoluble in water respectively.
	Dried blood for fertilising purposes	Amount of nitrogen.
	Fish residues or other product, obtained by drying and grinding or otherwise treating fish or fish waste, used for fertilising purposes.	Amounts of nitrogen and phosphoric acid respectively.
	Guano, including Peruvian and other raw guanos, but excluding poultry manure.	Amounts of nitrogen, phosphoric acid and potash respectively.
	Hoofs	Amount of nitrogen.
	Hoofs and horns	Amount of nitrogen.
	Horns	Amount of nitrogen.
	Meat and bone residues, or any product not specifically mentioned elsewhere in this table, obtained by drying and grinding or otherwise treating bone, flesh, flesh fibre and other slaughterhouse residues, used for fertilising purposes.	Amounts of nitrogen and phosphoric acid respectively.
	Oil seed fertilisers, including castor meal, rape meal, or any residue other than mowrah meal, which is obtained by the removal of oil from seeds.	Amount of nitrogen.
Group H	Burnt or quick lime, ground or otherwise.	Neutralising value.
	Burnt magnesian lime, ground or otherwise.	Neutralising value.
	Calcium hydroxide; hydrated lime; slaked lime; slaked magnesian lime.	Neutralising value.
	Chalk, ground.	Neutralising value.
	Chalk, screened.	Neutralising value. Amount of the material that will pass through a declared British Standard Test Sieve.
	Limestone, ground; magnesian limestone, ground.	Neutralising value. Amount of the material that will pass through a British Standard Test Seive Mesh No. 100.
	Mixed lime	Neutralising value.

PART II

FEEDING STUFFS

1. In the case of material of any description specified in the first column of the table in this Part 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 amount present (whether naturally present or added) of any copper (if present in excess of 50 parts per million) or magnesium (if present in excess of 0·5 per cent.);

(*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 molybdenum or selenium, a statement of the total amount of molybdenum or selenium present (whether naturally present or added);

any amount referred to—

(i) in sub-paragraph (*a*) above being 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);

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

(iii) in sub-paragraph (*d*) above being expressed in parts per million.

2. The provisions of this Part of this Schedule shall apply to material of any description specified therein under whatever name it may be sold or offered for sale and notwithstanding that it contains a substance not mentioned in this Part of this Schedule.

3. In the said particulars—

(*a*) the amount shall in each case be expressed as a definite percentage of the weight of the material, and not as a range of percentages;

(*b*) phosphoric acid shall be expressed in terms of phosphoric anhydride (P_2O_5).

4. In this Part of this Schedule and, as respects the definitions of "compound feeding stuff" and "feed supplement", in these regulations—

"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 nitrogen but not including ammoniacal or nitrate nitrogen, multiplied by 6·25;

"amount of protein equivalent of urea" means the amount of urea nitrogen multiplied by 6·25;

"compound feeding stuff" 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 the table in this Part of this Schedule; for the purposes of this definition the presence of any added substance of a kind referred to in regulation 7 shall be disregarded;

"feed supplement" means a product obtained by mixing two or more materials, being a product of a 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 Schedule 7;

"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 3·222 of Schedule 7;

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

TABLE

Description of material	*Particulars to be contained in statutory statement*
Compound feeding stuff	Amount, if any, of protein (stating as being included therein the amount, if any, of protein equivalent of urea) and amounts, if any, of oil and fibre respectively.
Alfalfa (lucerne) meal	Amounts of protein and fibre respectively.
Artificially dried grass, clover, lucerne, sainfoin, green cereals or any other artificially dried green crops or a mixture of any of them.	Amount of protein.
Clover meal	Amounts of protein and fibre respectively.
Coconut or copra cake or meal	Amounts of oil and protein respectively.
Cotton cakes or meals, not decorticated.	Amounts of oil and protein respectively.
Cotton cakes or meals from decorticated or partly decorticated cotton seed	Amounts of oil, protein and fibre respectively.
Dried brewery grains	Amounts of oil and protein respectively.
Dried distillery by-products (other than malt culms and dried yeast)	Amounts of oil and protein, of fibre if present in excess of 2% and of calcium if present in excess of 2%.
Dried plain beet pulp	Amount of fibre.
Dried molassed beet pulp	Amounts of sugar and fibre respectively.
Dried yeast	Amount of protein.
Feed supplement	Instructions for mixing with other feeding stuffs or information as to use where the supplement is fed direct to animals. Protein equivalent of urea, if any.
Feeding bone flour	Amounts of phosphoric acid and protein respectively.
Feeding bone meal, ground bone or any other bone product for feeding purposes.	Amounts of phosphoric acid and protein respectively.
Feeding dried blood	Amount of protein.
Feeding meat and bone meal, or any other product of meat and bone for feeding purposes.	Amounts of oil, protein and phosphoric acid respectively.
Feeding meat meal, or any other product of meat for feeding purposes.	Amounts of oil, protein and phosphoric acid respectively.
Fish meal, white fish meal, or other product obtained by drying and grinding or otherwise treating fish or fish waste.	Amounts of oil, protein, phosphoric acid and salt respectively.
Linseed cakes and the meals of such cakes; extracted linseed meal.	Amounts of oil and protein respectively.
Linseed meal	Amount of oil.
Maize by-products not otherwise mentioned in this table	Amounts of oil, protein and fibre respectively.
Maize, flaked.	Amounts of oil and protein respectively.
Maize germ cake or meal	Amounts of oil and protein respectively.
Maize gluten feed	Amounts of oil and protein respectively.
Milk powders, including oil and/or fat fortified milk powders.	Amounts of oil and protein respectively.
Mixtures of molasses and urea	Sugar and protein equivalent of urea.

Description of material	*Particulars to be contained in statutory statement*
Molasses feeds (other than dried molassed beet pulp and mixtures of molasses and urea) including any feeding stuffs, composed of treacle or molasses with an absorbent, containing not less than 10% of sugar.	Amounts of sugar and fibre respectively.
Oatmeal by-products	Amount of fibre.
Oil cakes or meals, not otherwise mentioned in this table, which are the product of any one undecorticated substance or seed from which oil has been removed.	Amounts of oil and protein respectively.
Oil cakes or meals, not otherwise mentioned in this table, which are the product of any one decorticated or partly decorticated substance or seed from which oil has been removed.	Amounts of oil, protein and fibre respectively.
Palm kernel cake or meal	Amounts of oil and protein respectively.
Rape cake or meal	Amounts of oil and protein respectively.
Rice bran or rice meal, or the by-product produced in milling shelled rice.	Amounts of oil, protein and fibre respectively.
Soya cake or meal	Amounts of oil and protein repectively.
Treacle or molasses	Amount of sugar.
Malt culms	Amounts of protein and fibre respectively.
Wheat offals or millers' offals	Amount of fibre.
Barley meal, barley meal Grade II, bean meal, dari or durra meal, ground oats, Indian or maize meal, locust bean meal, pea meal, wheat meal.	None.

SCHEDULE 3

PERMITTED ADDITIVES AND PROVISIONS RELATING TO THEIR USE AND MARKING

(Section 74A and Regulation 7)

1. In this Schedule, in Schedule 2 Part II and in regulation 7—

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

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

"colourant" means any substance, other than a basic feed ingredient, which is added to a feeding stuff only to impart colour to the feeding stuff or to an animal product, but does not include a substance which is added to material only for the purpose of rendering that material fit only for animal feeding;

"daily ration" means the total quantity of feeding stuffs, expressed on a 12 per cent. moisture basis, necessary on average per day 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;

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

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

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

(*a*) any added antioxidant other than an antioxidant of a name and description specified in Part I of the table below nor any added antioxidant of a name and description so specified in proportions which, taking account of any such antioxidant which is naturally 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 below nor in the case of material intended for use as a feeding stuff for poultry any added colourant of a name specified in Chapter A of the said Part II in proportions which, taking account of any such colourant which is naturally present, exceed 80 parts per million in whole feeding stuffs 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 below;

(*d*) any added vitamin D_2 or vitamin D_3, save that material intended for use as a feeding stuff for any animal of a kind specified in the second column of—

(i) Chapter A of Part IV of the table below may contain vitamin D_2 or D_3 (but not both added vitamin D_2 and vitamin D_3) 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 the kind of animal;

(ii) Chapter B of the said Part IV may contain vitamin D_3 in proportions which, taking account of any vitamin D_3 which is naturally present, do not exceed those specified 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 below 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 (Cu) save that material intended for use as a feeding stuff—

(i) for pigs may contain copper (whether naturally present or added) in proportions not exceeding 100 parts per million in whole feeding stuffs;

(ii) for any kind of animal other than pigs or sheep may contain copper (whether naturally present or added) in proportions not exceeding 50 parts per million in whole feeding stuffs;

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

3. If the material is intended for mixing with other materials before use as a feeding stuff and it contains an added substance mentioned in Part I, II, IV or V of the table below or added copper in proportions which, taking account of any such substance or copper which is naturally present, do not exceed in each case 5 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 Great Britain or sold or possessed with a view to sale for use as a feeding stuff if it is marked in a manner specified in regulation 10 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 there may be substituted for the words "grammes per kilogramme", the symbol "%" "lb. per cwt." or "lb. per ton".

4. If the material, not being material to which paragraph 5 of this Schedule applies, contains an added substance mentioned in Part I, II, IV or V of the table below or added copper in proportions which, taking account of any such substance or copper

which is naturally present, exceed in each case 5 times the maximum permitted content specified in relation to the substance in paragraph 2 above or in the table below, that material may be imported into Great Britain, sold or possessed with a view to sale for use as a feeding stuff if it is marked in a manner specified in regulation 10 with the statement required by paragraph 3 above in respect of material to which that paragraph applies and with a statement to the effect that the material is not for direct feeding to animals and that accurate mixing is essential.

5.—(1) If the nature of the material intended for use as a feeding stuff or the means by which it is to be fed to the animal is such that the animal can consume no more of the material than is represented by a proportion of its normal daily consumption of feeding stuffs—

(*a*) each statement of maximum content in paragraph 2(*a*), (*b*) and (*f*) of this Schedule and Parts IV and V of the table below may be read as if that content had been divided by that proportion;

(*b*) the material shall be marked in a manner specified in regulation 10 with instructions as to the kind and age group of animals for which it is intended.

(2) If material to which sub-paragraph (1) of this paragraph applies is also suitable for mixing with other materials before use as a feeding stuff, sub-paragraph (1)(*b*) above shall not apply to that material but that material may be imported into Great Britain, sold or possessed with a view to sale for use as a feeding stuff if it is marked in a manner specified in regulation 10 with the statement required by paragraph 3 of this Schedule in respect of material to which that paragraph applies and with a statement to the effect that accurate mixing is essential.

6. If material containing any added iron, iodine, cobalt, manganese, 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 68 or 69, containing the name and a statement of the total amount present (whether naturally present or added) of any iron, iodine, cobalt, manganese, zinc, vitamins (other than vitamins A, D or E) or pro-vitamins respectively, it shall be marked in a manner specified in regulation 10.

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-*tert* butyl 4-hydroxyanisole
BHT	2,6-di(*tert* butyl)-4-methylphenol
Ethoxyquin	6-ethoxy-1,2-dihydro-2,2,4-trimethyl-quinoline

PART II

PERMITTED COLOURANTS

Chapter A

Capsanthen
Lycopen
Beta-8′-apo-carotenal
Ethyl ester of beta-8′-apo-carotenoic acid
Lutein
Cryptoxanthin
Violaxanthin
Canthaxanthin
Zeaxanthin

Chapter B

Patent Blue V
Curcumin
Amaranth
Tartrazine
Orange G
Green S
Indigo carmine
Brilliant Black (Black PN)
Carmoisine
Ponceau 4R
Sunset Yellow FCF
Brown FK
Red 6B

PART III

PERMITTED EMULSIFIERS, STABILISERS AND BINDERS

NAME OR DESCRIPTION

Lecithins
Alginic acid
Sodium alginate
Potassium alginate
Ammonium alginate
Calcium alginate
1, 2-dihydroxypropyl alginate
Agar-agar
Carrageen
Carob seed flour
Tamarind seed flour
Guar seed flour, guar gum
Gum tragacanth
Gum arabic
Sorbitol
Mannitol
Glycerol
Pectins
Methylcellulose
Carboxymethylcellulose
Hydroxypropylmethylcellulose
Ethylmethylcellulose
Sodium, potassium or calcium salts of food fatty acids derived either from edible oils and fats, or from distilled food fatty acids
Mono- and di-glycerides of food fatty acids

NAME OR DESCRIPTION—(*cont.*)

Mono-and di-glycerides of food fatty acids esterified with the following acids:

(*a*) acetic
(*b*) lactic
(*c*) citric
(*d*) tartaric
(*e*) mono-acetyltartaric and di-acetyltartaric

Sucrose esters: esters of sucrose and of food fatty acids
Sucro glycerides: esters of sucrose and of mono- and di-glycerides of food fatty acids
Polyglycerol esters of the non-polymerized fatty acids
Propylene glycol (1,2-propane diol) esters of the food fatty acids
Sodium stearoyl-2-lactylate
Calcium stearoyl-2-lactylate
Lignosulphonates
Kaolin
Bentonite and other montmorillonite clays
Silica and silicates
Gelatine
Sodium hexametaphosphate
Sorbitan esters
Polyoxyethylene sorbitan esters
Disodium ethylenediamine tetra acetate
Vermiculite
Esters of polyethylene glycol

PART IV
VITAMINS D

Vitamin	Kind of animal	Maximum content (international units per kilogramme in whole feeding stuff)
	Chapter A	
	Pigs	2,000
	Piglets	10,000 in milk replacer feeds only.
Vitamin D_2	Cattle	4,000
or	Calves	10,000 in milk replacer feeds only.
	Sheep	4,000
Vitamin D_3	Lambs	10,000 in milk replacer feeds only.
	Horses	4,000
	Other kinds except poultry	2,000
	Chapter B	
Vitamin D_3	Laying hens	3,000
	Poultry other than laying hens	2,000

In this Part of this table "milk replacer feed" means a manufactured feed used as a substitute for natural milk.

PART V

TRACE ELEMENTS

Element	Maximum content (parts per million in whole feeding stuff)
Iron-Fe	1,250
Iodine-I	40
Cobalt-Co	10
Manganese-Mn	250
Zinc-Zn	250
Molybdenum-Mo	2·5
Selenium-Se	0·5

SCHEDULE 4

MEANINGS OF NAMES OF MATERIAL

(Section 70(1) and Regulation 13)

PART I

FERTILISERS

Name of Material	*Meaning*
Ammonium nitrate	Ammonium nitrate for fertilising purposes.
Ammonium sulphate nitrate	A mixture of, or combination of, ammonium sulphate and ammonium nitrate in which the nitrate nitrogen content is not less than one-fifth of the total nitrogen present.
Calcium cyanamide	Commercial calcium cyanamide.
Nitrate of lime	Calcium nitrate for fertilising purposes.
Nitrate of soda	Sodium nitrate for fertilising purposes.
Nitrogenous gas liquor; ammoniacal gas liquor; gas liquor.	Ammoniacal liquor produced in the carbonisation of coal and free from tar visible to the naked eye, containing less than 0·4% thiocyanate as CNS.
Sulphate of ammonia ...	Ammonium sulphate for fertilising purposes.
Urea	Commercially pure urea containing not more than 1·5% biuret.
Basic slag	A by-product, containing phosphorus, obtained in the manufacture of steel and to which no addition has been made at the time of leaving or after it has left the furnace.
Dicalcium phosphate ...	Dicalcium phosphate for fertilising purposes.
Phosphate rock, ground or otherwise.	The substance obtained from mineral calcium phosphate deposits, to which no other matter has been added.
Superphosphate	Phosphate rock which has been treated with sulphuric acid.
Superphosphate, concentrated.	Phosphate rock which has been treated with sulphuric acid and phosphoric acid.

Name of Material	*Meaning*
Superphosphate, triple. ...	Phosphate rock which has been treated with phosphoric acid only.
Kainit	A mineral potassium salt with or without magnesium.
Magnesium kainit	Mineral potassium salt containing at least 3·6% of magnesium (Mg).
Muriate of potash	Potassium chloride for fertilising purposes.
Sulphate of potash	Potassium sulphate for fertilising purposes.
Nitrate of potash	Potassium nitrate for fertilising purposes.
Potassic nitrate of soda; Chilean potash nitrate.	A mixture of sodium nitrate and potassium nitrate for fertilising purposes.
Potassic basic slag	A mixture of basic slag and muriate or sulphate of potash.
Bone meal	Commercially pure bone, raw or degreased, which has been ground or crushed, of which not less than 90% will pass through a sieve of ¼" square apertures.
Dissolved or vitriolised bone...	Commercially pure bone which has been treated with sulphuric acid.
Precipitated bone phosphate; dicalcium bone phosphate.	An insoluble calcium phosphate prepared by treating commercially pure bone with acid and precipitation of phosphate from the solution.
Steamed bone flour	Commercially pure bone, degreased and ground or crushed, from which the nitrogen has been partly or wholly removed by steam, of which not less than 75% will pass through a British Standard Test Sieve Mesh No. 16.
Steamed bone meal	Commercially pure bone, degreased and ground or crushed, from which the nitrogen has been partly or wholly removed by steam, of which not less than 90% will pass through a sieve of ¼" square apertures.
Castor meal	The residue which is obtained by the removal of oil from commercially pure castor seed.
Dried blood	Blood which has been dried, to which no other matter has been added.
Fish guano; fish manure. ...	A product obtained by drying and grinding or otherwise treating fish or fish waste, to which no other matter has been added.
Hoofs	The product obtained by crushing or grinding hoof, to which no other matter has been added.
Hoofs and horns	A mixture of hoof and horn, crushed or ground, to which no other matter has been added.
Horns	The product obtained by crushing or grinding horn, to which no other matter has been added.
Meat and bone meal; meat meal; carcase meal; meat and bone tankage.	The product of drying and grinding or otherwise treating bone, flesh, flesh fibre and other slaughterhouse residues, to which no other matter has been added.
Rape meal	The residue which is obtained by the removal of oil from commercially pure rape seed.

Name of Material	*Meaning*
Raw guano	The excrement and remains of any birds, except poultry, containing both nitrogen and phosphorus, prepared for use by screening where necessary, but to which no addition has been made.
Shoddy manure; wool waste; wool combings; wool manure; flock dust.	Waste of wool, or of wool mixed with fibrous materials such as are associated with wool in the textile industries including cotton and similar non-wool materials, to which no other matter has been added, the fibre content of which contains not less than 50% of wool by weight.
Burnt or quick lime, ground or otherwise.	Commercial calcium oxide containing not more than 5·5% of magnesium (Mg).
Burnt magnesian lime, ground or otherwise.	Commercial calcium and magnesium oxides, containing more than 5·5% of magnesium (Mg).
Calcium hydroxide; hydrated lime; slaked lime.	The product obtained by slaking burnt lime.
Chalk	Cretaceous limestone.
Chalk, ground.	Cretaceous limestone which has been reduced in size so that it will pass through a sieve of $\frac{1}{4}$" square apertures.
Chalk, screened.	Cretaceous limestone that will pass through a sieve having apertures not exceeding 3" square.
Limestone, ground.	Sedimentary rock consisting largely of calcium carbonate, but containing not more than 3% of magnesium (Mg), which has been reduced in size so that 100% will pass through a sieve of $\frac{3}{16}$" square apertures, not less than 95% will pass through a sieve of $\frac{1}{8}$" square apertures and not less then 40% will pass through a British Standard Test Sieve Mesh No. 100.
Magnesian limestone, ground.	Sedimentary rock consisting largely of the carbonates of calcium and magnesium, but containing more than 3% of magnesium (Mg), which has been reduced in size so that 100% will pass through a sieve of $\frac{3}{16}$" square apertures, not less than 95% will pass through a sieve of $\frac{1}{8}$" square apertures, and not less than 40% will pass through a British Standard Test Sieve Mesh No. 100.
Mixed lime	A product, not being a by-product or a mixture of by-products from manufacturing or other processes, obtained by mixing two or more of the forms of liming materials in this Part of this Schedule.
Slaked magnesian lime ...	The product obtained by slaking burnt magnesian lime.

Any meaning contained in the second column of this Part of this Schedule shall be taken not to exclude the presence of a substance added to improve the handling qualities of the material, or the presence of boron, cobalt, copper, iron, magnesium, manganese or molybdenum (or a compound of any such element) or any herbicide or pesticide as defined in Schedule 2 the name of which appears in a statutory statement in accordance with Schedule 2.

PART II

FEEDING STUFFS

Name of Material	*Meaning*
Alfalfa meal; lucerne meal. ...	Alfalfa (lucerne), as grown, dried by natural means and ground, to which no other matter has been added.
Barley meal	The meal obtained by grinding barley, as grown, which shall be the whole grain together with only such other substances as may reasonably be expected to have become associated with the grain in the field and which contains not less than 96% pure barley.
Barley meal, grade II. ...	The meal, other than barley meal as defined above, obtained by grinding barley, as grown, which shall be the whole grain together with only such other substances as may reasonably be expected to have become associated with the grain in the field and which contains not less than 90% pure barley.
Bean meal	The meal obtained by grinding commercially pure beans of the species (1) *Vicia faba* or any of its varieties, commonly known as "horse bean", "field bean" or "broad bean"; or (2) *Phaseolus vulgaris*, the "true haricot bean" or any of its varieties, white or coloured.
Clover meal	Whole clover, as grown, dried by natural means and ground, to which no other matter has been added.
Cotton cakes or meals, not decorticated.	The residue resulting from the removal of oil from commercially pure cotton seed, not decorticated.
Cotton cakes or meals from decorticated or partly decorticated cotton seed.	The residue resulting from the removal of oil from commercially pure cotton seed from which the cortex, in whole or in part, has been removed.
Dari meal; durra meal. ...	The meal obtained by grinding commercially pure dari or durra seed.
Dried brewery grains... ...	The material produced by drying the residue of malted and unmalted cereals used in brewing, to which no other matter has been added.
Dried grass	Any product whether ground or not which: (*a*) is obtained by artificially drying any of the following:—grass, clover, lucerne, sainfoin, green cereals, or any mixture consisting of any of them, and (*b*) is otherwise as grown (that is to say, including any growths harvested therewith but with no other substance added thereto), and contains not less than 13% protein calculated on the assumption that it contains 10% moisture.
Dried grass (maintenance quality)	Dried grass within the meaning prescribed in this Part of this Schedule except that it may contain less than 13% but not less than 10% protein calculated on the assumption that it contains 10% moisture.
Dried green fodder crops ...	Any product whether ground or not which: (*a*) is obtained by artificially drying any green crop or crops suitable for use as dried fodder, and (*b*) is otherwise as grown (that is to say, including any growths as harvested therewith but with no other substance added thereto) and contains not less than 10% protein calculated on the assumption that it contains 10% moisture, but is not dried grass or dried grass (maintenance quality).

Name of Material	*Meaning*
Dried green roughage ...	Any product whether ground or not which contains less than 10% protein calculated on the assumption that is contains 10% moisture, but which in all other respects complies with the meaning for dried grass or dried green fodder crops given in this Part of this Schedule.
Dried plain beet pulp ...	The material produced by drying the sugar beet residue produced in the manufacture of sugar from sugar beet, with or without the addition of molasses, to give less than 10% of sugar.
Dried molassed beet pulp ...	The material produced by drying the sugar beet residue produced in the manufacture of sugar from sugar beet, with the addition of molasses, to give 10% or more of sugar.
Dried yeast	A material produced by drying yeast or yeast residues, to which no other matter has been added.
Extracted linseed meal ...	The residue resulting from the removal of oil from commercially pure linseed by means of a solvent.
Feeding bone flour	Commercially pure bone, degreased and ground or crushed, from which the nitrogen has been partly or wholly removed by steam.
Feeding bone meal; ground bone.	Commercially pure bone, raw or degreased, which has been ground or crushed.
Feeding dried blood	Blood which has been dried, to which no other matter has been added.
Feeding meat and bone meal	The product, containing not less than 40% of protein and not more than 4% of salt, obtained by drying and grinding animal carcases or portions thereof (excluding hoof, horn and feathers) and bone, to which no other matter has been added but which may have been preliminarily treated for the removal of fat.
Feeding meat meal	The product, containing not less than 55% of protein and not more than 4% of salt, obtained by drying and grinding animal carcases 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.
Fish meal; fish residue meal.	A product obtained by drying and grinding or otherwise treating fish or waste of fish, to which no other matter has been added.
Flaked maize	The product obtained by cooking and flaking commercially pure maize or Indian corn, either as grown or from which the germ, in whole or in part, has been removed.
Ground oats	The meal obtained by grinding commercially pure oats, as grown.
Linseed cakes or the meals of such cakes	The residue resulting from the removal of oil from commercially pure linseed.
Linseed meal	The meal obtained by grinding or crushing commercially pure linseed.
Locust bean meal	The meal obtained by grinding or crushing commercially pure locust beans.

Name of Material	*Meaning*
Maize germ cake or meal ...	A meal or cake resulting from the grinding of maize germs or from maize germs from which the oil has been removed in whole or in part.
Maize gluten feed	A by-product resulting from the removal of starch and germ from maize, to which no other matter has been added.
Maize meal; Indian meal. ...	The meal obtained by grinding commercially pure maize or Indian corn, as grown.
Malt culms	The rootlets and shoots arising from the screening of malt, to which no other matter has been added.
Molasses feeds	Any mixture (other than dried molassed beet pulp and mixtures of molasses and urea) containing not less than 10% of sugar, of an absorbent material and treacle or molasses.
Nut cakes or meals, including coconut, copra, palm kernel and ground nut cakes and meals.	The residue resulting from the removal of oil from commercially pure nut kernels.
Oatfeed	The by-product of oatmeal milling consisting of hulls, floury materials, mealy matter and scree dust, all finely ground, and containing not more than 27% of fibre.
Pea meal	The meal obtained by grinding commercially pure peas, as grown, of varieties of *Pisum sativum* or *Pisum arvense*.
Rape cake or meal	The residue resulting from the removal of oil from commercially pure rape seed.
Rice bran; rice meal. ...	The by-product produced in milling shelled rice, to which no other matter has been added.
Soya cake or meal	The residue resulting from the removal of oil from commercially pure soya beans.
Sugar beet treacle; sugar beet molasses.	A concentrated syrup product obtained in the manufacture of sugar from sugar beet, to which no other matter has been added.
Sugar cane treacle; sugar cane molasses.	A concentrated syrup product obtained in the manufacture of sugar from sugar cane, to which no other matter has been added.
Wheat meal	The meal obtained by grinding commercially pure wheat, as grown.
Wheat offals; millers' offals.	A product of wheat separated in the process of milling and containing not more than 4% of vegetable substances, other than wheat, extracted from wheat in the process of cleaning by the maker of the offals in the production of flour.
White fish meal	A product (containing not more than 6% of oil and not more than 4% of salt) obtained by drying and grinding or otherwise treating white fish or waste of white fish, to which no other matter has been added.

1. Any meaning contained in the second column of this Part of this Schedule shall be taken not to exclude the presence of a substance added to improve the keeping or handling qualities of the material or the presence of any added magnesium or any added substance of a kind referred to in regulation 7.

2. In this Part of this Schedule "commercially pure" means that no other matter has been added.

SCHEDULE 5

LIMITS OF VARIATION

(Section 74 and Regulation 14)

PART I

FERTILISERS

Material	*Limits of variation (percentages are percentages of the whole bulk)*
Compound fertiliser (as defined in paragraph 4 of Part I of Schedule 2)	Nitrogen, potash, phosphoric acid soluble in water, and phosphoric acid insoluble in water respectively, (*a*) 0·5% where the amount stated does not exceed 5%; (*b*) 0·75% where the amount stated exceeds 5% but does not exceed 8%; (*c*) one-eighth of the amount stated where the amount stated exceeds 8% and the quantity sampled does not exceed one ton; (*d*) one-tenth of the amount stated where the amount stated exceeds 8% and the quantity sampled exceeds one ton; provided that the variation from each amount stated shall not exceed 1·75% and, where the total of the amounts stated is 25% or over, the amount of all variations taken together, after setting off deficiencies against excesses, shall not exceed one-twentieth of the aforesaid total.
Any material mentioned in this Part of this Schedule containing boron, cobalt, copper, magnesium, manganese or molybdenum.	(*a*) Where the amount of boron, cobalt, copper, magnesium, manganese or molybdenum stated does not exceed 250 parts per million, one-half of the amount stated, or (*b*) where the amount of boron, cobalt, copper, magnesium, manganese or molybdenum stated exceeds 250 parts per million, three-tenths of the amount stated.
Any material mentioned in this Part of this Schedule containing iron.	(*a*) Where the amount of iron stated does not exceed 250 parts per million, in the case of a deficiency, one-half of the amount stated; in the case of an excess, no limit. (*b*) Where the amount of iron stated exceeds 250 parts per million, in the case of a deficiency, three-tenths of the amount stated; in the case of an excess, no limit.
Ammonium nitrate and mixtures of ammonium nitrate with any material mentioned in Group H of the table in Part I of Schedule 2	Nitrogen, one-twentieth of the amount stated.
Ammonium sulphate nitrate	Nitrogen, one-twentieth of the amount stated.
Calcium cyanamide	Nitrogen, 0·5%.
Nitrate of lime	Nitrogen, 0·5%.
Nitrate of soda	Nitrogen, 0·5%.

Material	*Limits of variation (percentages are percentages of the whole bulk)*
Nitrogenous gas liquor; ammoniacal gas liquor; gas liquor.	Nitrogen, 0·3%.
Sulphate of ammonia	Nitrogen, 0·3%.
Urea	Nitrogen, 0·3%.
Basic slag	Total phosphoric acid, 1%; phosphoric acid soluble in 2% citric acid, 1%; amount of the material that will pass through a 0·5 mm sieve, one-twentieth of the amount stated.
Dicalcium phosphate	Phosphoric acid soluble in citric acid, 1%.
Phosphate rock, ground or otherwise.	Phosphoric acid, one-twentieth of the amount stated; amount of the material that will pass through a British Standard Test Sieve Mesh No. 100, one-twentieth of the amount stated.
Superphosphate	Phosphoric acid soluble in water, one-twentieth of the amount stated.
Superphosphate, concentrated. ...	Phosphoric acid soluble in water, one-twentieth of the amount stated.
Superphosphate, triple.	Phosphoric acid soluble in water, one-twentieth of the amount stated.
Potassium salts used as fertilisers as described in Group D of the table in Part I of Schedule 2.	(*a*) 1%, where the percentage of potash stated does not exceed 15, or (*b*) 2%, where the percentage of potash stated exceeds 15.
Nitrate of potash	Nitrogen, 0·5%, potash, 2%.
Potassic nitrate of soda	Nitrogen, 0·5%, potash, 0·75%.
Potassic basic slag	Potash:— (*a*) 1%, where the percentage of potash stated does not exceed 15, or (*b*) 2%, where the percentage of potash stated exceeds 15, Total phosphoric acid, 1%; phosphoric acid soluble in 2% citric acid, 1%; amount of slag that will pass through a 0·5 mm sieve, one-twentieth of the amount stated.
Bone meal or other bone product as described in the table in Part I of Schedule 2	Nitrogen, 0·5%; phosphoric acid, 1·5%.
Bone phosphate precipitated; dicalcium bone phosphate.	Phosphoric acid soluble in citric acid, 1%.
Dissolved or vitriolised bone—	
(i) When the total of the percentages of phosphoric acid (soluble and insoluble) stated amounts to 14 or more, then:	
(*a*) if the excess of the actual percentage of insoluble phosphoric acid over that stated is 1·5 or more	Nitrogen, 0·3%; phosphoric acid soluble in water, 2%.

Material	*Limits of variation (percentages are percentages of the whole bulk)*
(*b*) if such excess is not less than 1, but is less than 1·5	Nitrogen, 0·3%; phosphoric acid soluble in water, 1·5%.
(*c*) if such excess is not less than 0.5, but is less than 1 ...	Nitrogen, 0·3%; phosphoric acid soluble in water, 1%.
(ii) in all other cases	Nitrogen, 0·3%; phosphoric acid soluble in water, 0·5%; phosphoric acid insoluble in water, 0·5%.
Dried blood for fertilising purposes ...	Nitrogen 0·5%.
Fish residues or other fish product as described in the table in Part I of Schedule 2	Nitrogen, 0·5% and phosphoric acid, 1%; provided that the aforesaid limits may be extended if:— (*a*) an excess of one of the said constituents is offset by a deficiency of the other in the proportion of 0·25% nitrogen to 1% phosphoric acid, and (*b*) the extension of the aforesaid limits does not exceed for nitrogen 0·75% and for phosphoric acid 3%.
Guano as described in the table in Part I of Schedule 2	Nitrogen, one-fifth of the amount stated, with a minimum of 0·25% and a maximum of 1·5%; phosphoric acid, one-tenth of the amount stated, with a maximum of 2%; and potash, one-fifth of the amount stated.
Hoofs	Nitrogen, 0·5%.
Hoofs and horns	Nitrogen, 0·5%.
Horns	Nitrogen, 0·5%.
Meat and bone residues as described in the table in Part I of Schedule 2	Nitrogen, 0·5% and phosphoric acid, 1%; provided that the aforesaid limits may be extended if:— (*a*) an excess of one of the said constituents is offset by a deficiency of the other in the proportion of 0·25% nitrogen to 1% phosphoric acid, and (*b*) the extension of the aforesaid limits does not exceed for nitrogen 0·75% and for phosphoric acid 3%.
Oilseed fertilisers as described in the table in Part I of Schedule 2	Nitrogen, 0·5%.
Burnt or quick lime, ground or otherwise.	Neutralising value, one-tenth of the amount stated.
Burnt magnesian lime, ground or otherwise.	Neutralising value, one-tenth of the amount stated.
Calcium hydroxide; hydrated lime; slaked lime; slaked magnesian lime.	Neutralising value, one-tenth of the amount stated.
Chalk, ground.	Neutralising value, one-twentieth of the amount stated.
Chalk, screened.	Neutralising value, one-eighth of the amount stated; amount of the material that will pass through a declared British Standard Test Sieve, one-tenth of the amount stated.

Material	*Limits of variation (percentages are percentages of the whole bulk)*
Limestone, ground; magnesian limestone, ground.	Neutralising value, one-twentieth of the amount stated; amount of the material that will pass through a British Standard Test Sieve Mesh No. 100, one-twentieth of the amount stated.
Mixed lime	Neutralising value, one-tenth of the amount stated.

PART II

FEEDING STUFFS

Compound feeding stuff	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; protein equivalent of urea, 1·25% or one-fifth of the amount stated, whichever is the greater; fibre, if the actual amount exceeds that stated, 0·5% or one-eighth of the amount stated, whichever is the greater, if the actual amount is less than that stated, 0·5% or one-half of the amount stated, whichever is the greater.
Any material, not being a feed supplement, mentioned in this Part of this Schedule containing cobalt, iodine, manganese, molybdenum, selenium or zinc	One-half the amount of cobalt, iodine, manganese, molybdenum, selenium or zinc stated.
Any material, not being a feed supplement, mentioned in this Part of this Schedule containing copper	Where the amount of copper stated is between 50 parts per million and 200 parts per million, one-half of the amount stated.
Any material, not being a feed supplement, mentioned in this Part of this Schedule containing iron	(*a*) Where the amount of iron stated is less than 250ppm, one-half of the amount stated, or (*b*) where the amount of iron stated is 250ppm, or greater then 30% of the amount stated.
Any material, not being a feed supplement, mentioned in this Part of this Schedule containing vitamins other than vitamin D_2 or vitamin D_3	Where the actual amount is less than that stated, 30% of the amount stated; in the case of an excess, no limit.
Any material, not being a feed supplement, mentioned in this Part of this Schedule containing vitamin D_2 or vitamin D_3	(*a*) Where the amount of vitamin D_2 or vitamin D_3 stated does not exceed 4000 IU/kg, 50% of the amount stated, or (*b*) where the amount of vitamin D_2 or vitamin D_3 stated exceeds 4000 IU/kg, 30% of the amount stated.
Any material mentioned in this Part of this Schedule containing magnesium	In the case of a deficiency of magnesium, three-tenths of the amount stated; in the case of an excess, no limit.
Alfalfa meal; lucerne meal	Protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Clover meal	Protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Coconut or copra cake or meal ...	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated.

Material	*Limits of variation (percentages are percentages of the whole bulk)*
Cotton cakes or meals not decorticated	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated.
Cotton cakes or meals from decorticated or partly decorticated cotton seed	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Dried brewery grains...	Oil, 0·75%, or one-fifth of the amount stated, whichever is the greater; protein, one-fifth of the amount stated.
Dried distillery by-products (other than malt culms and dried yeast)	Oil, 0·75%, or one-fifth of the amount stated, whichever is the greater; protein, one-fifth of the amount stated; fibre, if present in excess of 2%, if the actual amount exceeds that stated, one-eighth of the amount stated, if the actual amount is less than that stated, one-half of the amount stated; lime expressed as calcium (Ca), if present in excess of 2%, one-fifth of the amount stated.
Dried grass Dried grass (maintenance quality) Dried green fodder crops Dried green roughage	Protein, one-tenth of the amount stated, provided that this limit of variation shall not operate so as to permit the application of the name "dried grass" to any article containing less than 13% protein or the names "dried grass (maintenance quality)" or "dried green fodder crops" to any material containing less then 10% protein.
Dried plain beet pulp...	Fibre, one-eighth of the amount stated.
Dried yeast	Protein, one-twentieth of the amount stated.
Dried molassed beet pulp	Sugar, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Feed supplement	Protein equivalant of urea, 1·25%, or one-fifth of the amount stated whichever is the greater; iodine, cobalt, copper, iron, manganese, zinc, molybdenum, selenium, vitamin D_2 and vitamin D_3, 30% of the amount stated; vitamins other than vitamin D_2 or vitamin D_3, if the actual amount is less than that stated, 30% of the amount stated, in the case of an excess, no limit.
Feeding bone flour	Phosphoric acid, one-twentieth of the amount stated; protein, one-fifth of the amount stated.
Feeding bone meal, ground bone, or any other bone product for feeding purposes.	Phosphoric acid and protein, one-tenth of the respective amounts stated.
Feeding dried blood	Protein, one-twentieth of the amount stated.
Feeding meat meal or any other product of meat for feeding purposes Feeding meat and bone meal or any other product of meat and bone for feeding purposes	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein and phosphoric acid, one-tenth of the respective amounts stated; provided that these limits of variation shall not operate so as to permit the application of the names "feeding meat meal" and "feeding meat and bone meal" to materials containing less than 55% and less than 40% of protein respectively.

Material	*Limits of variation (percentages are percentages of the whole bulk)*
Fish meal, white fish meal, or any other product obtained by drying or grinding or otherwise treating fish or fish waste.	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; phosphoric acid, one-sixth of the amount stated; salt, 0·75%; provided that these limits of variation shall not operate so as to permit the application of the name "white fish meal" to material containing more than 6% of oil or 4% of salt.
Linseed cakes and the meals of such cakes; extracted linseed meal.	Oil, 0·75%, or one-eighth of the amount stated, whichever is the greater; protein, one-eighth of the amount stated.
Linseed meal	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater.
Maize by-products, not otherwise mentioned in the table in Part II of Schedule 2.	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Maize, flaked.	Oil, 0·75%, or one-eighth of the amount stated, whichever is the greater; protein, one-eighth of the amount stated.
Maize germ cake or meal	Oil, 0·75%, or one-eighth of the amount stated, whichever is the greater; protein, one-eighth of the amount stated.
Maize gluten feed	Oil, 0·75%, or one-eighth of the amount stated, whichever is the greater; protein, one-eighth of the amount stated.
Malt culms	Protein, one-fifth of the amount stated; fibre, one-eighth of the amount stated.
Milk powders including oil and/or fat fortified milk powders	Oil, 0.75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated.
Molasses feeds, as described in the table in Part II of Schedule 2.	Sugar, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Mixtures of molasses and urea ...	Sugar, one-tenth of the amount stated; protein equivalent of urea, one-fifth of the amount stated.
Oatmeal by-products	Fibre, one-eighth of the amount stated; provided that this limit of variation shall not operate so as to permit the application of the name "oatfeed" to any material containing more than 27% of fibre.
Oil cakes or meals not otherwise mentioned in the table in Part II of Schedule 2 which are the product of any one decorticated or partly decorticated substance or seed from which oil has been removed	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Oil cakes or meals not otherwise mentioned in the table in Part II of Schedule 2 which are the product of any one undecorticated substance or seed from which oil has been removed	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated.

Material	*Limits of variation (percentages are percentages of the whole bulk)*
Palm kernel cake or meal	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated.
Rape cake or meal	Oil, 0·75%, or one-eighth of the amount stated, whichever is the greater; protein, one-eighth of the amount stated.
Rice bran or rice meal, or the by-product produced in milling shelled rice.	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
Soya cake or meal	Oil, 0·75%, or one-eighth of the amount stated, whichever is the greater; protein, one-eighth of the amount stated.
Treacle or molasses	Sugar, one-twentieth of the amount stated.
Wheat offals or millers' offals ...	Fibre, if the actual amount exceeds that stated, one-eighth of the amount stated; if the actual amount is less than that stated, one-half of the amount stated.

SCHEDULE 6

METHODS OF ANALYSIS OF FERTILISERS

(Sections 68(5), 69(5), 70(4), 71(3), 75(1), 77(4), 78(6) and 79(3) and Regulation 15)

(In this Schedule a "decimal" system has 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 this 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 thiocyanate in ammoniacal gas liquor; nitrogenous gas liquor; gas liquor.
9. Determination of biuret.
10. Determination of boron.
11. Determination of cobalt.
12. Determination of copper.
13. Determination of iron.
14. Determination of magnesium.
15. Determination of manganese.
16. Determination of molybdenum.
17. Determination of fineness.

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

1· PREPARATION OF THE 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 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 fertilisers, 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 granular fertilisers

Grind the sample as rapidly as possible to pass through a sieve having apertures of about 1 mm square*†. Mix thoroughly and take a representative portion of about 250 g. Grind this portion to pass through the appropriate sieve† prescribed in paragraph 1·16 and transfer to a non-corrodible container provided with an air-tight closure.

1·12 Crystalline fertilisers, e.g. sulphate of potash and nitrate of soda

Grind the sample as rapidly as possible to pass through the appropriate sieve prescribed in paragraph 1·16. Mix, withdraw a portion for analysis and grind to a fine condition in a mortar. (If the sample is in a damp condition, grind thoroughly in a mortar until a uniformly fine texture is obtained.) Transfer to a non-corrodible container provided with an air-tight 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 to pass through the appropriate sieve prescribed in paragraph 1·16 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.

1·14 Fertilisers 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 fertilisers in which ammonia is lost on heating or of fertilisers 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 until the sample passes through the appropriate sieve prescribed in paragraph 1·16. 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.

1·15 Liquid fertilisers

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

---

* British Standard Test Sieve. Mesh No. 16 is suitable (British Standard for Test Sieves 410: 1962).

† Where an analysis for copper has to be carried out a stainless steel sieve shall be used.

1·16 **Sieve**

Type of fertiliser	*Sieve apertures*
Ground mineral phosphate and granular fertilisers with the exception of basic slag and potassic basic slag	About 0·25 mm square†
Other dry powdered fertilisers	About 0·5 mm square‡
Crystalline fertilisers and fertilisers containing organic matter	About 1·0 mm square§

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.

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.

3·5 Nitrogen in the form of ammonium salts, and nitrogen in nitrogenous gas liquor, ammoniacal gas liquor and gas liquor.

3·6 Nitrogen in nitrates.

3·7 Nitrate nitrogen in the presence of ammoniacal and urea nitrogen.

3·1 REAGENTS

Aluminium ammonium sulphate.

Devarda alloy—finely powdered—not less than 80 per cent to pass through a sieve having apertures of about 0·25 mm square†.

4-*Dimethylaminobenzaldehyde solution*—Dissolve 0·4 g 4-dimethylaminobenzaldehyde in 10 ml concentrated hydrochloric acid and dilute to 100 ml with propan-2-ol.

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 1 minute.

†British Standard Test Sieve, Mesh No. 60 is suitable
‡British Standard Test Sieve, Mesh No. 30 is suitable
§British Standard Test Sieve, Mesh No. 16 is suitable
} British Standard for Test Sieves 410: 1962.

Mercuric oxide.

Methyl red-methylene blue mixed indicator solution—Mix 2 volumes of methyl red solution and 1 volume of methylene blue solution prepared as follows:

Methyl red solution—Dissolve 0·05 g methyl red in ethanol and dilute to 100 ml with ethanol.

Methylene blue solution—Dissolve 0·05 g methylene blue in ethanol and dilute to 100 ml with ethanol.

Paraffin wax.

Sodium sulphate or potassium sulphate—anhydrous.

Sodium hydroxide solution, 5 per cent w/v—Dissolve 50 g sodium hydroxide in water and dilute to 1 litre.

Sodium hydroxide solution, 50 per cent w/v—Dissolve 500 g sodium hydroxide in water and dilute to 1 litre.

Sodium hydroxide, 0·2 N—carbonate free.

Sodium thiosulphate.

Sodium tungstate solution, 10 per cent w/v—Dissolve 10 g sodium tungstate $Na_2WO_4.2H_2O$ in water and dilute to 100 ml.

Sulphuric acid, concentrated (d=1·84)—nitrogen free.

Sulphuric acid, 5 per cent v/v—To 50 ml water cautiously add 5 ml concentrated sulphuric acid (d=1·84). Cool and dilute to 100 ml.

Sulphuric acid, 10 per cent v/v—To 500 ml water cautiously add 100 ml concentrated sulphuric acid. Cool and dilute to 1 litre.

Sulphuric acid, 50 per cent 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.

Urease tablets—of known activity.

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 the 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 mercuric oxide, 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 2 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 per cent sodium hydroxide solution to neutralise the acid and 10 ml in excess; then add 5 g sodium thiosulphate, mix well and connect immediately to a distillation apparatus. Distil into an appropriate volume of 0·2 N acid, controlling the rate of distillation so that not less than 150 ml distils in 30 minutes. Titrate the excess of acid with 0·2 N sodium hydroxide solution using methyl red-methylene blue mixed indicator 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 nitrates**

Weigh to the nearest mg about 2 g of the sample (or such an amount as shall contain not more than 250 mg nitrogen), transfer to a 500 ml 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 a tap funnel and a delivery tube connected with two 'U'-tubes (with bulbs) in series, each containing 10 ml 10 per cent sulphuric acid. Add 5 ml 50 per cent sodium hydroxide solution through the tap funnel, allow to stand for 30 minutes, and then heat just below boiling point for 60 minutes. Cool, add 20 ml 50 per cent 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 2 hours. If frothing is excessive add 0·5 g paraffin wax. Cool, carefully dilute with water, cool and transfer quantitatively to a 250 ml volumetric flask. Dilute to 250 ml, mix well and transfer an aliquot of 100 ml to a 500 ml distillation flask. Add 200 ml water and 50 per cent sodium hydroxide solution, until the solution is neutral, cooling during the addition. Add an additional 10 ml 50 per cent sodium hydroxide, quickly close the distillation flask and distil about 150 ml into 50 ml 0·2 N hydrochloric acid. Titrate the excess acid using 0·2 N sodium hydroxide solution and methyl red-methylene blue mixed indicator. 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.

3·5 **Nitrogen in the form of ammonium salts and nitrogen in nitrogenous gas liquor, ammoniacal gas liquor or gas liquor**

3·51 *In the absence of organic matter*

Weigh to the nearest mg about 5 g of the sample, transfer to a 250 ml volumetric flask, dissolve in about 200 ml water and dilute with water to 250 ml. Transfer 50 ml of the solution (or such a volume as shall contain not more than 250 mg nitrogen) to a distillation flask, add approximately 300 ml water and 20 ml 50 per cent sodium hydroxide solution. Distil into an appropriate volume of 0·2 N acid at the rate of 250-300 ml in 30 minutes. Titrate the excess of acid with 0·2 N sodium hydroxide solution using methyl red-methylene blue mixed indicator solution. Carry out a blank test on the reagents and water used omitting only the sample. Calculate the nitrogen content. 1 ml 0·2 N acid ≡ 0·0028 g nitrogen.

3·52 *In the presence of organic matter other than urea*

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 the water-soluble matter. Dilute to 250 ml, filter, and complete the determination with 50 ml of the filtrate by the method described in paragraph 3·51.

3·53 *In the presence of urea*

Weigh to the nearest mg about 5 g of the sample (or such an amount as shall contain not more than 1 g ammoniacal nitrogen), and transfer to a 250 ml volumetric flask. Add 200 ml water, shake well to dissolve soluble salts, dilute to 250 ml and mix well. Filter a portion through a suitable dry filter paper, rejecting the first 25 ml and transfer an aliquot of 25 ml to a thick walled, 1 litre, round bottom flask. Fit a tap funnel, thermometer and an "air-bleed" terminating in a capillary tube reaching to the bottom of the flask as used in distillations under reduced pressure, an efficient spray trap, a double surface condenser and a 750 ml Buchner flask as receiver. The condenser should reach almost to the bottom of the Buchner flask. Connect the sidearm of the Buchner flask to a vacuum pump and fit a mercury manometer into the

system. Control the flow of air through the capillary in the distillation flask by means of a screw clip or similar device, on a thick walled rubber tube fixed to the open end of the capillary tube. Place 50 ml 0·2 N acid in the Buchner flask and connect to the condenser. Add 250 ml water and 20 ml 50 per cent sodium hydroxide solution through the tap funnel to the contents of the distillation flask. Start the vacuum pump, adjust the "air-bleed" to a pressure of approximately 55 mm of mercury. Heat to a temperature of not greater than 40 °C. Distil for twenty minutes. At the end of the distillation, admit air to the apparatus via the tap tunnel and disconnect the vacuum pump. Titrate the excess acid with 0·2 N sodium hydroxide solution using methyl red-methylene blue mixed indicator. Carry out a similar determination using all the reagents omitting only the sample. Calculate the nitrogen content. 1 ml 0·2 N acid≡0·0028 g nitrogen.

3·6 **Nitrogen in nitrates**

Weigh to the nearest mg about 3 g of the sample, transfer to a 250 ml volumetric flask, add 200 ml water, shake well to ensure complete solution, dilute to 250 ml and, if necessary, filter. Transfer 50 ml of the solution or filtrate (or such a volume as shall contain not more than 250 mg nitrogen) to a distillation flask. Add 10 g Devarda alloy, 250 ml water and 15 ml 50 per cent sodium hydroxide solution. Connect the flask immediately to the distillation apparatus and allow to stand in the cold for 15 minutes. Warm gently for a further 30 minutes, slowly increasing the temperature, and then distil into an appropriate volume of 0·2 N acid at the rate of not less than 150 ml in 30 minutes (the residual bulk should be small). Titrate the excess acid with 0·2 N sodium hydroxide solution using methyl red-methylene blue mixed indicator solution. Carry out a blank test on the reagents omitting the water solution of the sample. Calculate the nitrogen content. 1 ml 0·2 N acid ≡0·0028 g nitrogen.

NOTE: If nitrogen in other forms is also present, the method described in paragraph 3·7 should be used.

3·7 **Nitrate nitrogen in the presence of ammoniacal and urea nitrogen**

Weigh to the nearest mg about 5 g of the sample (or such an amount as shall contain not more than 2·5 g nitrate nitrogen), and transfer to a 500 ml volumetric flask. Add 400 ml water, shake well to ensure dissolution of all soluble salts, dilute to 500 ml with water and shake well. Transfer 100 ml to a 250 ml beaker, and adjust to pH 6-7 with 5 per cent sodium hydroxide or 5 per cent sulphuric acid solution. Transfer quantitatively to a 250 ml volumetric flask, dilute to 200 ml and add sufficient crushed urease tablets to hydrolyse all the urea present, and add 25 per cent in excess. Stopper the flask and allow to stand in a constant temperature bath at 37 °C for 3 hours.

Dilute the solution to 250 ml and mix well. Filter a portion through a suitable dry filter paper and test for absence of urea. (see note 1). If free from urea, filter the remainder, transfer a 100 ml aliquot of the urea-free filtrate to a 250 ml volumetric flask, add 10 ml sodium tungstate solution or sufficient to precipitate all the protein matter, dilute to 250 ml with water and mix well. Filter a portion through a suitable dry filter paper, and transfer a 100 ml aliquot to a 500 ml distillation flask. Add 200 ml water, 15 ml 50 per cent sodium hydroxide solution and distil until 150 ml is collected. Discard the distillate. Cool the flask, dilute the contents to 300 ml, add 3 g Devarda alloy and 15 ml 50 per cent sodium hydroxide solution and immediately connect the flask to the distillation apparatus with the condenser outlet below the surface of 50 ml 0·2 N acid in the receiver. Allow to stand in the cold for 15 minutes, warm for 30 minutes, slowly increasing the temperature, and then distil 150 ml into the receiver. Titrate the excess acid with 0·2 N sodium hydroxide using methyl red-methylene blue mixed indicator. Calculate the nitrate nitrogen content of the sample after making allowance for the reagent blank. (see note 2).

NOTE 1

The test for urea is carried out as described in (i) and (ii) below.

(i) *Instrumental method*

Transfer a 50 ml aliquot of the solution after urease treatment to a 100 ml stoppered cylinder, adjust to pH 5, add 1 g of activated charcoal and 5 ml each of Carrez solutions 1 and 2 (see method for determination of urea—paragraph 5, Schedule 7). Dilute to 70 ml, mix well and filter a portion through a suitable dry filter paper. Transfer 35 ml of the filtrate to a 50 ml flask, add 10 ml 4-dimethylaminobenzaldehyde solution, dilute to 50 ml and mix well. Allow to stand 10 minutes and compare the extinction at 435 nm in a 1 cm cell with that of a blank test carried through the method described, omitting only the sample. The amount of urea present should not exceed 0·5 mg, determined from a previously prepared calibration curve.

(ii) *Visual Method*

Transfer a 50 ml aliquot of the solution after urease treatment to a 100 ml stoppered cylinder, adjust to pH 5, add 1 g activated charcoal and 5 ml each of Carrez solutions 1 and 2 (see method for determination of urea—paragraph 5, Schedule 7).

Dilute to 70 ml, mix well and filter a portion through a suitable dry filter paper. Transfer 35 ml of the filtrate to a Nessler tube, add 10 ml of 4-dimethylaminobenzaldehyde solution, dilute to 50 ml with water and mix well. To a 50 ml aliquot of the blank solution prepared for the determination of nitrate, add 1 mg of urea and treat exactly as described above. This is the control solution. The depth of colour of the sample solution should not exceed that of the control solution.

NOTE 2

Carry out a similar determination using all the reagents as in the method described, omitting only the sample. The reagent blank is required at two stages in the determination, namely in the test for absence of urea, and in the determination of the nitrate nitrogen.

4 DETERMINATION OF PHOSPHORIC ACID

For the purposes of the Agriculture Act 1970, Part IV, "phosphoric acid" means P_2O_5 (molecular weight 142·04).

Phosphoric acid may be determined by the quinolinium phosphomolybdate method or, alternatively, by the spectrophotometric (vanadium phosphomolybdate) method.

The quinolinium phosphomolybdate method depends on the precipitation of quinolinium phosphomolybdate under carefully controlled conditions; citric acid is added in appropriate amounts to prevent interference by soluble silica or ammonium salts in the amounts present in the materials to be analysed. 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 wavelength of 420 nm is required; alternatively a filter instrument can be used.

Phosphoric acid in materials other than basic slag, potassic basic slag, dicalcium phosphate, precipitated bone phosphate and dicalcium bone phosphate may be required to be determined as water-soluble and water-insoluble and as total phosphoric acid. In the analysis of basic slag, potassic basic slag,

dicalcium phosphate, precipitated bone phosphate and dicalcium bone phosphate, solubility in a 2 per cent solution of citric acid is substituted for solubility in water. Because of the chemical composition of basic slag and potassic basic slag, the methods of analysis differ in several respects from the methods for other fertilisers when the quinolinium phosphomolybdate method is used; these modified methods are given separately in paragraphs 4·16 and 4·17. When phosphoric acid soluble in citric acid is being determined by the spectrophotometric method, certain modifications in the procedure for the standardisation of the spectrophotometer are necessary and these are given separately in paragraphs 4·26 and 4·27.

The relevant methods of analysis are described in the following paragraphs:—

4·12 and 4·22 Total phosphoric acid in fertilisers other than basic slag and potassic basic slag.

4·13 and 4·23 Water-soluble phosphoric acid.

4·14 and 4·24 Water-insoluble phosphoric acid.

4·15 and 4·25 Citric acid-soluble phosphoric acid in fertilisers other than basic slag and potassic basic slag.

4·16 and 4·26 Total phosphoric acid in basic slag and potassic basic slag.

4·17 and 4·27 Citric acid-soluble phosphoric acid in basic slag and potassic basic slag.

4·1 QUINOLINIUM PHOSPHOMOLYBDATE METHOD

4·11 REAGENTS

Calcium oxide—finely ground.

Calcium carbonate.

Citric acid—monohydrate.

Citric-molybdic acid solution (*A*), for use in the determination of water-soluble, citric acid-soluble and total phosphoric acid in fertilisers other than basic slag and potassic basic slag—Stir 54 g molybdenum trioxide (MoO_3) with 200 ml water, add 11 g sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdenum trioxide dissolves. Dissolve 60 g citric acid in about 250 to 300 ml water and add 140 ml concentrated hydrochloric acid. Pour the molybdate solution into the acid solution, which is stirred throughout the addition. Then cool, and, if necessary, filter the solution through a paper pulp pad. Dilute the solution to 1 litre. If the solution is slightly green or blue in colour, add dropwise a dilute (0·5 or 1·0 per cent) solution of potassium bromate until the colour is discharged. This reagent should be kept in the dark.

Citric-molybdic acid solution (*B*), for use in the determination of citric acid-soluble and total phosphoric acid in basic slag and potassic basic slag—Stir 54 g molybdenum trioxide (MoO_3) with 200 ml water, add 11 g sodium hydroxide and stir the mixture, whilst heating to boiling point until the molybdenum trioxide dissolves. Dissolve 120 g citric acid in about 250 to 300 ml water and add 140 ml concentrated hydrochloric acid. Pour the molybdate solution into the acid solution, which is stirred throughout the addition. Then cool and, if necessary, filter the solution through a paper pulp pad. Dilute the solution to 1 litre. If the solution is slightly green or blue in colour, add dropwise a dilute (0·5 or 1·0 per cent) solution of potassium bromate until the colour is discharged. This reagent should be kept in the dark.

Hydrochloric acid, concentrated (d = 1·18).

Hydrochloric acid, 25 per cent v/v—Dilute 25 ml concentrated hydrochloric acid with water to 100 ml.

Hydrochloric acid, 0·1 *N*.

Hydrochloric acid, 0·5 *N*.

Indicator solution—Mix 3 volumes of thymol blue solution and 2 volumes of phenolphthalein solution prepared as follows:—

Thymol blue solution—Dissolve 0·25 g thymol blue in 5·5 ml 0·1 N sodium hydroxide solution and 125 ml industrial methylated spirit. Dilute with water to 250 ml.

Phenolphthalein solution—Dissolve 0·25 g phenolphthalein in 150 ml industrial methylated spirit and dilute with water to 250 ml.

Nitric acid, concentrated (d = 1·42).

Quinoline solution—Measure 60 ml concentrated hydrochloric acid and 300 to 400 ml water into a 1 litre beaker and warm to 70-80 °C. Pour 50 ml quinoline in a thin stream into the diluted acid, whilst stirring. When the quinoline has dissolved, cool the solution, dilute to 1 litre and, if necessary, filter through a paper pulp filter.

Sodium hydroxide, 5 *N*.

Sodium hydroxide, 0·1 *N*—carbonate free.

Sodium hydroxide, 0·5 *N*—carbonate free.

Surface active agent—0·5 per cent solution of sodium dodecylbenzene-sulphonate is suitable.

4·12 **Total phosphoric acid in fertilisers other than basic slag and potassic basic slag.**

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 10 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.

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 1 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 10 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 10 minutes. Then cool, transfer to a 500 ml volumetric flask and dilute to the mark. Filter.

4·122 PROCEDURE

Transfer a volume of the solution prepared according to paragraph 4·1211 or paragraph 4·1212 containing less than 70 mg phosphoric acid and preferably

about 50 mg to a 500 ml stoppered conical flask marked at 150 ml. Dilute the solution with water to 100 ml. If the sample does not contain calcium add 100 to 200 mg calcium carbonate. Then add 5 N sodium hydroxide solution dropwise until a faint permanent turbidity or precipitate is formed. Dissolve the precipitate by the dropwise addition of 25 per cent hydrochloric acid, but avoid an excess.

Dilute to 150 ml, add 50 ml of the citric-molybdic acid reagent (A), heat the solution to incipient ebullition, maintain it at this temperature for 3 minutes and then bring it to the boiling point. From a burette slowly add 25 ml of the quinoline solution, with constant swirling throughout, the first few ml being added dropwise, the rest in a slow stream. Keep the solution gently boiling during the addition. Immerse the flask in boiling water for 5 minutes, then cool it to 15 °C in running water.

Filter with suction the contents of the flask on a paper pulp pad and wash the flask, precipitate and filter with successive small washes of cold water until they are free from acid. Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask. If necessary, wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate, and place the paper in the flask. Add water to a total of about but not exceeding 100 ml. Stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

Remove the stopper and wash it with water, returning the washings to the flask. Add a measured volume of 0·5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml in excess. Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after addition of 0·5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary.) Add 0·5-1·0 ml of the indicator solution and titrate the excess of sodium hydroxide with the 0·5 N hydrochloric acid until the indicator changes from violet to green-blue and then very sharply to yellow at the end point. Deduct the number of ml of 0·5 N hydrochloric acid used from the number of ml 0·5 N sodium hydroxide, to ascertain the volume of 0·5 N sodium hydroxide equivalent to the phosphoric acid.

Carry out a blank determination on all the reagents, omitting only the sample, and using 0·1 N standard alkali and acid instead of 0·5 N for the titration. Calculate the blank in terms of 0·5 N alkali and subtract it from the original result.

Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1·0 ml 0·5 N sodium hydroxide$\equiv$1·366 mg P_2O_5.

4·13 Water soluble phosphoric acid

4·131 Extraction of the sample

Weigh to the nearest centigram about 10 g of the sample and transfer to a 500 ml volumetric flask; add 400 ml water at 20 °C and shake the flask continuously for 30 minutes. Dilute the contents to the mark, mix well and filter.

4·132 Procedure

Transfer a volume of the aqueous extract containing less than 70 mg of phosphoric acid and preferably about 50 mg to a 500 ml stoppered conical flask marked at 150 ml. Dilute with water to 150 ml, add 50 ml of the citric-molybdic acid reagent (A), heat the solution to incipient ebullition, maintain it at this temperature for 3 minutes, and then bring it to the boiling point. From a burette slowly add 25 ml of the quinoline solution with constant swirling throughout, the first few ml being added dropwise, the rest in a slow stream. Keep the solution gently boiling during the addition. Immerse the flask in boiling water for 5 minutes, then cool it to 15 °C in running water.

Filter with suction the contents of the flask on a paper pulp pad, and wash the flask, precipitate and filter with successive small washes of cold water until they are free from acid. Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask. If necessary ,wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate, and place the paper in the flask. Add water to a total of about but not exceeding 100 ml. Stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

Remove the stopper and wash it with water, returning the washings to the flask. Add a measured volume of 0·5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml in excess. Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after addition of 0·5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary.) Add 0·5-1·0 ml of the indicator solution, and titrate the excess of sodium hydroxide with the 0·5 N hydrochloric acid until the indicator changes from violet to green-blue and then very sharply to yellow at the end point. Deduct the number of ml of 0·5 N hydrochloric acid used from the number of ml 0·5 N sodium hydroxide, to ascertain the volume of 0·5 N sodium hydroxide equivalent to the phosphoric acid.

Carry out a blank determination on all the reagents, omitting only the sample and using 0·1 N standard alkali and acid instead of 0·5 N for the titration. Calculate the blank in terms of 0·5 N alkali and subtract it from the original result.

Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1·0 ml 0·5 N sodium hydroxide≡1·366 mg P_2O_5.

4·14 **Water-insoluble phosphoric acid**

Determine the water-insoluble phosphoric acid as the difference between the total phosphoric acid determined by the method described in paragraph 4·12 and the water-soluble phosphoric acid determined by the method described in paragraph 4·13.

4·15 **Citric acid-soluble phosphoric acid in fertilisers other than basic slag and potassic basic slag**

4·151 PREPARATION OF THE SOLUTION

Weigh to the nearest mg about 5 g of the sample and transfer to a stoppered bottle of about 1 litre capacity. Dissolve 10 g citric acid monohydrate in water, dilute to 500 ml and adjust the temperature to 20 °C. Add the solution to the sample in the bottle, shaking so as to avoid the possibility of caking. Shake the bottle continuously for 30 minutes. Pour the whole of the liquid at once on to a large medium-fine filter and collect the filtrate. If the filtrate is not clear, pass it again through the same filter.

4·152 PROCEDURE

Transfer a volume of the solution prepared according to paragraph 4·151 containing less than 70 mg phosphoric acid and preferably about 50 mg to a 500 ml stoppered conical flask marked at 150 ml. Dilute the solution with water to 100 ml. If the sample does not contain calcium add 100 to 200 mg calcium carbonate. Then add 5 N sodium hydroxide solution dropwise until a faint permanent turbidity or precipitate is formed. Dissolve the precipitate by the dropwise addition of 25 per cent hydrochloric acid, but avoid an excess.

Dilute to 150 ml and add 50 ml of the citric-molybdic acid reagent (A), heat the solution to incipient ebullition, maintain it at this temperature for 3 minutes and then bring it to the boiling point. From a burette slowly add 25 ml of the quinoline solution with constant swirling throughout, the first few ml being added dropwise, the rest in a slow stream. Keep the solution gently boiling during the addition. Immerse the flask in boiling water for 5 minutes, then cool it to 15 °C in running water.

Filter with suction the contents of the flask on a paper pulp pad, and wash the flask, precipitate and filter with successive small washes of cold water until they are free from acid. Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask. If necessary, wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate, and place the paper in the flask. Add water to a total of about but not exceeding 100 ml. Stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

Remove the stopper and wash it with water, returning the washings to the flask. Add a measured volume of 0·5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml in excess. Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after addition of 0·5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary.) Add 0·5-1·0 ml of the indicator solution, and titrate the excess of sodium hydroxide with the 0·5 N hydrochloric acid until the indicator changes from violet to green-blue and then very sharply to yellow at the end point. Deduct the number of ml of 0·5 N hydrochloric acid used from the number of ml 0·5 N sodium hydroxide, to ascertain the volume of 0·5 N sodium hydroxide equivalent to the phosphoric acid.

Carry out a blank determination on all the reagents, omitting only the sample, and using 0·1 N standard alkali and acid instead of 0·5 N for the titration. Calculate the blank in terms of 0·5 N alkali and subtract it from the original result.

Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1·0 ml 0·5 N sodium hydroxide≡1·366 mg P_2O_5.

4·16 Total phosphoric acid in basic slag and potassic basic slag

4·161 Preparation of the solution

Weigh to the nearest mg about 2·5 g of the sample into a 400 ml beaker, wet the solid thoroughly with 20 to 30 ml water and then add a further 70 ml water with continuous stirring. Warm the mixture and add dropwise with stirring, 10 ml concentrated hydrochloric acid, then 5 ml concentrated nitric acid. Gently boil the solution for 10 minutes, cool, and dilute to 250 ml in a volumetric flask. Mix well. Filter the solution through a dry medium-fine filter paper into a dry beaker, rejecting the first 20 to 30 ml of the filtrate.

4·162 Procedure

Transfer a volume of the solution prepared according to paragraph 4·161, containing less than 70 mg phosphoric acid and preferably about 50 mg to a 500 ml stoppered conical flask marked at 150 ml. Dilute the solution with water to about 100 ml, heat almost to boiling and then add 5 N sodium hydroxide solution dropwise until a faint permanent turbidity or precipitate is formed. Add a few drops of concentrated hydrochloric acid to clear the solution while it is still boiling. Dilute to 150 ml and add 1 g citric acid and then 50 ml of the citric-molybdic acid reagent (B). Boil the solution gently for 3 minutes. From a burette slowly add 25 ml of the quinoline solution with constant swirling throughout, the first few ml being added dropwise, the rest in a slow stream. Again heat to boiling and boil gently for 1 to 2 minutes. Immerse the flask in boiling water for 5 minutes and then cool the flask and its contents to 15 °C in running water.

Filter with suction the contents of the flask on a paper pulp pad and wash the flask, precipitate and filter with successive small washes of cold water until they are free from acid. Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask. If necessary, wipe the funnel with a small piece of damp filter paper to ensure

complete removal of the precipitate and place the paper in the flask. Add water to about but not exceeding 100 ml, stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

Remove the stopper and wash it with water, returning the washings to the flask. Add a measured volume of 0·5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml in excess. Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after addition of 0·5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary.) Add 0·5-1·0 ml of the indicator solution and titrate the excess of sodium hydroxide with the 0·5 N hydrochloric acid until the indicator changes from violet to green-blue and then very sharply to yellow at the end point. Deduct the number of ml of 0·5 N hydrochloric acid used from the number of ml 0·5 N sodium hydroxide to ascertain the volume of 0·5 N sodium hydroxide equivalent to the phosphoric acid.

Carry out a blank determination on all the reagents, omitting only the sample, and using 0·1 N standard alkali and acid instead of 0·5 N for the titration. Calculate the blank in terms of 0·5 N alkali and subtract it from the original result.

Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1 ml 0·5 N sodium hydroxide solution $\equiv$ 1·366 mg P_2O_5.

4·17 Citric acid-soluble phosphoric acid in basic slag and potassic basic slag

4·171 Preparation of the solution

Weigh to the nearest mg about 5 g of the sample and transfer to a stoppered bottle of about 1 litre capacity. Dissolve 10 g citric acid monohydrate in water, dilute to 500 ml and adjust the temperature to 20 °C. Add the solution to the sample in the bottle, shaking so as to avoid the possibility of caking. Shake the bottle continuously for 30 minutes. Pour the whole of the liquid at once on to a large medium-fine filter and collect the filtrate. If the filtrate is not clear, pass it again through the same filter.

4·172 Procedure

Transfer a volume of the solution prepared according to paragraph 4·171 containing less than 70 mg phosphoric acid and preferably about 50 mg to a 500 ml stoppered conical flask marked at 150 ml. Dilute the solution with water to 150 ml, heat almost to boiling and then add 50 ml of the citric-molybdic acid reagent (B). Boil the solution gently for 3 minutes. From a burette slowly add 25 ml of the quinoline solution with constant swirling throughout, the first few ml being added dropwise, the rest in a slow stream. Again heat to boiling and boil gently for 1 to 2 minutes. Immerse the flask in boiling water for 5 minutes and then cool the flask and its contents to 15 °C in running water.

Filter with suction the contents of the flask on a paper pulp pad and wash the flask, precipitate and filter with successive small washes of cold water until they are free from acid. Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask. If necessary wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate, and place the paper in the flask. Add water to about but not exceeding 100 ml, stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

Add a measured volume of 0·5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml in excess. Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after addition of 0·5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary.) Add 0·5-1·0 ml of the indicator

solution and titrate the excess of sodium hydroxide with the 0·5 N hydrochloric acid until the indicator changes from violet to green-blue and then very sharply to yellow at the end point. Deduct the number of ml of 0·5 N hydrochloric acid used from the number of ml 0·5 N sodium hydroxide to ascertain the volume of 0·5 N sodium hydroxide equivalent to the phosphoric acid.

Carry out a blank determination on all the reagents, omitting only the sample, and using 0·1 N standard alkali and acid instead of 0·5 N for the titration. Calculate the blank in terms of 0·5 N alkali and subtract it from the original result.

Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1 ml 0·5 N sodium hydroxide solution≡1·366 mg P_2O_5.

4·2 SPECTROPHOTOMETRIC (VANADIUM PHOSPHOMOLYBDATE) METHOD

4·21 REAGENTS

Calcium oxide—finely ground.

Citric acid—monohydrate.

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 °C 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_2O_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.

4·22 **Total phosphoric acid in fertilisers other than basic slag and potassic basic slag**

4·221 DISSOLUTION OF THE SAMPLE

4·2211 *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 10 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.

4·2212 *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 1 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 10 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 10 minutes. Then cool, transfer to a 500 ml volumetric flask and dilute to the mark. Filter.

4·222 PROCEDURE

4·2221 *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 10 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 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 solutions referred to the 5·0 mg phosphoric acid solution as standard.

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

4·2222 *Analysis of sample*

Successively dilute a portion of the solution prepared according to paragraph 4·2211 or paragraph 4·2212 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 10 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 10 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.

4·23 **Water-soluble phosphoric acid**

4·231 EXTRACTION OF THE SAMPLE

Weigh to the nearest centigram about 10 g of the sample and transfer to a 500 ml volumetric flask; add 400 ml water at 20 °C, and shake the flask continuously for 30 minutes. Dilute the contents to the mark, mix well and filter.

4·232 PROCEDURE

4·2321 *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 10 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 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, 5·5, 5·8, 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 readings against known phosphoric acid content.

4·2322 *Analysis of sample*

To 25 ml of the solution prepared according to paragraph 4·231, add 5 ml concentrated nitric acid; heat to boiling and continue to boil for 10 minutes. Allow to cool, neutralise with N sodium hydroxide solution and then successively dilute until a 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 10 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 10 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.

4·24 **Water-insoluble phosphoric acid**

Determine the water-insoluble phosphoric acid as the difference between the total phosphoric acid determined by the method described in paragraph 4·22 and the water-soluble phosphoric acid determined by the method described in paragraph 4·23.

4·25 **Citric acid-soluble phosphoric acid in fertilisers other than basic slag and potassic basic slag**

4·251 PREPARATION OF THE SOLUTION

Weigh to the nearest mg about 5 g of the sample and transfer to a stoppered bottle of about 1 litre capacity. Dissolve 10 g citric acid monohydrate in water, dilute to 500 ml and adjust the temperature to 20 °C. Add the solution to the sample in the bottle, shaking so as to avoid the possibility of caking. Shake the bottle continuously for 30 minutes. Pour the whole of the liquid at once on to a large medium-fine filter and collect the filtrate. If the filtrate is not clear, pass it again through the same filter.

4·252 PROCEDURE

4·2521 *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 to each flask a quantity of citric acid equal to that in the "final volume of about 25 ml" of the sample under examination quoted in paragraph 4·2522. 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 10 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 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 solutions referred to the 5·0 mg phosphoric acid solution as standard. Plot a calibration graph of scale readings against known phosphoric acid content.

4·2522 *Analysis of sample*

Successively dilute a portion of the solution prepared according to paragraph 4·251 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 10 minutes. At the same time, transfer 25 ml of the standard phosphate solution (at 20 °C) into a second 100 ml volumetric flask, add sufficient citric acid to obtain a concentration in the final 100 ml equal to that of the sample solution. Then add 25 ml of the vanado-molybdate reagent (at 20 °C), dilute to the mark, mix, and allow to stand for 10 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.

4·26 Total phosphoric acid in basic slag and potassic basic slag

4·261 PREPARATION OF THE SOLUTION

Weigh to the nearest mg about 2·5 g of the sample into a 400 ml beaker, wet the solid thoroughly with 20 to 30 ml water and then add a further 70 ml water with continuous stirring. Warm the mixture and add dropwise with stirring, 10 ml concentrated hydrochloric acid, then 5 ml concentrated nitric acid. Gently boil the solution for 10 minutes, cool, transfer to a 250 ml volumetric flask, and dilute to the mark with water. Mix well. Filter the solution through a dry medium-fine filter paper into a dry beaker, rejecting the first 20 to 30 ml of the filtrate.

4·262 PROCEDURE

4·2621 *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 10 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 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 solutions referred to the 5·0 mg phosphoric acid solution as standard.

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

4·2622 *Analysis of sample*

Successively dilute a portion of the solution prepared according to paragraph 4·261 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 temperture 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 10 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 10 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.

4·27 Citric acid-soluble phosphoric acid in basic slag and potassic basic slag

4·271 PREPARATION OF THE SOLUTION

Weigh to the nearest mg about 5 g of the sample and transfer to a stoppered bottle of about 1 litre capacity. Dissolve 10 g citric acid monohydrate in water, dilute to 500 ml and adjust the temperature to 20 °C. Add the solution to the sample in the bottle, shaking so as to avoid the possibility of caking. Shake the bottle continuously for 30 minutes. Pour the whole of the liquid at once on to a large medium-fine filter and collect the filtrate. If the filtrate is not clear, pass it again through the same filter.

4·272 PROCEDURE

4·2721 *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 to each flask a quantity of citric acid equal to that in the "final volume of about 25 ml" of the sample under examination quoted in paragraph 4·2722. 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 10 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 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 solutions referred to the 5·0 mg phosphoric acid solution as standard.

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

4·2722 *Analysis of sample*

Successively dilute a portion of the solution prepared according to paragraph 4·271 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 10 minutes. At the same time, transfer 25 ml of the standard phosphate solution (at 20 °C) into a second 100 ml volumetric flask, add sufficient citric acid to obtain a concentration in the final 100 ml equal to that of the sample solution. Then add 25 ml of the vanado-molybdate reagent (at 20 °C), dilute to the mark, mix, and allow to stand for 10 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.

5· DETERMINATION OF POTASH

For the purposes of the Agriculture Act 1970, Part IV, "potash" means potassium oxide (K_2O).

Potash in all kinds of fertilisers may be determined by the perchloric acid method, or by the potassium chloroplatinate method or, in fertilisers containing not more than 20 per cent of potash, by the flame photometric method.

5·1 PERCHLORIC ACID METHOD

This method depends on the insolubility of potassium perchlorate and the solubility of sodium perchlorate in alcohol, and is applicable in the presence of alkali metals, chlorides and nitrates. Sulphates and ammonium salts must be absent on account of the low solubility of sodium sulphate and of ammonium perchlorate in alcohol. Phosphates must be removed. Methods are given for the elimination of the effect of interfering substances.

5·11 REAGENTS

Alcohol—industrial methylated spirit.

Ammonium carbonate solution—saturated aqueous solution.

Ammonia solution (d = 0·88).

Ammonium oxalate solution—saturated aqueous solution.

Barium chloride solution—Dissolve 100 g barium chloride in water, filter the solution and dilute to 1 litre.

Calcium hydroxide—finely ground.

Hydrochloric acid, concentrated (d = 1·18).

Hydrochloric acid, 25 per cent v/v—Dilute 25 ml concentrated hydrochloric acid with water to 100 ml.

Perchloric acid, 20 per cent w/w.

Wash solution—Add potassium perchlorate to alcohol and shake until a saturated solution is obtained. Keep the solution over solid potassium perchlorate and filter *immediately* before use.

5·12 **Potassium salts free from sulphates and other interfering substances**

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 and 50 ml water and bring the contents to boiling point, breaking down with a stirring rod and crystals or lumps. Dilute the solution to 100 ml and boil gently for a few minutes. Cool the solution to 20 °C and dilute in a volumetric flask to 250 ml or to such a volume that 50 ml of the solution contains from 150 to 200 mg of potash (K_2O). Determine the potash in 50 ml of the solution by precipitating with perchloric acid as described in paragraph 5·15.

5·13 **Potassium salts containing sulphates or other interfering substances**

A method is given in paragraph 5·131 for eliminating the interference caused by the presence of sulphate. If the salts contain phosphates, iron, manganese or substances other than sulphate that interfere with the determination of potash, the method described in paragraph 5·14 should be used.

5·131 Weigh to the nearest mg a portion of the sample, containing from 1·5 to 2·0 g potash, into a 500 ml beaker, add about 300 ml water and 10 ml concentrated hydrochloric acid and heat the solution to boiling. Remove from the heat, allow to cool to between 60 °C and 80 °C. Cautiously add, drop by drop, barium chloride solution in an amount slightly in excess of that previously determined as necessary to ensure the complete precipitation of sulphate. Allow to stand for 10 minutes with occasional stirring. Transfer to a 500 ml volumetric flask, dilute to 500 ml, mix, and filter through a dry filter paper. Take 50 ml of the filtrate and evaporate to dryness in a basin; moisten the residue with concentrated hydrochloric acid, again evaporate to dryness, dissolve the residue with 5-10 ml 25 per cent hydrochloric acid and filter. Determine the potash in the solution by the method described in paragraph 5·15.

5·14 **Potash in guanos and mixed fertilisers**

Weigh to the nearest centigram about 10 g of the sample and, if organic matter is present, gently incinerate at a temperature not exceeding 500 °C. Grind to remove any lumps. Transfer the weighed portion of the sample or the incinerated residue to a 500 ml beaker with a little water and 10 ml concentrated hydrochloric acid and then warm for 10 minutes. Dilute with water to about 300 ml and bring gradually to the boiling point. Add 10 g calcium hydroxide made into a paste with water. Bring the contents again gently to the boiling point, and keep so heated for about half an hour with frequent stirring. Cool to 20 °C, transfer to a 500 ml volumetric flask, dilute to 500 ml and, after thoroughly shaking, filter through a dry filter paper. Transfer 250 ml of the filtrate to another 500 ml volumetric flask, make just acid with hydrochloric acid and heat to boiling point. To the boiling solution cautiously add, drop by drop, barium chloride solution until there is no further precipitation of barium sulphate. Render the contents of the flask alkaline with ammonia solution, and precipitate the calcium and any excess of barium by adding ammonium carbonate solution until no further visible precipitation occurs, followed by the addition of about 1 ml ammonium oxalate solution. Cool to 20 C, transfer to a 500 ml volumetric flask, dilute with water to 500 ml and, after thoroughly shaking, filter through a dry filter paper. Measure 100 ml of the filtrate and evaporate to dryness in a basin. Expel the ammonium salts from the residue

by gently heating the basin over a low flame, being careful to keep the temperature below that of faint redness. Cool the residue, moisten with concentrated hydrochloric acid and again evaporate to dryness. Take up the residue with water and filter. Determine the potash in the solution by precipitation with perchloric acid as described in paragraph 5·15.

5·15 **Precipitation of potash as potassium perchlorate**

Transfer the solution obtained as described in paragraph 5·12, 5·131 or 5·14 into a basin and add about 7 ml perchloric acid solution. Place the basin on a hot plate or sand bath and evaporate the contents until white fumes are copiously evolved. Cool, and dissolve the precipitate in a little hot water. Add about 1 ml perchloric acid solution and again concentrate to the fuming stage. Thoroughly cool the residue in the basin and stir in 20 ml alcohol. Allow the precipitate to cool and settle; then pour the clear liquid through a dry filter paper, draining the precipitate in the basin as completely as possible. Redissolve the precipitate on the paper and that remaining in the basin with hot water, add 2 ml perchloric acid solution to the combined solution and evaporate the whole down to the fuming stage. Cool the residue in the basin and thoroughly stir the contents with 20 ml alcohol. Allow the precipitate to cool and settle and pour the clear liquid through a weighed Gooch or sintered glass crucible, draining the precipitate as completely as possible from the liquid before adding 5 ml of the wash solution. Wash the precipitate by decantation with several similar small portions of the wash solution, pouring the washings through the crucible. Transfer the precipitate to the crucible and wash it well with the wash solution until free from acid. Dry the precipitate at 100 °C and weigh. Regard the precipitate as potassium perchlorate ($KClO_4$) and calculate its equivalent as potash (K_2O) by multiplying its weight by 0·34.

5·2 POTASSIUM CHLOROPLATINATE METHOD

This method depends on the insolubility of potassium chloroplatinate in alcohol. Preliminary treatment is necessary for the removal of calcium, iron and aluminium which are precipitated by ammonium hydroxide and ammonium oxalate. Ammonium salts are then removed by boiling with aqua regia, and potassium chloroplatinate precipitated from the resultant solution.

5·21 REAGENTS

Alcohol—industrial methylated spirit.

Ammonia solution (d = 0·88).

Ammonium oxalate solution—saturated aqueous solution.

Chloroplatinic acid solution—Dissolve a weighed quantity of platinum by gentle heating in a mixture of 4 volumes concentrated hydrochloric acid, 1 volume concentrated nitric acid and 1 volume water in a covered beaker or flask. When the platinum is dissolved, transfer the solution to a basin and evaporate to a syrupy consistency. Add 10 ml 50 per cent hydrochloric acid and evaporate again to a syrup. Repeat the evaporation with 50 per cent hydrochloric acid twice. Dilute the residue with water and filter the solution, thoroughly washing the filter. Combine the filtrate and washings and dilute with water to give a solution containing 0·5 g platinum in 10 ml.

Hydrochloric acid, concentrated (d = 1·18).

Hydrochloric acid, 50 per cent v/v—Dilute 50 ml concentrated hydrochloric acid with water to 100 ml.

Nitric acid, concentrated (d = 1·42).

Wash solution—Dissolve 200 g ammonium chloride in 1 litre of water. Add potassium chloroplatinate to this solution and shake until a saturated solution is obtained. Keep the solution over solid potassium chloroplatinate and filter immediately before use.

5·22 **Potassium salts**

If the salts contain calcium, iron, aluminium or other substances that interfere with the potassium chloroplatinate method, the procedure described in paragraph 5·23 should be used instead of the following procedure.

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 and 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 and dilute in a volumetric flask to 250 ml or to such larger volume that 50 ml of the solution contains from 30 to 100 mg potash (K_2O). Mix the solution and filter through a dry filter paper. Determine the potash in the filtrate by the method described in paragraph 5·25.

5·23 **Potash in mixed fertilisers 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, treat with about 50 ml of water and 5 ml of concentrated hydrochloric acid, and evaporate to dryness on a water bath. To the residue add 125 ml water and 50 ml ammonium oxalate solution. Boil the contents for 30 minutes. If necessary a small quantity of potassium-free anti-foaming agent may be added. Cool the liquid, add a slight excess of ammonia solution and cool to 20 °C; dilute to 250 ml or to such larger volume that 50 ml shall contain from 30 to 100 mg potash (K_2O). Mix the solution and filter through a dry filter paper. Determine the potash in the filtrate by the method described in paragraph 5·25.

5·24 **Potash in mixed fertilisers 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 any lumps and treat with 50 ml water and 10 ml of concentrated hydrochloric acid, and evaporate to dryness on a water bath. Boil the residue for 30 minutes with 125 ml water and 50 ml ammonium oxalate solution. Cool the solution, add a slight excess of ammonia solution, cool to 20 °C and dilute to 500 ml or to such larger volume that 50 ml shall contain from 30 to 100 mg potash (K_2O). Mix the solution and filter through a dry filter paper. Determine the potash in the filtrate by the method described in paragraph 5·25.

5·25 **Precipitation of potash as potassium chloroplatinate**

From the solution obtained as described in paragraph 5·22, 5·23 or 5·24, take 50 ml and place in a digestion flask of capacity about 300 to 500 ml together with 10 ml concentrated nitric acid. A small silica bead or granule weighing about 0·25 g may be added to prevent bumping. (This bead or granule should have been previously tared with a prepared Gooch crucible or sintered glass crucible having an average pore diameter of 5 to 15 microns.) Boil the mixture for 2 minutes, then add 10 ml concentrated hydrochloric acid. Boil the liquid down to approximately 25 ml and add 5 ml concentrated hydrochloric acid followed by chloroplatinic acid solution in excess over that required by the total alkalis present. Boil the mixture down to 10 to 15 ml, rotating the flask occasionally, and then add 5 ml concentrated hydrochloric acid. Reduce the heat and gently boil the mixture down to 3 to 5 ml (depending on the amount of precipitate), rotating the flask frequently near the end of the evaporation. Remove the flask from the heat and swirl to dissolve any soluble residue of the salts on the walls of the flask. Cool and immediately add 25 ml alcohol so that it washes completely the neck of the flask. Chill the flask by swirling under running water and then allow to stand for at least 5 minutes. Filter the clear liquid through the prepared Gooch crucible or sintered glass

crucible, using gentle suction, and draining the liquid as completely as possible from the precipitate. Wash the precipitate several times by decantation with alcohol until the washings are free from platinum; then, with the aid of alcohol, transfer the precipitate, together with the silica bead or granule, if used, to the crucible. Cut off the suction, add 10 ml of the wash solution to the precipitate and allow to stand for 5 minutes; then operate the suction at a low pressure and drain. Wash with a further five consecutive portions of 10 ml each of the wash solution; finally increase the suction and wash the precipitate with alcohol until the filtrate is free from ammonium salts. Dry the crucible and contents at 100°C, weigh, and calculate the weight of the precipitate to its equivalent of potash (K_2O) by multiplying its weight by 0·1938.

5·3 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 radiations lie in the spectral range 766-770 nm. These radiations may be isolated by either a monochromator or the use of a suitable filter.

This method must not be used where the potash content of the material being analysed exceeds 20 per cent by weight.

5·31 REAGENTS

Ammonia solution, 30 *per cent 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 1 hour at 105°C and dilute to 1 litre.

Potassium dihydrogen phosphate solution (standard potash solution)—Dilute 50 ml stock solution to 1 litre with water. This solution contains 100 ppm potash (K_2O).

5·32 **Potassium salts**

If the salts contain calcium, iron, aluminium or other interfering substances, the procedure described in paragraph 5·33 should be used instead of the following procedure.

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 and 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 5·35.

5·33 **Potash in mixed fertilisers containing little or no organic matter**

Weigh to the nearest mg about 2·5g 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 30 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 and determine the potash in the filtrate by the method described in paragraph 5·35.

5·34 **Potash in mixed fertilisers 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 any lumps, add 50 ml of water, 10 ml of concentrated hydrochloric acid, and evaporate to dryness on a water bath. Boil the residue for 30 minutes, with 125 ml water and 50 ml 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 5·35.

5·35 **Determination of potash by flame photometry**

5·351 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 sensititvity of the flame photometer has not changed.

5·352 ANALYSIS OF SAMPLE

Reset the instrument at 100 scale divisions (full scale deflection) with 20 ppm potash solution. Spray the diluted fertiliser solution prepared in accordance with paragraph 5·32, 5·33 or 5·34 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 fertiliser 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.

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 PREPARATION OF THE SAMPLE

Prepare a portion of at least 50 g of the sample for analysis as described in paragraph 1·11. When a small part of the sample has been used for a sieving test as described in paragraph 17, grind the remainder of the dried portion to pass through a sieve of approximately ¼″ square apertures, and quarter down

until about 100 g remains. Grind this portion for analysis as described in paragraph 1·11. Where the greater part of the sample has been used for the sieving test, mix the various fractions obtained in the test together and grind until the whole passes a sieve of approximately $\frac{1}{8}''$ square apertures, quarter down until about 100 g remains. Grind this portion for analysis 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 5 minutes. Cool the mixture, add 2 or 3 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). Correct the neutralising value for the moisture lost if the sample has been dried for sieving purposes. Express the neutralising value as a percentage of the original.

7· DETERMINATION OF MAGNESIUM IN LIME AND GROUND LIMESTONE

7·1 REAGENTS

Ammonia solution, 25 per cent v/v—Dilute 25 ml concentrated ammonia solution (d=0·88) 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 1 week.

Buffer solution—Dissolve 6·75 g ammonium chloride, 62 mg magnesium sulphate ($MgSO_4 . 7H_2O$), 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 120 ml 0·5 N hydrochloric acid and dilute to 1 litre.

EDTA solution, 0·025 M—Dissolve 10 g disodium ethylenediamine-tetra-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 ($CaCO_3$).

Hydrochloric acid, 0·5 N.

Hydrogen peroxide solution, 6 per cent w/v (20 volumes).

Mordant Black 11 indicator (colour index No. 14645)—Mix 200 mg Mordant Black 11 and 50 g sodium chloride uniformly together and grind to pass through a sieve having apertures of about 0·3 mm 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.

*British Standard Test Sieve, Mesh No. 52 is suitable (British Standard for Test Sieves 410:1962).

7·2 PROCEDURE

Weigh to the nearest mg about 1 g of 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 per cent 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 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 washings 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 per cent 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 THIOCYANATE IN AMMONIACAL GAS LIQUOR; NITROGENOUS GAS LIQUOR; GAS LIQUOR

8·1 REAGENTS

Ammonium ferric sulphate solution—Saturated aqueous solution of ammonium ferric sulphate.

Copper sulphate solution—Dissolve 10 g copper sulphate in water and dilute to 100 ml.

Lead carbonate.

Nitric acid, 50 per cent v/v—Dilute 50 ml concentrated nitric acid (d 1·42) with water to 100 ml.

Potassium thiocyanate, 0·1 N.

Silver nitrate, 0·1 N.

Sodium hydroxide solution—Dissolve 4 g sodium hydroxide (free from chloride) in water and dilute to 100 ml.

Sodium metabisulphite (or potassium metabisulphite) solution—Saturated aqueous solution.

Sulphuric acid, 10 per cent v/v—To 50 ml water cautiously add 10 ml concentrated sulphuric acid. Cool and dilute to 100 ml.

8·2 PROCEDURE

Place 25 or 50 ml of the liquor in a 100 ml beaker and add 2 g lead carbonate. Stir well and allow to stand for ten minutes (in order to remove sulphide). Filter the solution into a 150 ml beaker, washing the beaker and the filter twice with distilled water. Slightly acidify the filtrate with sulphuric acid solution, warm to about 40 °C and add a few drops of ammonium ferric sulphate solution to clarify the liquor and remove any ferrocyanide which may be present. Filter the solution through paper pulp with the aid of a suction pump and wash the beaker and the filter with water. To the filtrate contained in a 250 ml flask add 10 drops of sodium metabisulphite (or potassium metabisulphite) solution, and heat the mixture to about 60 °C. Add an excess of copper sulphate solution and continue the heating to incipient boiling. Allow to stand from five to ten minutes with occasional agitation. Filter and well wash the beaker and the filter with hot water until the washings remain colourless upon the addition of a drop of ferrocyanide solution. Pierce the filter paper and wash the residue back into the original flask, and finally wash with 25 ml sodium hydroxide solution. Warm the solution to about 50 °C to decompose the cuprous salt and add a few drops of ammonium ferric sulphate solution to promote coagulation. Filter the solution through paper pulp, well wash the flask and the filter with water. Acidify the filtrate with 5 ml nitric acid solution, add two drops of ammonium ferric sulphate solution and titrate with 0·1 N silver nitrate.

$$\text{Thiocyanate as CNS in g per 100 ml} = \frac{0{\cdot}58 \times \text{ml } 0{\cdot}1 \text{ N } AgNO_3}{\text{ml liquor taken}}$$

9· DETERMINATION OF BIURET

9·1 REAGENTS

Alkaline tartrate solution—Dissolve 40 g sodium hydroxide in 500 ml water, cool and add 50 g potassium sodium tartrate. Dilute to 1 litre and allow to stand for 24 hours before use.

Biuret standard solution—Dissolve 0·100 g biuret in carbon dioxide-free water and dilute to 100 ml. 1 ml ≡ 1 mg biuret. The sample of biuret, when taken through the procedure for the preparation of the standard curve, should have an E (1 per cent 1 cm) not less than 2·5.

Copper sulphate solution—Dissolve 15 g copper sulphate $CuSO_4.5H_2O$ in carbon dioxide-free water and make up to 1 litre.

Methanol.

Methyl red indicator solution—Dissolve 0·025 g methyl red in 5 ml 90 per cent industrial methylated spirit with the aid of 0·5 ml 0·1 N sodium hydroxide solution. Dilute to 250 ml with 50 per cent industrial methylated spirit. If desired, a screened methyl red indicator may be used.

Sulphuric acid, 0·1 *N*.

9·2 PROCEDURE

Place 10 g of the sample in a small glass evaporating dish, add 30 ml methanol and evaporate to dryness on a water bath. Treat the residue with 50 ml water and digest at 50 °C for half an hour. Filter and wash into a 250 ml flask and dilute to volume with carbon dioxide-free water. Transfer a 50 ml aliquot to a 100 ml volumetric flask, add 1 drop methyl red indicator solution and neutralise with 0·1 N sulphuric acid to a pink colour. Add with swirling 20 ml alkaline tartrate solution and then 20 ml copper sulphate solution.

Dilute to volume, shake for 10 seconds and place in a water bath at 30 ± 5 °C for 15 minutes. Prepare a reagent blank solution. Determine the extinction of each solution against the blank at 555 nm using 4 cm cells.

Calculate the biuret content of the sample by reference to a calibration graph prepared at the same time as the test sample.

Establish the calibration graph as follows:—

Transfer 5, 10, 20, 30, 40, 50 ml aliquots of standard biruet solution to 100 ml volumetric flasks. Adjust the volumes to about 50 ml with carbon dioxide-free water, and proceed as described above commencing at "add 1 drop methyl red indicator solution.....". Construct a graph relating the extinctions of the solutions to the milligrams of biuret.

10· DETERMINATION OF BORON

For levels above 1000 ppm, boron is determined by titration as boric acid and for levels up to 1000 ppm by the carmine spectrophotometric method.

10·1 TITRIMETRIC METHOD

10·11 REAGENTS

Calcium oxide.

Hydrochloric acid, 50 per cent 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.

Methyl red indicator solution—Dissolve 0·025 g methyl red in 5 ml 90 per cent industrial methylated spirit with the aid of 0·5 ml 0·1 N sodium hydroxide. Dilute to 250 ml with 50 per cent industrial methylated spirit.

Phenolphthalein indicator solution—Dissolve 0·25 g phenolphthalein in 150 ml industrial methylated spirit and dilute with water to 250 ml.

Sodium carbonate.

Sodium hydroxide, 0·5 N.

Sodium hydroxide, 0·05 N—Prepare from a 50 per cent 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.

10·12 DISSOLUTION OF THE SAMPLE

10·121 *In the absence of organic matter*

Weigh to the nearest mg about 2 g of the sample, if it contains 0·5 per cent or less of boron, and 1 g if it contains from 0·5-1·0 per cent 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 per cent hydrochloric acid.

10·122 *In the presence of organic matter*

Weigh to the nearest mg about 2 g of the sample, if it contains 0·5 per cent or less of boron, and 1 g if it contains from 0·5-1·0 per cent of boron. Place in a silica dish, add 0·2 g calcium oxide for each 1 g of the sample, moisten with water, mix thoroughly, evaporate the mixture to dryness and ignite gently in a muffle furnace at 450 °C. Allow the ashing to proceed for about three hours. Cool. Moisten with 10 ml 50 per cent hydrochloric acid, warm on a water bath for 15 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.

10·13 PROCEDURE

To the solution prepared in accordance with paragraph 10·121 or 10·122, add 20 ml lead nitrate solution for each 12 per cent P_2O_5 in the sample if 2 g of the sample have been used and 10 ml lead solution for each 12 per cent 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 solution and then make just acid with 0·5 N hydrochloric acid. Cover with a watch glass and boil gently for 5 minutes to expel any remaining carbon dioxide. Cool rapidly.

Place the electrodes of a 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 0·05 N sodium hydroxide solution to a 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 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 $= \frac{0{\cdot}1082\ (x - 0{\cdot}1)}{\text{Weight of sample taken}}$

10·2 SPECTROPHOTOMETRIC (CARMINE) METHOD

10·21 REAGENTS

Boric acid (*stock boron solution*)—Dissolve 1·905 g boric acid in water and dilute to 1000 ml at 20 °C. 1 ml≡0·333 mg boron.

Boric acid (*standard boron solutions*)—Dilute 10 ml stock solution with water to 100 ml at 20 °C. 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 µg of boron per 3 ml.

Calcium oxide.

Carminic acid solution—Dissolve 0·025 g carminic acid in concentrated sulphuric acid and dilute to 100 ml with concentrated sulphuric acid.

Hydrochloric acid, 20 *per cent v/v*—Dilute 20 ml concentrated hydrochloric acid (d=1·18) with water to 100 ml.

Sulphuric acid, concentrated (d=1·84).

10·22 DISSOLUTION OF SAMPLE

This procedure should be followed even in the absence of organic matter in the fertiliser. Weigh to the nearest mg about 5 g of the sample. Transfer to a silica dish and add 1 g calcium oxide, moisten with water, mix thoroughly, evaporate the sample to dryness, and ignite gently in a muffle furnace at 450°C. Allow ashing to proceed for about 3 hours. Cool and add 20 per cent hydrochloric acid solution until the mixture is just acid. Add 5 ml 20 per cent hydrochloric acid in excess and digest the mixture at 70°C for 15 minutes. Cool and filter the contents of the dish into a suitable volumetric flask, making up to the mark with washings. Dilute an aliquot of this solution so that 3 ml contain between 5 and 25 µg of boron.

10·23 PROCEDURE

Transfer 3 ml to a small flask. Add cautiously 15 ml concentrated sulphuric acid. Swirl the flask and add 10 ml carminic acid solution. Cool the flask rapidly to room temperature, mix well and allow to stand for exactly 2 hours. Measure the extinction of the coloured complex at 625 nm using a 1 cm cell, and against a blank which has been taken through all the stages of the determination. Read from a previously prepared calibration graph the number of micrograms of boron corresponding to the observed extinction, and calculate the boron content of the sample.

Establish the calibration graph as follows:

Pipette 3 ml of each standard solution into a series of small flasks and proceed as described above commencing at "Add cautiously 15 ml.....". Measure the extinctions of the solutions, and construct a graph relating the extinctions to the number of micrograms of boron.

11· DETERMINATION OF COBALT

11·1 REAGENTS

Ammonium cobaltous sulphate, $(NH_4)_2Co(SO_4)_2.6H_2O$, (*stock cobalt solution*)—Dissolve 0·670 g ammonium cobaltous sulphate in water and dilute to 100 ml at 20°C.

Ammonium cobaltous sulphate (*standard cobalt solution*)—Dilute 1 ml stock solution to 1000 ml with water at 20°C immediately before use. 1 ml = 1 µg cobalt.

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

Hydrochloric acid, 2 *N*.

Hydrogen peroxide solution, 3 *per cent w/v* (10 volumes).

Nitric acid, 30 *per cent v/v*—Dilute 30 ml concentrated nitric acid (d 1·42) with water to 100 ml.

2-*Nitroso*-1-*naphthol reagent*—Dissolve 1 g 2-nitroso-1-naphthol in 100 ml glacial acetic acid, and add 1 g activated carbon. Shake the solution before use, and filter the required amount.

Sodium citrate solution—Dissolve 40 g sodium citrate in water and dilute to 100 ml.

Sodium hydroxide, 2 *N*.

Sodium sulphate, anhydrous.

Toluene, redistilled.

11·2 DISSOLUTION OF THE SAMPLE

11·21 In the absence of organic matter

Weigh to the nearest mg about 5 g of the sample, and transfer into a 100 ml beaker, add 10 ml 50 per cent v/v hydrochloric acid, and evaporate to dryness on a water bath. Extract the soluble salts with three successive 10 ml portions of boiling 2 N hydrochloric acid, decanting the solution each time through the same Whatman No. 541 (or equivalent) filter paper into a 50 ml volumetric flask. Dilute the combined extracts to the mark with distilled water, washing the filter paper in the process.

11·22 In the presence of organic matter

Weigh to the nearest mg about 5 g of the sample, and transfer 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 ash 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 per cent 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. 541 (or equivalent) filter paper into a 50 ml volumetric flask. Then add 5 ml 50 per cent v/v hydrochloric acid and about 5 ml 30 per cent v/v nitric acid to the residue in the basin, and take the mixture to dryness on a hot-plate at low heat. Finally, add a further 10 ml of boiling 2 N hydrochloric acid to the residue and filter the 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.

11·3 PROCEDURE

Transfer a suitable aliquot of the solution prepared in accordance with paragraph 11·2 to a 100 ml beaker, add 15 ml sodium citrate solution, and dilute to approximately 50 ml with distilled water. Adjust the pH to between 3 and 4 by the addition of 2 N hydrochloric acid and 2 N sodium hydroxide, using pH test paper (a precipitate of ferric hydroxide may form, but this can be dissolved by heating the solution), and cool to room temperature. Add 10 ml 3 per cent hydrogen peroxide solution and, after 5 minutes, 1 ml of 2-nitroso-1-naphthol reagent, heat to about 90°C, and then allow to stand for 30 minutes at room temperature.

Transfer the solution to a 125 ml separating funnel, add exactly 10 ml toluene, shake vigorously for 2 minutes, and discard the lower aqueous phase. To the toluene extract add 20 ml 2 N hydrochloric acid, shake for 1 minute, and run off and discard the lower aqueous phase. Add 20 ml 2 N sodium hydroxide, shake for 1 minute, and again run off and discard the lower aqueous phase. Repeat the treatment of the toluene extract with a further 20 ml 2 N sodium hydroxide. Finally run off the toluene solution through a little anhydrous sodium sulphate and a cotton-wool plug into a glass-stoppered tube.

Carry out a blank determination by repeating the procedure, omitting only the sample.

Measure the extinctions of the test and blank solutions at a wavelength of 367 nm, using a 1 cm cell and toluene in the comparison cell. Read the number of micrograms of cobalt equivalent to the observed extinctions of the test and blank solutions from a previously prepared calibration graph. Determine the amount of cobalt in the sample from the difference between the test and blank solutions.

Establish the calibration graph as follows:

Measure amounts of standard cobalt solution corresponding to 0, 3, 6, 9, 12, 15 µg 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 of the solutions, and construct a graph relating the extinctions to the number of micrograms of cobalt.

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 tetrachloride, redistilled.

Copper sulphate, stock solution—Dissolve 0·393 g copper sulphate, $CuSO_4.5H_2O$, in 100 ml 2 N sulphuric acid and dilute to 1 litre at 20°C with distilled water.

Copper sulphate standard solution—Dilute 5 ml stock solution to 250 ml with 2 N sulphuric acid at 20°C immediately before use. 1 ml≡2 µg 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 diethyldithiocarbamate solution—Dissolve 1 g 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 7 days.

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.

12·12 DISSOLUTION OF THE SAMPLE

Prepare a solution of the sample as described in paragraph 11·2.

12·13 PROCEDURE

Transfer to a separating funnel a suitable aliquot (containing not more than 50 µg of copper) of the solution prepared in accordance with paragraph 11·2. 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 2 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:—

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.

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, $CuSO_4.5H_2O$, 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 11·2, 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 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 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 IRON

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

13·1 o-PHENANTHROLINE METHOD

13·11 REAGENTS

Ammonium ferric sulphate solution (stock iron solution)—Dissolve 0·863 g ammonium ferric sulphate, $Fe_2(SO_4)_3.(NH_4)_2SO_4.24H_2O$, in water containing 2 ml perchloric acid, and dilute to 100 ml at 20°C.

Ammonium ferric sulphate solution (standard iron solution)—Dilute 10 ml stock solution to 100 ml with water at 20°C immediately before use. 1 ml≡100 µg iron.

Bromophenol blue indicator solution—Dissolve 0·4 g bromophenol blue in 95 per cent ethanol and dilute to 100 ml.

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

Hydrochloric acid, 20 per cent v/v—Dilute 20 ml concentrated hydrochloric acid (d = 1·18) with water to 100 ml.

Nitric acid, 30 per cent 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 per cent ethanol and dilute to 100 ml.

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

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

13·12 DISSOLUTION OF THE SAMPLE

Prepare a solution of the sample as described in paragraph 11·2.

13·13 PROCEDURE

Transfer a suitable aliquot of the solution, prepared in accordance with paragraph 11·2, 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 quinol 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 nm, 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 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 µg of iron into a series of 100 ml volumetric flasks. To each add 50 ml 20 per cent 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.

13·2 TITRIMETRIC METHOD

13·21 REAGENTS

Hydrochloric acid, concentrated (d = 1·18).

Hydrochloric acid, 30 per cent 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 per cent v/v hydrochloric acid and dilute to 100 ml with 30 per cent v/v hydrochloric acid. This solution should be prepared immediately before use.

Stannous chloride solution, dilute—Dilute 5 ml stannous chloride solution with 30 per cent v/v hydrochloric acid to 50 ml.

Sulphuric acid, 16 *per cent v/v*—To 50 ml water cautiously add 16 ml concentrated sulphuric acid (d 1·84). Cool and dilute to 100 ml.

13·22 DISSOLUTION OF THE SAMPLE

Prepare a solution of the sample as described in paragraph 11·2.

13·23 PROCEDURE

Transfer a suitable aliquot of the solution, prepared in accordance with paragraph 11·2, 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 and add the stannous chloride solution dropwise 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 too much stannous chloride has been added. In either case the solution must be discarded). Add 200 ml water, 10 ml 16 per cent 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.

14· DETERMINATION OF MAGNESIUM

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

14·1 PYROPHOSPHATE METHOD

14·11 REAGENTS

Ammonia solution, (d 0·88).

Ammonia solution, 5 per cent v/v—Dilute 5 ml concentrated ammonia solution (d 0·88) with water to 100 ml.

Ammonium oxalate solution—saturated aqueous solution.

Ammonium phosphate solution—Dissolve 20 g diammonium hydrogen phosphate, $(NH_4)_2\ HPO_4$, in water and dilute to 100 ml.

Calcium wash solution—Dissolve 1 g oxalic acid, $(COOH)_2.2H_2O$ and 2 g ammonium oxalate, in water and dilute to 1000 ml.

Citric acid, monohydrate.

Hydrochloric acid, concentrated (d=1·18).

Hydrochloric acid, 20 per cent 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 per cent industrial methylated spirit with the aid of 0·5 ml 0·1 N sodium hydroxide. Dilute to 250 ml with 50 per cent industrial methylated spirit.

Oxalic acid solution—Dissolve 10 g oxalic acid, $(COOH)_2.2H_2O$, in water and dilute to 100 ml.

14·12 DISSOLUTION OF THE SAMPLE

Prepare a solution of the sample as described in paragraph 11·2.

14·13 PROCEDURE

Transfer a suitable aliquot (containing approximately 50 mg magnesium) of the solution, prepared in accordance with paragraph 11·2, to a 500 ml beaker, and add 5 per cent 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 (4 drops) of methyl red indicator solution. Neutralise with 5 per cent 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 1 to 2 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 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 4 hours or, preferably, overnight. Filter through a No. 4 sintered-silica crucible, and wash the residue with cold 5 per cent 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 $\frac{1}{2}$ to 1 hour. Allow the crucible to cool in a desiccator, and weigh. Calculate the weight of the precipitate to its equivalent of magnesium by multiplying its weight by 0·2184.

14·2 ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

14·21 APPARATUS

Atomic absorption spectrophotometer.

Magnesium hollow-cathode lamp.

14·22 REAGENTS

Hydrochloric acid, 0·5 N.

Magnesium sulphate standard solution—Dissolve 1·013 g magnesium sulphate, $MgSO_4.7H_2O$, 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, $SrCl_2.6H_2O$, in 0·5 N hydrochloric acid and dilute to 100 ml with 0·5 N hydrochloric acid.

14·23 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 accordance with paragraph 11·2, with 0·5 N hydrochloric acid to produce a standard volume of solution containing between 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 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.

15· DETERMINATION OF MANGANESE

15·1 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 per cent v/v sulphuric acid and dilute with water to 1 litre at 20°C.

Potassium permanganate (standard manganese solution)—Dilute 10 ml stock solution to 100 ml with water at 20°C immediately before use. 1 ml ≡ 10 µg manganese.

Sulphuric acid, concentrated (d = 1·84).

Sulphuric acid, 25 per cent v/v—To 50 ml water cautiously add 25 ml concentrated sulphuric acid (d = 1·84). Cool and dilute to 100 ml.

15·2 DISSOLUTION OF THE SAMPLE

Prepare a solution of the sample as described in paragraph 11·2.

15·3 PROCEDURE

Transfer to a small beaker a suitable aliquot (containing not more than 70 µg of manganese) of the solution prepared in accordance with paragraph 11·2. 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 at 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 waterbath for 30 minutes. Cool, and adjust the volume to the mark with water. Carry out a blank determination on all the reagents used.

Measure the extinctions 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 amounts of the standard manganese solution corresponding to 0, 10, 20, 30, 40, 50, 60, 70 µg 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 extinctions of the solutions, and construct a graph relating the extinctions to the number of micrograms of manganese.

16· DETERMINATION OF MOLYBDENUM

16·1 REAGENTS

Ammonium molybdate (*stock molybdenum solution*)—Dissolve 1·840 g ammonium molybdate, $(NH_4)_6Mo_7O_{24}.4H_2O$, in water and dilute to 1000 ml at 20°C.

Ammonium molybdate (*standard molybdenum solution*)—Dilute 1 ml stock solution to 1000 ml with water at 20°C immediately before use. 1 ml≡1 µg 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 thiocyanate 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-ol.

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.

16·2 DISSOLUTION OF THE SAMPLE

Prepare a solution of the sample as described in paragraph 11·2.

16·3 PROCEDURE

Transfer a suitable aliquot of the solution prepared in accordance with paragraph 11·2 to a 125 ml separating funnel, add 1 ml ammonium ferrous sulphate solution and sufficient N hydrochloric acid to bring the volume to 50 ml (see Note), then add 1 ml potassium thiocyanate 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 15 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 μg 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 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.

17· DETERMINATION OF FINENESS

17·1 METHOD OF SIEVING

Use the method appropriate to the size of the sieve which is prescribed or declared, or referred to in schedule 4.

17·11 Sieves having apertures of less than $\frac{1}{8}$″ square

Thoroughly mix the sample and quarter down until a portion of about 100 g is obtained. Heat this portion at 100 C until dry, and thoroughly mix. Weigh to the nearest centigram about 20 g and transfer to the sieve with the lower receiver attached. Continue as in paragraph 17·2. In the case of potassic basic slag continue as in paragraph 17·21.

17·12 Sieves having apertures of $\frac{1}{8}$″ square or more but less than $\frac{1}{4}$″ square

Oven dry the sample, at 100 C for 24 hours, and thoroughly mix. Weigh to the nearest centigram about 200 g and transfer to the sieve with the lower receiver attached. Continue as in paragraph 17·2.

17·13 Sieves having apertures of $\frac{1}{4}$″ square or more

If the sample appears moist or damp, oven dry at 100 C for 24 hours, but if the sample appears dry, heating is not necessary. Thoroughly mix the sample and weigh to the nearest centigram about 500 g and transfer to the sieve with the lower receiver attached. Continue as in paragraph 17·2.

17·2 Shake the sieve for 5 mins., frequently tapping the side. Disintegrate soft lumps such as can be caused to crumble by the application of the fibres of a soft brush, taking care that the hard part of the brush does not make contact with the sieve, and that the brush is not used to brush particles through the sieve. Brush out the powder in the lower receiver and weigh. Replace the receiver and repeat the shaking and tapping procedure for 2 mins. Add the powder in the receiver to the first portion and weigh. Repeat the process until not more than 40 mg passes through the sieve during 2 minutes.

17·21 Weigh to the nearest centigram 20 g of the dry sample and transfer to a 0·5 mm sieve with the lower receiver attached. Shake the sieve for 5 minutes, frequently tapping the sides. Disintegrate soft lumps such as can be caused to crumble by the application of a soft brush, taking care that the hard part of the brush does not make contact with the sieve, and that the brush is not used to brush particles through the sieve. Transfer the finer portion from the container into a 500 ml beaker, and add 200 ml of previously boiled water, stir, and then filter through a weighed, sintered glass crucible. Thoroughly wash the residue with water, dry and reweigh the crucible. Calculate the weight of slag in the mixture with a particle size less than 0·5 mm. (A).

Weigh to the nearest centigram 20 g of the dry sample and transfer to a 500 ml conical flask. Add 200 ml of previously boiled water, and shake for 30 minutes. Filter through a weighed, sintered glass crucible, wash the residue thoroughly with water, dry and reweigh the crucible. Calculate the total weight of slag in the mixture. (B).

Calculate the fineness of the slag by expressing A as a percentage of B.

17·3 CALCULATION

Calculate the fineness by expressing the weight of the material passing through the sieve as a percentage of the weight of the portion of the dried, or undried as the case may be, sample taken for sieving.

NOTE: Where a neutralising value is to be determined, the loss in weight on drying at 100 °C must be determined and due allowance for the moisture made after the determination of neutralising value.

SCHEDULE 7

METHODS OF ANALYSIS OF FEEDING STUFFS

(Sections 68(5), 69(5), 70(4), 71(3), 73(1), 75(1), 77(4), 78(6) and 79(3) and Regulation 15)

(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 sixth 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 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" mean purified water as defined in the British Pharmacopoeia. All reagents used should be of analytical quality.

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 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 If the sample is in a fine condition and passes through a sieve having apertures of about 1 mm square*‡, mix thoroughly and transfer a portion of not less than 100 g to a non-corrodible container provided with an air-tight closure.

1·2 If the sample does not wholly pass through a sieve having apertures of about 1 mm square*‡, and wholly passes through a sieve having apertures from 2 to 3 mm square†‡, mix thoroughly and further grind a portion of not less than 100 g to pass through a sieve having apertures of about 1 mm square*‡. Transfer the portion so prepared to a non-corrodible container provided with an air-tight closure.

1·3 If the sample is in coarse condition as, for example, pieces of broken cake, carefully grind until the whole passes through a sieve having apertures of from 2 to 3 mm square†‡. Mix thoroughly and further grind a portion of not less than 100 g to pass through a sieve having apertures of about 1 mm square*‡. Transfer the portion so prepared to a non-corrodible container provided with an air-tight closure.

1·4 If the sample is appreciably moist or if for any reason the processes 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.

1·5 If, because of its physical condition, grinding is difficult, take a portion 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. Dry the sample until grinding with an iron mortar and pestle, or by other means, enables the sample to be passed completely through a sieve having apertures of about 1 mm square*‡. 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.

1·6 Treat by any other suitable means materials which cannot conveniently be ground or passed through a sieve.

2· DETERMINATION OF MOISTURE

Weight 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.

---

*British Standard Test Sieve, Mesh No. 16 is suitable
†British Standard Test Sieve, Mesh Nos. 8, 7 or 6 is suitable
} British Standard for Test Sieves 410:1962
‡Where an analysis for copper has to be carried out, a stainless steel sieve should be used. (See para. 12·12)

3· DETERMINATION OF OIL

3·1 REAGENT

Light petroleum—boiling point 40-60 °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 4 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 quantity 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 the 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 °C for 2 hours, cool and weigh. Reheat at 100 °C for 30 minutes, cool and again weigh. This 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 10 per cent or where there is reason to believe that the whole of the oil will not be removed from the feeding stuff in a 5 hours 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.

3·221 REAGENTS

Ammonia solution (d=0·88).
Diethyl ether, peroxide free.
Industrial methylated spirit, 95 *per cent 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 90 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 90 seconds. Allow to stand for 15 minutes, or until the solvent layer separates cleanly. Remove the stopper, insert a siphon 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 diethyl 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

*British Standard 1743:1968, fig 1 is suitable.

vigorously for 90 seconds, cool, remove the stopper, add 15 ml light petroleum and again shake for 90 seconds. Allow to stand for 15 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 followed by 15 ml light petroleum in the same way, collecting the solvent in the flask. Remove the solvent from the flask by evaporation and dry the flask lying on its side at 100 °C for 2 hours; cool in a desiccator and weigh. Reheat at 100 °C for 30 minutes, cool and weigh. The second weight should not differ by more than 2 mg from the first weight. 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 1 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

Methyl red indicator solution—Dissolve 0·025 g methyl red in 5 ml 90 per cent industrial methylated spirit with the aid of 0·5 ml 0·1 N sodium hydroxide solution. Dilute to 250 ml with 50 per cent industrial methylated spirit. If desired a screened methyl red indicator may be used.

Mercuric oxide.

Paraffin wax.

Sodium hydroxide, 0·2 *N*—carbonate free.

Sodium hydroxide solution, 50 *per cent w/v*—Dissolve 500 g sodium hydroxide in water and dilute to 1 litre.

Sodium sulphate or potassium sulphate—anhydrous.

Sodium thiosulphate.

Sucrose.

Sulphuric acid, concentrated (d = 1·84)—nitrogen free.

Sulphuric acid, (or hydrochloric acid), 0·2 *N*.

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 mercuric oxide, 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 2 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 per cent sodium hydroxide solution to neutralise the acid and 10 ml in excess; then add 5 g sodium thiosulphate, mix well and connect immediately to a distillation apparatus. Distil into an appropriate volume of 0·2 N acid, controlling the rate of distillation so that not less than 150 ml distil in 30 minutes. Titrate the excess of acid with 0·2 N sodium hydroxide solution, using methyl red solution as an indicator. Carry out a blank test on the reagents using 2 g sucrose in place of the sample. Express the result in terms of nitrogen. 1 ml 0·2 N acid ≡ 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 3·52, 3·53, 3·6 or 3·7 of Schedule 6 or paragraph 5 of this Schedule, and the amount so obtained deducted from the total nitrogen content. In the case of compound feeding stuffs containing urea, the deduction of the nitrogen content of urea is unnecessary for the calculation of the protein content.

5· DETERMINATION OF UREA NITROGEN

5·1 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 2 g 4-dimethylaminobenzaldehyde in 10 ml concentrated hydrochloric acid and dilute to 100 ml with propan-2-ol.

Hydrochloric acid, 0·02 *N*.

Sodium acetate solution—Dissolve 136 g sodium acetate ($CH_3COONa.3H_2O$) in water and dilute to 1 litre.

Urea standard solution—Dissolve 1 g urea in water and dilute to 100 ml.

5·2 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 30 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 15 minutes. Add 5 ml Carrez solution 1, followed by 5 ml Carrez solution 2, mixing well between additions. Dilute to 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 10 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 × 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 solutions, 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 up to 5 g. The final solution after filtering should be colourless.

6· DETERMINATION OF PHOSPHORIC ACID

For the purposes of the Agriculture Act 1970, Part IV, "phosphoric acid" means P_2O_5 (molecular weight 142·04).

Phosphoric acid may be determined by the quinolinium phosphomolybdate method or, alternatively, by the spectrophotometric (vanadium phosphomolybdate) method.

The quinolinium phosphomolybdate method depends on the precipitation of quinolinium phosphomolybdate under carefully controlled conditions. The spectrophotometric method compares the amount of light transmitted by the solution to that by a solution of known phosphoric acid content.

6·1 QUINOLINIUM PHOSPHOMOLYBDATE METHOD

6·11 REAGENTS

Calcium oxide—finely ground.

Citric-molybdic acid solution—Stir 54 g molybdenum trioxide (MoO_3) with 200 ml water; add 11 g sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdenum trioxide dissolves. Dissolve 60 g citric acid in about 250 to 300 ml water and add 140 ml concentrated hydrochloric acid. Pour the molybdate solution into the acid solution, which is stirred throughout the addition. Then cool and, if necessary, filter the solution through a paper pulp pad. Dilute the solution to 1 litre. If the solution is slightly green or blue in colour, add dropwise a dilute (0·5 or 1·0 per cent) solution of potassium bromate until the colour is discharged. This reagent should be kept in the dark.

Hydrochloric acid, concentrated (d = 1·18).

Hydrochloric acid, 25 per cent v/v—Dilute 25 ml concentrated hydrochloric acid with water to 100 ml.

Hydrochloric acid, 0·5 *N*.

Hydrochloric acid, 0·1 *N*.

Indicator solution—Mix 3 volumes of thymol blue solution and 2 volumes of phenolphthalein solution prepared as follows:—

Thymol blue solution—Dissolve 0·25 g thymol blue in 5·5 ml 0·1 N sodium hydroxide solution and 125 ml industrial methylated spirit. Dilute with water to 250 ml.

Phenolphthalein solution—Dissolve 0·25 g phenolphthalein in 150 ml industrial methylated spirit and dilute with water to 250 ml.

Nitric acid, concentrated (d = 1·42).

Quinoline solution—Measure 60 ml concentrated hydrochloric acid and 300 to 400 ml water into a 1 litre beaker and warm to 70-80 °C. Pour 50 ml quinoline in a thin stream into the diluted acid, whilst stirring. When the quinoline has dissolved, cool the solution, dilute to 1 litre and, if necessary, filter through a paper pulp filter.

Sodium hydroxide, 5 *N*.

Sodium hydroxide, 0·5 *N*—carbonate free.

Sodium hydroxide, 0·1 *N*—carbonate free.

Surface active agent—0·5 per cent solution of sodium dodecylbenzenesulphonate is suitable.

6·12 DISSOLUTION OF THE SAMPLE

Weigh to the nearest mg about 5 g of the sample into a capsule or dish; add 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 concentrated nitric acid. Heat to incipient boiling and keep at this temperature for 10 minutes. Dilute with about 10 ml 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 a 250 ml volumetric flask, dilute to the mark, mix well and filter. Discard the first 10 or 20 ml of the filtrate.

6·13 PROCEDURE

Transfer a volume of the filtrate prepared according to paragraph 6·12 containing less than 70 mg phosphoric acid and preferably about 50 mg, to a 500 ml stoppered conical flask marked at 150 ml. Dilute the solution with water to 100 ml and add 5 N sodium hydroxide solution until a faint permanent turbidity or precipitate is formed. Dissolve the precipitate by the dropwise addition of 25 per cent hydrochloric acid, but avoid an excess.

Dilute to 150 ml, add 50 ml of the citric-molybdic acid solution, heat the solution to incipient ebullition, maintain it at this temperature for 3 minutes and then bring it to the boiling point. From a burette slowly add 25 ml of the quinoline solution with constant swirling throughout, the first few ml being added dropwise, the rest in a slow stream. Keep the solution gently boiling during the addition. Immerse the flask in boiling water for 5 minutes, then cool it to 15 °C in running water.

Filter with suction the contents of the flask on a paper pulp pad, and wash the flask, precipitate and filter with successive small washes of cold water until they are free from acid. Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsings in the flask. If necessary, wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate, and place the paper in the flask. Add water to a total of about but not exceeding 100 ml. Stopper the flask and shake it vigorously until the pulp and precipitate are completely dispersed.

Remove the stopper and wash it with water, returning the washings to the flask. Add a measured volume of 0·5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml in excess. Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate after addition of 0·5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary.) Add 0·5-1·0 ml of the indicator solution, and titrate the excess of sodium hydroxide with the 0·5 N hydrochloric acid until the indicator changes from violet to green-blue and then very sharply to yellow at the end point. Deduct the number of ml of 0·5 N hydrochloric acid used from the number of ml 0·5 N sodium hydroxide to ascertain the volume of 0·5N sodium hydroxide equivalent to the phosphoric acid.

Carry out a blank determination on all the reagents, omitting only the sample, and using 0·1 N standard alkali and acid instead of 0·5 N for the titration. Calculate the blank in terms of 0·5 N alkali and subtract it from the original result.

Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1·0 ml 0·5 N sodium hydroxide ≡ 1·366 mg P_2O_5.

6·2 SPECTROPHOTOMETRIC (VANADIUM PHOSPHOMOLYBDATE) METHOD

6·21 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 °C 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_2O_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.

6·22 DISSOLUTION OF THE SAMPLE

Weigh to the nearest mg about 5 g of the sample into a capsule or dish; add 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 10 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 a 250 ml volumetric flask, dilute to the mark, mix well and filter. Discard the first 10 or 20 ml of the filtrate.

6·23 PROCEDURE

6·231 *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 10 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 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 solutions referred to the 5·0 mg phosphoric acid solution as standard.

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

6·232 *Analysis of sample*

Successively dilute a portion of the solution prepared according to paragraph 6·22 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 10 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 10 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.

7· DETERMINATION OF FIBRE

7·1 REAGENTS

Alcohol—industrial methylated spirit.

Diethyl ether.

Hydrochloric acid, 1 per cent v/v—Dilute 10 ml concentrated hydrochloric acid with water to 1 litre.

Light petroleum—boiling point 40-60 °C.

Sodium hydroxide, 0·313 *N*—This solution must be free or nearly 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 by stirring, settling and decanting three times. Air dry the extracted sample and transfer to a dry 1000 ml conical flask (see Note). Add 200 ml 0·255 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 30 minutes, maintaining a constant volume and rotating the flask every few minutes in order to mix the contents and remove particles from the sides.

Meantime, prepare a Buchner funnel 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 30 minutes boiling period, allow the acid mixture to stand for 1 minute and then pour immediately into a shallow layer of hot water under gentle suction in the prepared funnel. Adjust the suction so that the filtration of the bulk of the 200 ml is completed within 10 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 30 minutes with the same precautions as those used in the earlier boiling and treatment. Allow to stand for 1 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 per cent 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 °C 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 per cent 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 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 g.

Transfer the air-dried extracted sample to a 1000 ml conical flask, add a quantity of 1 per cent 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 1 minute. Continue the determination as described in paragraph 7·2.

8· DETERMINATION OF SUGAR

For the purposes of the Agriculture Act 1970, 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 ($CuSO_4.5H_2O$) in water and dilute to 1 litre.

Sodium potassium tartrate solution—Dissolve 346 g sodium potassium tartrate 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 phenolphthalein 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 per cent 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 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 30 minutes. Add 5 ml potassium oxalate 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 2 minutes, cool, add 2 or 3 drops of phenolphthalein indicator solution, just neutralise with 10 per cent 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 1 minute. Add 3 drops of methylene blue solution and titrate from the burette at the rate of 1 ml per 15 seconds until the blue colour is discharged, the 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 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 1½ minutes and add 3 drops of methylene blue solution. Titrate from the burette at the rate of approximately 0·25 ml per 15 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 1½ minutes. Then the total number of ml used in the determination equals the sugar equivalent of 10 ml Fehling'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 3 minutes. If this time is exceeded by more than about 20 seconds, the titration should be repeated.

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

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

Dissolve 2·375 g sucrose (dried at 100 °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 2 minutes, cool, add 2 or 3 drops of phenolphthalein solution, just neutralise with 10 per cent sodium 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 ≡ 0·005g 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 temperature not exceeding 500 °C until all the organic matter has been thoroughly 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 sodium 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 °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.

11·1 OXALATE METHOD

11·11 REAGENTS

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

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

Ammonium oxalate solution—saturated aqueous solution.

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

Citric acid—monohydrate.

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

Potassium permanganate, 0·1 N.

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

11·12 DISSOLUTION OF THE SAMPLE

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

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 15 minutes, allow to cool and stand for at least 4 hours. Decant the supernatant liquid through a sintered glass crucible (porosity 4). Wash down the sides of the beaker with hot water, stir up the calcium oxalate precipitate and allow to settle. Decant the supernatant liquid through the sintered glass crucible. Transfer the precipitate to the sintered glass crucible with 2 per cent v/v ammonia solution and wash the beaker and crucible with 2 per cent v/v ammonia solution until the washings are free from chloride. Remove the crucible and carefully rinse the outside with water, discarding the rinsings. Transfer the bulk of the precipitate to the original beaker and wash the remainder through with hot 20 per cent v/v sulphuric acid, adding the washings to the beaker. Add 70-80 ml boiling water and mix to dissolve the precipitate. Heat the contents to 75-80°C and titrate with 0·1 N potassium permanganate until a faint pink colour persists for 30 seconds, transferring the crucible to the beaker towards the end of the titration.

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

11·2 ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

11·21 APPARATUS

Atomic absorption spectrophotometer.

Calcium hollow-cathode lamp.

11·22 REAGENTS

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

Calcium dilute solution—Dilute 20 ml calcium stock solution to 200 ml. 1 ml ≡ 100 µg calcium.

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

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

11·23 PROCEDURE

Set up the instrument using the line at 422·7 nm. Use a fuel rich flame. Add releasing agent and water to a suitable aliquot of the sample solution, prepared in accordance with paragraph 11·12 to produce a standard volume of solution to contain between 5 and 10 µg of calcium per ml and 10 per cent v/v releasing agent. Prepare a blank solution from which only the sample has been omitted. Spray water into the flame and zero the instrument. Spray successively, in triplicate, the standard solutions, sample and blank, washing the instrument through with water between each spraying. Plot the mean reading obtained for each standard solution against its calcium content. Determine the calcium content of the sample and blank solutions from the graph and from the difference between them calculate the calcium content of the sample. If a number of samples is being examined, one or more standard solutions must be resprayed at intervals during the course of the analyses.

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 tetrachloride, redistilled.

Copper sulphate, stock solution—Dissolve 0·393 g copper sulphate, $CuSO_4.5H_2O$, in 100 ml 2 N sulphuric acid and dilute to 1 litre at 20 °C with distilled water.

Copper sulphate standard solution—Dilute 5 ml stock solution to 250 ml with 2 N sulphuric acid at 20 °C immediately before use. 1 ml ≡ 2 μg 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 diethyldithiocarbamate solution—Dissolve 1 g 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 7 days.

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.

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 ash 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 per cent 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. 541 (or equivalent) filter paper into a 50 ml volumetric flask. Then add 5 ml 50 per cent v/v hydrochloric acid and about 5 ml 30 per cent v/v nitric acid to the residue in the basin, and take the mixture to dryness on a hot-plate at low heat. Finally, add a further 10 ml boiling 2 N hydrochloric acid to the residue and filter the 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 μg of copper) of the solution prepared in accordance with paragraph 12·13. Add 10 ml EDTA-citrate solution, 2 drops 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 2 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:—

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 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, $CuSO_4.5H_2O$, 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 para 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 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=0·88).

Ammonia solution, 5 per cent v/v—Dilute 5 ml concentrated ammonia solution (d=0·88) with water to 100 ml.

Ammonium oxalate solution—saturated aqueous solution.

Ammonium phosphate solution—Dissolve 20 g diammonium hydrogen phosphate, $(NH_4)_2HPO_4$, in water and dilute to 100 ml.

Calcium wash solution—Dissolve 1 g oxalic acid, $(COOH)_2.2H_2O$ and 2 g ammonium oxalate in water and dilute to 1000 ml.

Citric acid—monohydrate.

Hydrochloric acid, concentrated (d=1·18).

Hydrochloric acid, 20 per cent 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 per cent industrial methylated spirit with the aid of 0·5 ml 0·1 N sodium hydroxide. Dilute to 250 ml with 50 per cent industrial methylated spirit.

Oxalic acid solution—Dissolve 10 g oxalic acid, $(COOH)_2.2H_2O$, 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 per cent 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 (4 drops) methyl red indicator solution. Neutralise with 5 per cent 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 1 to 2 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 1 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 4 hours or, preferably, overnight.

Filter through a No. 4 sintered silica crucible and wash the residue with cold 5 per cent 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 ½ to 1 hour. Allow the crucible to cool in a desiccator 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, $MgSO_4.7H_2O$, 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, $SrCl_2.6H_2O$, 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 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 accordance with para 12·13, with 0·5 N hydrochloric acid to produce a standard volume of solution containing between 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 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 diluted sample solution, before adjusting to standard.

SCHEDULE 8

FORMS OF CERTIFICATE OF ANALYSIS

(Sections 77(4), 78(3) and 79(5) to (8) and Regulation 16)

PART I

CERTIFICATE OF ANALYSIS OF FERTILISER(1)

I, the undersigned, agricultural analyst for the(2) , in pursuance of the provisions of the Agriculture Act 1970, Part IV, hereby certify that I received on the day of , 19 , from(3) one part of a sample of(4) for analysis; which was duly sealed and fastened up and marked (5) and was accompanied by a (6) , as follows:—(7)

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:—(8)

	%		%	ppm
Nitrogen (N)		Boron (Bo)		
Phosphoric acid (P_2O_5) Total ...		Cobalt (Co)		
: Soluble in water		Copper (Cu)		
: Insoluble in water		Iron (Fe)		
: Soluble in citric acid		Magnesium (Mg) ...		
Potash (K_2O)		Manganese (Mn) ...		
		Molybdenum (Mo) ...		

Neutralising value expressed in terms of calcium oxide ... ... %

Amount that will pass through the prescribed sieve(9) ... ... %

Names of herbicides and pesticides found ... ...

and I am of opinion that(10)

The analysis was made in accordance with the Fertilisers and Feeding Stuffs Regulations 1973.

As witness my hand this day of , 19 ,

(Signature and address of analyst)

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

(2) Here insert the name of the local authority.

(3) 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", "by rail", or as the case may be.

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

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

(6) 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.

(7) 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.

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

(9) Insert the number or size of the sieve used.

(10) Here enter information as follows:—

(*a*) If the material was sold under a name mentioned in the first column of Part I of Schedule 4, 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.

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

PART II

CERTIFICATE OF ANALYSIS OF FEEDING STUFF(1)

I, the undersigned, agricultural analyst for the(2) , in pursuance of the provisions of the Agriculture Act 1970, Part IV, hereby certify that I received on the day of , 19 , from(3) one part of a sample of(4) for analysis; which was duly sealed and fastened up and marked(5) and was accompanied by a (6) , as follows:—(7)

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:—(8)

	% ppm		units/kg or IU/kg
Oil		Vitamin A	
Protein: Total, including protein equivalent of urea		Vitamin D_2	
Protein equivalent of urea		Vitamin D_3	
Fibre		Vitamin E	
Sugar		Other vitamins or pro vitamins	
Salt (NaCl)		Permitted antioxidant(9)	
Phosphoric acid (P_2O_5)		Permitted colourant(9)	
Calcium (Ca)		(10)	
Copper (Cu)		(11)	
Magnesium (Mg)			
Molybdenum (Mo)			
Selenium (Se)			
Iron (Fe)			
Iodine (I)			
Cobalt (Co)			
Manganese (Mn)			
Zinc (Zn)			

(12) Analysis for oil was completed on

and I am of opinion that(13)

The analysis was made in accordance with the Fertilisers and Feeding Stuffs Regulations 1973.

As witness my hand this day of 19 ,

(Signature and address of analyst)

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

(2) Here insert the name of the local authority.

(3) 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", "by rail", or as the case may be.

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

(5) Here insert the distinguishing mark on the sample and the date of sampling shown thereon.

(6) 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.

(7) 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.

(8) Insert relevant results under the appropriate headings, i.e. percentage, parts per million or units/kg or IU/kg.

(9) Here indicate whether the antioxidant or colourant is an antioxidant listed in Part I of the table to Schedule 3 or a colourant listed in Part II of the table to Schedule 3.

(10) Here indicate the presence of any emulsifier, stabiliser or binder not listed in Part III of the Table to Schedule 3.

(11) Here insert the name and estimated percentage of any ingredient found in the sample, being an ingredient deleterious to animals of any description prescribed for the purpose of the definition of feeding stuff in section 66(1) of the Agriculture Act 1970, having regard to section 73 of that Act or in the case of substances to which regulation 7 and Schedule 3 apply the name and estimated percentage of any such substance which is deleterious to human beings.

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

(13) Here enter information as follows:—

(*a*) If the material was sold under a name mentioned in the first column of Schedule 4, 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 statement of particulars contained in the statutory statement, or the particulars marked on or indicated by a mark associated with the material, as or 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 section 72, state in what respect.

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

EXPLANATORY NOTE

(This Note is not part of the Regulations.)

These Regulations, made under Part IV of the Agriculture Act 1970 (as amended by Schedule 4E to the European Communities Act 1972), supersede the Fertilisers and Feeding Stuffs Regulations 1968, S.I. 1968/218 as amended, made under the Fertilisers and Feeding Stuffs Act 1926 (c.45). They apply throughout Great Britain and are made after consultation with persons and organisations representing the interests concerned.

The Regulations prescribe a number of matters required by the Act of 1970 to be prescribed for the purposes of Part IV of that Act and include provisions implementing Directive 70/524/EEC of the Council (O.J. No. L. 270, 14. 12. 70. p. 1 (O.J./S.E. 1970 (III), p. 840)) as respects certain additives. These matters include—

(*a*) The fertilisers and feeding stuffs for which particulars must be given on sale and the details of those particulars (Regulation 6 and Schedule 2).

(*b*) The limits of variation in those particulars so far as they relate to the nature, substance or quality of specified fertilisers and feeding stuffs (Regulation 14 and Schedule 5).

(*c*) The meanings of names of fertilisers and feeding stuffs (Regulation 13 and Schedule 4).

(*d*) The manner of marking material for sale in the course of trade for use as a fertiliser or feeding stuff (Regulation 10).

(*e*) The circumstances in which a mark may be substituted in place of the required particulars (Regulation 12(1)), being a mark whose meaning can be ascertained by reference to a register kept in accordance with Regulation 12(2) and (4).

(*f*) A modification of the Act as to the time of marking specified imported material (Regulation 11).

(*g*) The manner of taking, dividing, marking, sealing and fastening of samples of fertilisers and feeding stuffs (Regulation 4 and Schedule 1).

(*h*) The amounts of fertilisers and feeding stuffs from which samples are to be taken (Regulation 3).

(*j*) The methods by which analyses are to be carried out (Regulation 15 and Schedules 6 and 7), and the forms of certificates of analysis (Regulation 16 and Schedule 8).

(*k*) The animals whose feeding stuffs are the subject of the Regulations (Regulation 2).

(*l*) The required qualifications of agricultural analysts and deputy agricultural analysts (Regulation 5).

The principal changes from the Regulations which have been superseded are—

(i) The Regulations apply to a wider range of materials, including material sold for use as an ingredient in a fertiliser or feeding stuff.

(ii) More animals are included among those whose feeding stuffs are the subject of the Regulations (Regulation 2).

(iii) The quantity of material from which samples may be taken is specified (Regulation 3).

(iv) The schedules of ingredients of little worth and of deleterious ingredients (Schedules 3 and 5 respectively to the Fertilisers and Feeding Stuffs Act 1926, as amended) are not repeated.

(v) After 1st September 1974 the option to apply a mark in place of the required statutory particulars on individual packages on a seller's premises is restricted to certain specified materials and to material made up to an individual purchaser's specification (Regulation 12).

(vi) The use of additives in animal feeding stuffs and in ingredients sold for inclusion in feeding stuffs is controlled; where additives fall within the defined groups only scheduled additives may be used, subject, in certain circumstances, to prescribed maximum levels expressed in relation to feeding stuffs as fed to animals; other scheduled additives are subject to specified restrictions; additives other than those falling within the defined groups or those specifically mentioned may only be used if the use of the additive would not be deleterious to animals or human beings (Regulation 7 and Schedule 3).

SI 1973/1521
ISBN 0-11-031521-9

