1960. No. 145 **[C]**

FERTILISERS AND FEEDING STUFFS

The Fertilisers and Feeding Stuffs Regulations (Northern Ireland), 1960

REGULATIONS, DATED THE 20TH DAY OF SEPTEMBER, 1960, MADE BY THE MINISTRY OF AGRICULTURE UNDER THE FERTILISERS AND FEEDING STUFFS ACT, 1926.

ARRANGEMENT OF REGULATIONS

SCHEDULES

Part III. Provisions applicable to feeding stuffs.
Part IV. Division of sample.

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EIGHTH SCHEDULE. Methods of analysis of feeding stuffs.

NINTH SCHEDULE. Limits of variation.
Part I. Limits of variation for fertilisers.
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The Ministry of Agriculture for Northern Ireland in exercise of the powers vested in it by Sections 23 and 29 of the Fertilisers and Feeding Stuffs Act 1926(a), and of every other power enabling it in that behalf, and acting on the advice of the Advisory Committee appointed for Great Britain under Section 23 of the said Act, hereby makes the following Regulations:—

Citation and commencement

1. These Regulations may be cited as the Fertilisers and Feeding Stuffs Regulations (Northern Ireland), 1960, and shall come into operation on the 1st day of October, 1960.

Revocation of Previous Regulations

2. The Fertilisers and Feeding Stuffs (Northern Ireland) Regulations, 1955(b), and the Fertilisers and Feeding Stuffs (Northern Ireland) (Amendment) Regulations, 1956(c), are hereby revoked.

Interpretation

3. In these Regulations, unless the context otherwise requires,
"the Act" means the Fertilisers and Feeding Stuffs Act, 1926;
"agricultural analyst" and "deputy agricultural analyst" means respectively an official agricultural analyst and a deputy agricultural analyst appointed in accordance with the Act;
"cattle" means bulls, cows, oxen, heifers, calves, sheep, goats and swine;
"feeding stuff" means any article intended for use as food for cattle or poultry;
"fertiliser" means any article intended for use as a fertiliser of the soil;
"Ministry" means the Ministry of Agriculture for Northern Ireland.

Variation of the Schedules to the Act

4. The First, Second, Third, Fourth and Fifth Schedules to the Act are hereby varied by substituting for the same the Schedules set forth in the First, Second, Third, Fourth and Fifth Schedules respectively.

Manner of Marking Particulars on Sales of Small Quantities

5. The label of a parcel to which paragraph (ii) of the proviso to subsection (1) of Section 1 of the Act relates shall bear the particulars required by the said Section 1 to be contained in the statutory statement in block capital letters and figures of not less than half an inch in height.

(a) 16 & 17 Geo. 5. c. 45.
(b) S.R. & O. (N.I.) 1955, No. 202.
(c) S.R. & O. (N.I.) 1956, No. 186.

Manner of Marking Parcels

6. A parcel required by subsection (1) of Section 4 of the Act to be marked shall be marked in writing, printing, stencilling or in any other appropriate manner either

(*a*) on the article itself,

(*b*) where the parcel consists of a single package, on the wrapper or container of, or on a label securely attached to or placed inside, the package,

(*c*) where the parcel consists of a number of separate packages, either

(i) on the wrapper or container of, or on a label securely attached to or placed inside, each of the packages, or

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

(*d*) where the parcel consists of a number of packages themselves enclosed in a larger package or packages, on the wrapper or container of, or on a label securely attached to or placed inside,

(i) each of the packages, or

(ii) such larger package, or

(iii) each of such larger packages;

provided that

(*a*) the marking shall be legible, and

(*b*) every parcel shall be marked in such a manner that it shall remain marked so long as it is on the premises where it has been marked.

Form of Register of Marks

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

Form of Register of Articles delivered or consigned ex *ship or quay*

8. The register of articles delivered or consigned direct from a ship or quay to a purchaser, required to be kept in accordance with subsection (2) of Section 5 of the Act, shall be kept in such a form that the particulars required, relating to each separate article, shall be readily ascertainable by an inspector.

Period for which Registers and Statutory Statements are to be preserved

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

Manner of Taking and Dividing Samples

10. The manner in which samples are to be taken and dealt with in cases where under the Act they are taken in the prescribed manner shall be as set forth in the Sixth Schedule.

Method of Dealing with Third Part of Sample

11. Where a sample has been taken by an official sampler and divided by him into three parts in accordance with subsection (1) of Section 13 of the Act and Regulation 10, the third part shall be delivered or sent by

registered post to the last seller or his agent. Where a sample has been taken by an inspector and divided by him as aforesaid, the third part shall be delivered or sent by registered post to the person who would be liable to prosecution in the event of an offence being disclosed by the result of analysis of the sample, or to the representative of such person.

Period for which One Part of Sample is to be Retained by Agricultural Analyst

12. The period for which an agricultural analyst shall in accordance with subsection (2) of Section 13 retain one part of a sample sent to him shall be six months from the date of his certificate of analysis of the sample unless he shall in the meantime have submitted such part of the sample to the Chief Agricultural Analyst for Northern Ireland pursuant to subsection (3) of Section 13 or subsection (1) of Section 20.

Methods of Analysis of Fertilisers

13. The methods in which analyses of fertilisers shall be made for the purposes of the Act are as set forth in the Seventh Schedule.

Methods of Analysis of Feeding Stuffs

14. The methods in which analyses of feeding stuffs shall be made for the purposes of the Act are as set forth in the Eighth Schedule.

Limits of Variation

15. The limits of variation in relation to the particulars of the nature, substance or quality of an article or as to the amount of any ingredient, for the purposes of subsection (5) of Section 2 and subsection (5) of Section 26 of the Act, shall be as set out in the Ninth Schedule.

Forms of Certificate of Analysis

16. The certificate of an agricultural analyst (*a*) of the analysis of a fertiliser and (*b*) of the analysis of a feeding stuff shall be in the forms respectively set forth in Parts I and II of the Tenth Schedule.

Qualifications of Agricultural Analysts and Deputy Agricultural Analysts

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

Sealed with the Official Seal of the Ministry of Agriculture for Northern Ireland this 20th day of September, nineteen hundred and sixty.

(L.S.) *J. C. Baird,*
Assistant Secretary.

FIRST SCHEDULE

The Schedule substituted for the First Schedule to the Act

(Section 23(1) and Regulation 4)

FIRST SCHEDULE Sections 1, 2, 3, 4, 5, 8, 10, 12.

ARTICLES TO WHICH ALL THE PROVISIONS OF THE ACT ARE APPLICABLE

PART I

FERTILISERS

Article	*Particulars to be contained in Statutory Statement*
Ammonium nitrate and mixtures of ammonium nitrate, with any article not mentioned elsewhere in this Schedule.	Amount of nitrogen.
A product not otherwise mentioned in this Part of this Schedule, obtained by mixing one or more of the articles mentioned in this Part of this Schedule with any other such article or with any other substance or substances.	Amounts, if any, of nitrogen, potash, phosphoric acid soluble in water, and phosphoric acid insoluble in water respectively.
Basic slag	Total amount of phosphoric acid. Amount of phosphoric acid soluble in citric acid. Amount of the article that will pass through a prescribed sieve.
Bonemeal, or other product not otherwise mentioned in this Part of this Schedule, obtained by grinding or otherwise treating bone, used for fertilising purposes.	Amounts of nitrogen and phosphoric acid respectively.
Calcium cyanamide	Amount of nitrogen.
Concentrated superphosphate	Amount of phosphoric acid soluble in water.
Dicalcium 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 Part of this Schedule, obtained by drying and grinding or otherwise treating bone, flesh, flesh fibre (including whale meat) and other slaughterhouse residues, used for fertilising purposes.	Amounts of nitrogen and phosphoric acid respectively.

FIRST SCHEDULE—*contd.*

Article	*Particulars to be contained in Statutory Statement*
Nitrate of lime	Amount of nitrogen.
Nitrate of potash	Amounts of nitrogen and potash respectively.
Nitrate of soda	Amount of nitrogen.
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.
Phosphate rock, ground or otherwise	Amount of phosphoric acid. Amount that will pass through a prescribed sieve.
Potassic nitrate of soda	Amounts of nitrogen and potash respectively.
Potassium salts used as fertilisers, including kainit, sylvinite, potash, manure salt, muriate of potash, sulphate of potash and sulphate of potash-magnesia.	Amount of potash.
Precipitated bone phosphate; dicalcium bone phosphate.	Amount of phosphoric acid soluble in citric acid.
Sulphate of ammonia	Amount of nitrogen. Amount of free acid if in excess of 0·025%.
Superphosphate	Amount of phosphoric acid soluble in water.
Triple superphosphate	Amount of phosphoric acid soluble in water.

The provisions of this Part of this Schedule shall apply to any article described 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.

The amount in each case is to be stated as a definite percentage of the weight of the article, and not as a range of percentages.

Nitrogen is to be stated in terms of nitrogen.

Phosphoric acid, soluble phosphoric acid and insoluble phosphoric acid are to be stated in terms of phosphoric anhydride (P_2O_5).

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

Free acid is to be stated in terms of sulphuric acid (H_2SO_4).

PART II

FEEDING STUFFS

Article	*Particulars to be contained in Statutory Statement*
Barley meal	None.
Barley meal, Grade II	None.
Bean meal	None.
Coconut or copra cake or meal	Amounts of oil and protein respectively.

FIRST SCHEDULE—*contd.*

Article	*Particulars to be contained in Statutory Statement*
Compound cakes or meals, that is to say, any cakes or meals (other than molasses feeds and dried molassed beet pulp) consisting of a mixture of one or more of the articles mentioned in this Part of this Schedule or in Part II of the Second Schedule with any other such article or with any other substance or substances.	Amounts, if any, of oil, protein and fibre 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.
Dari or durra meal	None.
Dried plain beet pulp	Amount of fibre.
Dried molassed beet pulp	Amounts of sugar and fibre respectively.
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 meat and bone meal, or any other product of meat (including whale meat) and bone for feeding purposes.	Amounts of oil, protein and phosphoric acid respectively.
Feeding meat meal, or any other product of meat (including whale 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.
Ground oats	None.
Linseed cakes and the meals of such cakes; extracted linseed meal.	Amounts of oil and protein respectively.
Linseed meal	Amount of oil.
Locust bean meal	None.
Maize by-products and otherwise specifically mentioned in this Schedule.	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.
Maize meal; Indian meal	None.
Molasses feeds (other than dried molassed beet pulp) 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 specifically mentioned in this Schedule which are the product of any one undecorticated substance or seed from which oil has been removed.	Amounts of oil and protein respectively.

FIRST SCHEDULE—*contd.*

Article	*Particulars to be contained in Statutory Statement*
Oil cakes or meals not otherwise specifically mentioned in this Schedule 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.
Pea meal	None.
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 respectively.
Treacle or molasses	Amount of sugar.
Wheat meal	None.
Wheat offals or millers' offals	Amount of fibre.

The provisions of this Part of this Schedule shall apply to any article described 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.

The amount, in each case, is to be stated as a definite percentage of the weight of the article, and not as a range of percentages.

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

The amount of protein means the amount of nitrogen, other than ammoniacal or nitric nitrogen, if present, multiplied by 6·25.

SECOND SCHEDULE

The Schedule substituted for the Second Schedule to the Act

(Section 23(1) and Regulation 4)

SECOND SCHEDULE

Sections 1, 2, 3, 12.

ARTICLES TO WHICH SOME ONLY OF THE PROVISIONS OF THE ACT ARE APPLICABLE

PART I

FERTILISERS

Article	*Particulars to be contained in Statutory Statement*
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	None.
Chalk, ground	Neutralising value.
Chalk, screened	Neutralising value. Amount that will pass through a declared British Standard Test Sieve.
Limestone, ground; magnesian limestone, ground.	Neutralising value. Amount that will pass through a prescribed sieve.
Mixed lime	Neutralising value.
Shoddy	None.

The provisions of this Part of this Schedule shall apply to any article described 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.

The amount, in each case, is to be stated as a definite percentage of the weight of the article, and not as a range of percentages.

Neutralising value is to be expressed in terms of calcium oxide (CaO).

PART II

FEEDING STUFFS

Article	*Particulars to be contained in Statutory Statement*
Alfalfa (lucerne) meal	Amounts of protein and fibre respectively.
Clover meal	Amounts of protein and fibre respectively.
Dried brewery and distillery grains	Amounts of oil and protein respectively.
Dried grass; Dried grass (maintenance quality); Dried green fodder crops; Dried green roughage (as defined in the Fourth Schedule)	Amount of protein.
Dried yeast	Amount of protein.
Feeding dried blood	Amount of protein.
Malt culms	Amounts of protein and fibre respectively.

SECOND SCHEDULE—*contd.*

The provisions of this Part of this Schedule shall apply to any article described 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.

The amount, in each case, is to be stated as a definite percentage of the weight of the article and not as a range of percentages.

The amount of protein means the amount of nitrogen, other than ammoniacal or nitric nitrogen, if present, multiplied by 6·25.

THIRD SCHEDULE

The Schedule substituted for the Third Schedule to the Act

(Sections 23(1) and Regulation 4)

THIRD SCHEDULE Sections 1, 2, 20.

INGREDIENTS IN FEEDING STUFFS THE PRESENCE OF WHICH MUST BE DECLARED

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

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

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

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

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

FOURTH SCHEDULE

The Schedule substituted for the Fourth Schedule to the Act

(Section 23(1) and Regulation 4)

FOURTH SCHEDULE

Section 2.

DEFINITIONS IMPLIED ON THE SALE OF ARTICLES UNDER CERTAIN NAMES

PART I

FERTILISERS

Name under which Article sold	*Implied Definition*
Ammonium nitrate	Ammonium nitrate for fertilising purposes.
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.
Bone meal	Commercially pure bone, raw or degreased, which has been ground or crushed, and which contains not less than 3·5% nitrogen and not less than 20% phosphoric acid.
Bone meal, Grade II	Commercially pure bone, raw or degreased, which has been ground or crushed, and which contains less than 3·5% nitrogen or less than 20% phosphoric acid.
Burnt magnesian lime, ground or otherwise.	Commercial calcium and magnesium oxides containing more than 5·5% of magnesium (Mg.).
Burnt or quick lime, ground or otherwise.	Commercial calcium oxide containing not more than 5·5% of magnesium (Mg.).
Calcium cyanamide	Commercial calcium cyanamide.
Calcium hydroxide; hydrated lime; slaked lime.	The product obtained by slaking burnt lime.
Castor meal	The residue which is obtained by the removal of oil from commercially pure castor seed.
Chalk	Cretaceous limestone.
Chalk, ground	Cretaceous limestone which has been reduced in size so that it will pass through a sieve of ¼ in. square apertures.
Chalk, screened	Cretaceous limestone that will pass through a sieve having apertures not exceeding 3 in. square.
Compound fertiliser; mixed fertiliser; fertiliser mixture.	A product, not otherwise mentioned in this Part of this Schedule, containing two or three of the elements nitrogen, phosphorus and potassium, and obtained by mixing one or more of the articles mentioned in Part I of the First Schedule with any other such article or with any other substance or substances.
Concentrated superphosphate	Phosphate rock which has been treated with sulphuric acid and phosphoric acid.
Dicalcium phosphate	Dicalcium phosphate for fertilising purposes.
Dissolved or vitriolised bone	Commercially pure bone which has been treated with sulphuric acid.
Dried blood	Blood which has been dried, to which no other matter has been added.

FOURTH SCHEDULE—*contd.*

Name under which Article sold	*Implied Definition*
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.
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 $\frac{3}{16}$ in. square apertures, not less than 95% will pass through a sieve of $\frac{1}{8}$ in. square apertures and not less than 40% will pass through a prescribed sieve.
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 $\frac{3}{16}$ in. square apertures, not less than 95% will pass through a sieve of $\frac{1}{8}$ in. square apertures and not less than 40% will pass through a prescribed sieve.
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 (including whale meat) and other slaughterhouse residues, to which no other matter has been added.
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 defined in this Schedule.
Muriate of potash	Potassium chloride for fertilising purposes.
Nitrate of lime	Calcium nitrate for fertilising purposes.
Nitrate of potash	Potassium nitrate for fertilising purposes.
Nitrate of soda	Sodium nitrate for fertilising purposes.
Phosphate rock, ground or otherwise.	The substance obtained from mineral calcium phosphate deposits, to which no other matter has been added.
Potassic nitrate of soda ..	A mixture of sodium nitrate and potassium nitrate for fertilising purposes.
Rape meal	The residue which is obtained by the removal of oil from commercially pure rape seed.
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.
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.

FOURTH SCHEDULE—*contd.*

Name under which Article sold	*Implied Definition*
Slaked magnesian lime ..	The product obtained by slaking burnt magnesian lime.
Steamed bone flour; steamed bone meal.	Commercially pure bone from which nitrogen has been removed by steam.
Sulphate of ammonia ..	Ammonium sulphate for fertilising purposes.
Sulphate of potash	Potassium sulphate for fertilising purposes.
Superphosphate	Phosphate rock which has been treated with sulphuric acid.
Triple superphosphate ..	Phosphate rock which has been treated with phosphoric acid only.

PART II

FEEDING STUFFS

Name under which Article sold	*Implied Definition*
Alfalfa (lucerne) meal ..	Alfalfa (lucerne), as grown, dried 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 (synonym Faba vulgaris) 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 and ground, to which no other matter has been added.
Compound cakes or meals ..	Cakes or meals (other than molasses feeds and dried molassed beet pulp) consisting of a mixture of one or more of the articles mentioned in Part II of the First Schedule or in Part II of the Second Schedule with any other such article or with any other substance or substances.
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.

FOURTH SCHEDULE—*contd.*

Name under which Article sold	*Implied Definition*
Dried brewery grains ..	The article produced by drying the residue of malted and unmalted cereals used in brewing, to which no other matter has been added.
Dried distillery grains ..	The article produced by drying the residues from distillery mash-tuns, to which no other matter has been added.
Dried grass	Any product 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 as defined in 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 which (*a*) is obtained by artificially drying any green crop or crops suitable for use as dried fodder for cattle or poultry, 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 10% protein calculated on the assumption that it contains 10% moisture, but is not dried grass to dried grass (maintenance quality).
Dried green roughage ..	Any product which contains less than 10% protein calculated on the assumption that it contains 10% moisture, but which in all other respects complies with the definition of dried grass or dried green fodder crops.
Dried plain beet pulp ..	The article 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 article 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	An article 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	The product obtained by grinding commercially pure steamed bone.
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.

FOURTH SCHEDULE—*contd.*

Name under which Article sold	*Implied Definition*
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 and horn) 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 and horn) 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.
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) 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, 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".

FOURTH SCHEDULE—*contd.*

Name under which Article sold	*Implied Definition*
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.

In the case of every article mentioned in this Schedule the definition of which includes the expression "commercially pure", it is implied that no other matter may be added.

FIFTH SCHEDULE

The Schedule substituted for the Fifth Schedule to the Act

(Section 23(1) and Regulation 4)

FIFTH SCHEDULE Section 7.

DELETERIOUS INGREDIENTS IN FEEDING STUFFS

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

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

(*c*) Sand, silicious matter or other insoluble mineral matter not naturally associated with ingredients of the feeding stuff which do not fall within the scope of this Schedule, or which, even if naturally so associated, are present in greater proportion than the maximum that may be expected to be due to such natural association.

For the purposes of this paragraph the term "insoluble" shall imply insolubility as determined by a prescribed method; the term "natural association" shall be construed as applying to average commercial samples of the feeding material with which it may be claimed that a particular mineral ingredient is associated.

SIXTH SCHEDULE

Manner of Taking and Dividing Samples

(Sections 3(1) and (2), 4(3), 5(3), 6, 7(1) and 12(1) and Regulation 10)

PART I

PROVISIONS APPLICABLE TO BOTH FERTILISERS AND FEEDING STUFFS

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

2. In the case of articles in packages, only unopened packages shall be selected for the purpose of the sample.

3. Samples shall not be drawn from part of any quantity which part bears the appearance of having received damage in transit or after delivery.

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

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

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

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

The packages or portions shall be selected one from each part and shall be drawn from different positions in each part.

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

SIXTH SCHEDULE—*contd.*

PART II

PROVISIONS APPLICABLE TO FERTILISERS

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

(a) *In packages*

Where the fertiliser is in packages and the quantity exceeds 2 cwt., a number of packages shall be selected as follows, viz.:—

	If the sample is drawn by an inspector under Section 12(1) of the Act		If the sample is drawn by an official sampler, after delivery of the article, under Section 3 of the Act	
	Quantity taken for sampling	But not fewer packages than	Quantity taken for sampling	But not fewer packages than
	%		%	
Where the quantity exceeds one package and does not exceed 20 packages	20	2	10	2
Where the quantity exceeds 20 packages and does not exceed 60 packages	10	4	5	2
Where the quantity exceeds 60 packages and does not exceed 200 packages	7	6	4	3
Where the quantity exceeds 200 packages and does not exceed 500 packages	5	15	3	8
Where the quantity exceeds 500 packages and does not exceed 1,000 packages	4	25	2	13
Where the quantity exceeds 1,000 packages	3	40	1	20

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

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. 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 for each selected package by means of a closed sampling spear. The separate portions thus taken shall be thoroughly mixed together.

SIXTH SCHEDULE—*contd.*

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 quantity exceeds 2 cwt. and does not exceed 1 ton ..	4
Where the quantity exceeds 1 ton and does not exceed 2 tons ..	6
Where the quantity exceeds 2 tons and does not exceed 5 tons ..	10
Where the quantity exceeds 5 tons and does not exceed 10 tons ..	15
Where the quantity exceeds 10 tons and does not exceed 25 tons ..	25
Where the quantity exceeds 25 tons and does not exceed 50 tons ..	40
Where the quantity exceeds 50 tons and does not exceed 100 tons ..	60
Where the quantity exceeds 100 tons for each additional 10 tons or part thereof	2

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 (such as shoddy, wool refuse, hair, etc.)*

(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*).

SIXTH SCHEDULE—*contd.*

4. *Where the fertiliser is in a fluid condition*

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

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

(b) *In drums, kegs, or other containers each containing more than one quart*

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

5. 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.

6. 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 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 whole quantity as follows:—

	Cakes
Where the quantity exceeds 2 cwt. and does not exceed 2 tons ..	5
Where the quantity exceeds 2 tons and does not exceed 5 tons ..	10
Where the quantity exceeds 5 tons and does not exceed 50 tons ..	15
Where the quantity exceeds 50 tons and does not exceed 100 tons ..	25
Where the quantity exceeds 100 tons for each additional 20 tons or part thereof	2

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 6 lb. 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 a fluid or semi-fluid condition*

The number of bottles or containers to be selected shall be taken in accordance with the appropriate scale shown in paragraph 1(*a*) of Part II of this Schedule,

SIXTH SCHEDULE—*contd.*

the contents well mixed by stirring or shaking, and a similar portion taken from each. These portions shall then be mixed together in a clean, dry vessel, and from the mixture a sample of from about 2 lb. to 4 lb. in weight shall be drawn.

4. 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 OF SAMPLE

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

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

The sample, drawn as prescribed 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 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 or cotton 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 fluid or semi-fluid condition*

The sample, drawn as prescribed 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 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 article, any mark applied to the article in compliance with the Act, the date and place of the sampling and some distinguishing number, in such a manner that the particulars so marked can be seen without breaking the seal or seals.

SEVENTH SCHEDULE

Methods of Analysis of Fertilisers

(Sections 3(1), 4(3), 5(3), 6, 7(1), 13(2), 20(1), 26(4) and 28(1) and Regulation 13)

(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 Free Acid in Sulphate of Ammonia.
7. Determination of Neutralising Value in Liming Materials.
8. Determination of Magnesium in Lime and Ground Limestone.
9. The Prescribed Sieve and Method of Sieving.

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 1·18 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 1·18. Mix, withdraw a portion

* British Standard Test Sieve, Mesh No. 16 is suitable (British Standards for Test Sieves 410:1943).

SEVENTH SCHEDULE—*contd.*

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 **Basic slag**

Grind the sample to pass through the appropriate sieve prescribed in paragraph 1·18. Mix thoroughly and transfer to a non-corrodible container provided with an air-tight closure.

1·14 **Wool, hair, shoddy, etc.**

Prepare coarse organic fertilisers by tearing apart and cutting into a fine condition; some organic fertilisers, e.g. shoddy, may be prepared by a mincing or shredding machine. Prepare for analysis by pulling out or teazing out small portions of approximately equal size from throughout the bulk prepared as above, mix thoroughly and transfer to a non-corrodible container provided with an air-tight closure.

1·15 **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 1·18 and transfer to a non-corrodible container provided with an air-tight closure. Determine the moisture in this prepared sample and calculate the results of analysis of this sample to the "as received" condition.

1·16 **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 1·18. 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.

1·17 **Liquid fertilisers**

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

1·18 **Sieve**

Type of fertiliser	*Sieve apertures*
Basic slag	About 0·15 mm. square*
Ground mineral phosphate and granular fertilisers	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§

* British Standard Test Sieve, Mesh No. 100 is suitable
† 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 Standards for Test Sieves 410:1943.

SEVENTH SCHEDULE—*contd.*

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 nitric) in the presence of nitrates.

3·5 Nitrogen in the form of ammonium salts.

3·6 Nitrogen in nitrates.

3·1 REAGENTS

Ammonium alum.

Standard indigo solution.—Cautiously add 40 ml. of concentrated sulphuric acid to 1 g. of indigo carmine (B.P. quality) and stir until dissolved. Pour the solution into 800 ml. of 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. of potassium nitrate in 20 ml. of water. Add rapidly 40 ml. of concentrated sulphuric acid and heat to boiling point; the blue colour is just discharged in 1 minute.

Concentrated sulphuric acid—nitrogen free.

Mercury or mercuric oxide.

Anhydrous sodium sulphate or potassium sulphate.

Paraffin wax.

50% *sodium hydroxide solution.*—Dissolve 500 g. of sodium hydroxide in water and dilute to 1 litre.

Sodium thiosulphate.

0·2 *N sulphuric acid or hydrochloric acid.*

0·2 *N sodium hydroxide solution*—carbonate free.

Methyl red indicator solution.—Dissolve 25 mg. of methyl red in 5 ml. of 90% industrial methylated spirit with the aid of 0·5 ml. of 0·1 N sodium hydroxide solution. Dilute to 250 ml. with 50% industrial methylated spirit. If desired, a screened methyl red indicator may be used.

Sucrose.

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

* British Standard Test Sieve. Mesh No. 60 is suitable (British Standards for Test Sieves 410:1943).

SEVENTH SCHEDULE—*contd.*

10% *sulphuric acid.*—To 500 ml. of water cautiously add 100 ml. of concentrated sulphuric acid. Cool and dilute to 1 litre.

50% *sulphuric acid.*—To 500 ml. of water cautiously add 500 ml. of concentrated sulphuric acid. Cool and dilute to 1 litre.

Sodium hydroxide.

Light magnesium oxide.

3·2 TEST FOR ABSENCE OF NITRATES

Shake 5 g. of the sample with 80 ml. of water in a 100 ml. volumetric flask. Add 1 g. of ammonium alum, dilute to 100 ml., shake well and filter into a dry beaker. Dilute 1 ml. of the filtrate with 8 ml. of water. Add 1 ml. of standard indigo solution and 10 ml. of 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. of nitrogen) and transfer to a Kjeldahl flask. Add 25 ml. of concentrated sulphuric acid, 2 small globules of mercury (approximately 400 mg.) or approximately 0·5 g. of mercuric oxide, and 10 g. of 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. of paraffin wax.

3·32 Dissolve the cooled digest in water, and make up to a total volume of about 250 ml. Taking precautions against loss of ammonia, add sufficient 50% sodium hydroxide solution to neutralise the acid and 10 ml. in excess; then add 5 g. of 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. of sucrose in place of the sample. Express the result in terms of nitrogen. 1 ml. 0·2 N acid=0·0028 g. nitrogen.

3·4 TOTAL NITROGEN (ORGANIC, AMMONIACAL AND NITRIC) 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. of nitrogen), transfer to a 500 ml. Kjeldahl flask, add 3 g. of Devarda alloy and wash down the inside wall of the flask with 50 ml. of water. Close the flask with a rubber stopper provided with (*a*) a tap funnel and (*b*) a delivery tube connected with a U tube (with bulbs) containing 10 ml. of 10% sulphuric acid. Add 5 ml. of 50% sodium hydroxide solution through the tap funnel; allow to stand for 30 minutes and then heat at just below boiling point for 60 minutes. Cool, add 20 ml. of 50% sulphuric acid through the tap funnel in such a manner that the sides of the Kjeldahl flask are washed down by the acid. Remove the rubber stopper, wash the contents of the U tube into the Kjeldahl flask, add 25 ml. of concentrated sulphuric acid and heat until all the water has boiled off. Add 2 globules of mercury (approximately 400 mg.) or approximately 0·5 g. of mercuric oxide, and 10 g. of anhydrous sodium sulphate or potassium sulphate and heat gently over a small flame until 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. of paraffin wax. Cool, and complete the determination by the method described in paragraph 3·32.

SEVENTH SCHEDULE—*contd.*

3·5 NITROGEN IN THE FORM OF AMMONIUM SALTS

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. of 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. of nitrogen) to a distillation flask, add approximately 300 ml. of water and 20 ml. of 50% sodium hydroxide solution. (If urea is known to be present, 10 g. of light magnesium oxide should be used.) 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 solution as an indicator. Carry out a blank test on the reagents and water used omitting only the sample. Express the result in terms of nitrogen. 1 ml. 0·2 N acid = 0·0028 g. nitrogen.

3·52 **In the presence of organic matter**

Weigh to the nearest mg. about 5 g. of the sample, transfer to a 250 ml. volumetric flask, add 200 ml. of 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.

3·6 NITROGEN IN NITRATES

Weigh to the nearest mg. about 3 g. of the sample, transfer to a 250 ml. volumetric flask, add 200 ml. of 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. of nitrogen) to a distillation flask. Add 10 g. of Devarda alloy, 250 ml. of water and 15 ml. of 50% 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 solution as an indicator. Carry out a blank test on the reagents omitting the water solution of the sample. Express the result in terms of nitrogen. 1 ml. 0·2 N acid=0·0028 g. nitrogen.

NOTE: This method determines ammoniacal and nitric nitrogen together. To obtain the nitric nitrogen content of the sample, deduct the ammoniacal nitrogen determined in accordance with the appropriate method described in paragraph 3·5.

4· DETERMINATION OF PHOSPHORIC ACID

For the purposes of the Fertilisers and Feeding Stuffs Act, 1926, "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 4,200 Å is required; alternatively a filter instrument can be used.

SEVENTH SCHEDULE—*contd.*

Phosphoric acid in materials other than 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, dicalcium phosphate, precipitated bone phosphate and dicalcium bone phosphate, solubility in a 2% solution of citric acid is substituted for solubility in water. Because of the chemical composition of 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.
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.
4·16 and 4·26 Total phosphoric acid in basic slag.
4·17 and 4·27 Citric acid-soluble phosphoric acid in basic slag.

4·1 QUINOLINIUM PHOSPHOMOLYBDATE METHOD

4·11 Reagents

Concentrated hydrochloric acid.
Concentrated nitric acid.
Calcium oxide—finely ground.
Calcium carbonate.
5 N sodium hydroxide solution.
Dilute hydrochloric acid.—Dilute 240 ml. of concentrated hydrochloric acid with water to 1 litre.

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.—Stir 54 g. of molybdic anhydride (MoO_3) with 200 ml. of water, add 11 g. of sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdic anhydride dissolves. Dissolve 60 g. of citric acid in about 250 to 300 ml. of water and add 140 ml. of 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%) 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.—Stir 54 g. of molybdic anhydride (MoO_3) with 200 ml. of water, add 11 g. of sodium hydroxide and stir the mixture, whilst heating to boiling point, until the molybdic anhydride dissolves. Dissolve 120 g. of citric acid in about 250 to 300 ml. of water and add 140 ml. of 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

SEVENTH SCHEDULE—*contd.*

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%) solution of potassium bromate until the colour is discharged. This reagent should be kept in the dark.

Quinoline solution.—Measure 60 ml. of concentrated hydrochloric acid and 300 to 400 ml. of water into a 1 litre beaker and warm to 70°-80°C. Pour 50 ml. of 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.

0·5 *N sodium hydroxide solution*—carbonate free.

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

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

Phenolphthalein solution.—Dissolve 250 mg. phenolphthalein in 150 ml. of industrial methylated spirit and dilute with water to 250 ml.

0·5 *N hydrochloric acid.*

0·1 *N sodium hydroxide solution*—carbonate free.

0·1 *N hydrochloric acid.*

Surface active agent.—0·5% solution of sodium dodecyl benzene sulphonate is suitable.

Crystallised citric acid—monohydrate.

4·12 **Total phosphoric acid in fertilisers other than 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. of water and stir thoroughly. Boil the mixture, add slowly to the boiling solution 10 ml. of concentrated hydrochloric acid in a thin stream, and then 10 ml. of 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. of 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. of water, stir thoroughly and heat to boiling point. Add slowly to the boiling solution 10 ml. of concentrated hydrochloric acid, and then 10 ml. of 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.

SEVENTH SCHEDULE—*contd.*

4·122 PROCEDURE

Transfer a volume of the solution prepared according to paragraph 4·1211 or paragraph 4·1212 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 the solution with water to 100 ml. If the sample does not contain calcium add 100 to 200 mg. of 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 dilute 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 to 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 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.

4·13 **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. of 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

SEVENTH SCHEDULE—*contd.*

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**

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. of pure crystallised 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. of 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

SEVENTH SCHEDULE—*contd.*

calcium add 100 to 200 mg. of 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 dilute 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 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**

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. of water, and then add a further 70 ml. of water with continuous stirring. Warm the mixture and add dropwise with stirring, 10 ml. of concentrated hydrochloric acid, then 5 ml. of 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. of 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

SEVENTH SCHEDULE—*contd.*

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. of 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 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.

4·17 **Citric acid-soluble phosphoric acid in 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. of pure crystallised 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. of 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

SEVENTH SCHEDULE—*contd.*

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**

Potassium dihydrogen phosphate—containing at least 99·8% monopotassium dihydrogen phosphate.

Ammonium molybdate.

Ammonium vanadate.

Concentrated hydrochloric acid.

Concentrated nitric acid.

Calcium oxide—finely ground.

Standard phosphate solution.—Dissolve in water 1·9173 g. of potassium dihydrogen phosphate previously dried at 105°C. for 1 hour, and dilute to 1 litre. Make a 5-fold dilution (1 ml.=0·2 mg. phosphoric acid (P_2O_5)).

Vanadium molybdate reagent.—Dissolve separately 20 g. of ammonium molybdate and 1 g. of ammonium vanadate in water, mix, acidify with 140 ml. of concentrated nitric acid and dilute to 1 litre.

Normal sodium hydroxide solution.

Crystallised citric acid—monohydrate.

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

4·221 DISSOLUTION OF THE SAMPLE

SEVENTH SCHEDULE—*contd.*

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. of water and stir thoroughly. Boil the mixture, add slowly to the boiling solution 10 ml. of concentrated hydrochloric acid in a thin stream, and then 10 ml. of 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. of 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. of water, stir thoroughly and heat to boiling point. Add slowly to the boiling solution 10 ml. of concentrated hydrochloric acid, and then 10 ml. of 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 vanadium 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, say 4,200 Å, fill two 1 cm. cells with the 5·0 mg. solution and check the optical density of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent optical density 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 vanadium 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 vanadium molybdate reagent (at 20°C.), dilute to the mark, mix, and allow to stand for 10 minutes.

SEVENTH SCHEDULE—*contd.*

Measure the difference in optical density 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. of 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 vanadium 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, say 4,200 Å, fill two 1 cm. cells with the 5·0 mg. solution and check the optical density of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent optical density 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.

4·2322 *Analysis of sample*

To 25 ml. of the solution prepared according to paragraph 4·231, add 1 ml. of concentrated nitric acid; heat to incipient ebullition on a hotplate and maintain it at this temperature for 10 minutes. Cool, neutralise with normal 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 vanadium 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 vanadium molybdate reagent (at 20°C.), dilute to the mark, mix, and allow to stand for 10 minutes.

Measure the difference in optical density 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.

SEVENTH SCHEDULE—*contd.*

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**

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. of pure crystallised 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 vanadium 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, say 4,200 Å, fill two 1 cm. cells with the 5·0 mg. solution and check the optical density of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent optical density 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 vanadium 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 vanadium molybdate reagent (at 20°C.), dilute to the mark, mix, and allow to stand for 10 minutes.

Measure the difference in optical density 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.

SEVENTH SCHEDULE—*contd.*

4·26 **Total phosphoric acid in 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. of water and then add a further 70 ml. of water with continuous stirring. Warm the mixture and add dropwise with stirring, 10 ml. of concentrated hydrochloric acid, then 5 ml. of 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 vanadium 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, say 4,200 Å, fill two 1 cm. cells with the 5·0 mg. solution and check the optical density of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent optical density 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 temperature of 20°C.

Transfer this final volume to a 100 ml. volumetric flask, add 25 ml. of the vanadium 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 vanadium molybdate reagent (at 20°C.), dilute to the mark, mix, and allow to stand for 10 minutes.

Measure the difference in optical density 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**

4·271 PREPARATION OF THE SOLUTION

Weight to the nearest mg. about 5 g. of the sample and transfer to a stoppered bottle of about 1 litre capacity. Dissolve 10 g. of pure crystallised 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

SEVENTH SCHEDULE—*contd.*

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 vanadium 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, say 4,200 A, fill two 1 cm. cells with the 5·0 mg. solution and check the optical density of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent optical density 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 vanadium 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 vanadium molybdate reagent (at 20°C.), dilute to the mark, mix, and allow to stand for 10 minutes.

Measure the difference in optical density 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 Fertilisers and Feeding Stuffs Act, 1926, "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% 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

SEVENTH SCHEDULE—*contd.*

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**

Concentrated hydrochloric acid.

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

Dilute hydrochloric acid.—Dilute 240 ml. of concentrated hydrochloric acid with water to 1 litre.

Calcium oxide—finely ground.

Ammonium hydroxide solution—sp. gr. 0·88.

Ammonium carbonate solution—saturated aqueous solution.

Ammonium oxalate solution—saturated aqueous solution.

20% *perchloric acid solution.*

Alcohol—industrial methylated spirit 95-96% v/v.

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**

Dissolve in water a portion of the sample weighed to the nearest mg., equivalent in potassium content to 1·5 to 2·0 g. of potash. Cool the solution to 20°C., dilute to 500 ml. in a volumetric flask, mix well and filter through a dry filter. 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, equivalent in potassium content to 1·5 to 2·0 g. of potash, into a 500 ml. beaker, add about 300 ml. of water and 20 ml. of concentrated hydrochloric acid and heat the solution to boiling. To the boiling solution 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. Cool the liquid to 20°C., transfer to a 500 ml. volumetric flask, dilute to 500 ml., mix, and filter through a dry filter. 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. of dilute hydrochloric acid and filter if necessary. 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. Transfer the weighed portion of the sample or the incinerated residue to a 500 ml. beaker with a little water and 10 ml. of 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. of calcium oxide made into a paste with water. Bring the contents

SEVENTH SCHEDULE—*contd.*

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 ammonium hydroxide 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. of ammonium oxalate solution. Cool to 20°C., dilute with water to 500 ml. and, after thoroughly shaking, filter through a dry paper filter. 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 if necessary. 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. of perchlorate 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. of perchloric acid solution and again concentrate to the fuming stage. Thoroughly cool the residue in the basin and stir in 20 ml. of 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. Re-dissolve the precipitate on the paper and that remaining in the basin with hot water, add 2 ml. of 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. of 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**

Concentrated hydrochloric acid.

Diluted hydrochloric acid.—Dilute 500 ml. of concentrated hydrochloric acid with water to 1 litre.

Ammonium oxalate solution—saturated aqueous solution.

SEVENTH SCHEDULE—*contd.*

Ammonium hydroxide solution—sp. gr. 0·88.

Concentrated nitric acid.

Chloroplatinic acid solution.—Dissolve a weighed quantity of platinum by gentle heating in a mixture of 4 volumes of concentrated hydrochloric acid, 1 volume of concentrated nitric acid and 1 volume of 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. of diluted hydrochloric acid and evaporate again to a syrup. Repeat the evaporation with diluted 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. of platinum in 10 ml.

Alcohol—industrial methylated spirit 95-96% v/v.

Wash solution.—Dissolve 200 g. of ammonium chloride in 1 litre of water, add 10 to 20 g. of pulverised potassium chloroplatinate and shake the mixture at intervals for 6 to 8 hours. Allow the mixture to settle and filter before use.

5·22 **Potassium salts**

If the salts contain ammonium, 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 beaker. Add 10 ml. of concentrated hydrochloric acid and 50 ml. of 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. of potash (K_2O). Mix the solution and filter through a dry filter. 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 250 ml. beaker. Add 125 ml. of water and 50 ml. of 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 ammonium hydroxide 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. of potash (K_2O). Mix the solution and filter through a dry filter. 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 boil the residue for 30 minutes with 125 ml. of water and 50 ml. of ammonium oxalate solution. Cool the solution, add a slight excess of ammonium hydroxide 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. of potash (K_2O). Mix the solution and filter through a dry filter. 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

SEVENTH SCHEDULE—*contd.*

to 500 ml. together with 10 ml. of 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. of concentrated hydrochloric acid. Boil the liquid down to approximately 25 ml. and add 5 ml. of 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. of 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. of 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 due to potash emitted from a flame into which a solution of the sample is sprayed. The chosen radiations lie in the spectral range 7660Å-7700Å. 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% by weight.

5·31 **Reagents**

Potassium dihydrogen phosphate—containing at least 99·8% monopotassium dihydrogen phosphate.

Concentrated hydrochloric acid.

Ammonium oxalate solution—saturated aqueous solution.

Ammonium hydroxide solution—sp. gr. 0·96.

Standard potash solution.—Dissolve in water 5·779 g. of potassium dihydrogen phosphate previously dried for 1 hour at 105°C., and dilute to 1 litre in a volumetric flask. Shake well. Transfer 50 ml. to a 1 litre volumetric flask and dilute to the mark. Shake well. This solution now contains 100 p.p.m. potash (K_2O).

5·32 **Potassium salts**

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

SEVENTH SCHEDULE—*contd.*

Weigh to the nearest mg. about 2·5 g. of the sample and transfer to a 400 ml. beaker. Add 10 ml. of concentrated hydrochloric acid and 50 ml. of 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. Successively dilute so that the final solution contains approximately 16 p.p.m. 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 2·5 g. of the sample and transfer to a 400 ml. beaker. Add 125 ml. of water and 50 ml. of 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 ammonium hydroxide 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. Successively dilute so that the final solution contains approximately 16 p.p.m. 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 and boil the residue for 30 minutes with 125 ml. of water and 50 ml. of ammonium oxalate solution. Cool the solution, add a slight excess of ammonium hydroxide 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. Successively dilute so that the final solution contains approximately 16 p.p.m. 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 p.p.m. potash. Set the sensitivity of the flame photometer so that 100 scale divisions (full scale deflection) is equivalent to 20 p.p.m. potash solution. Spray the 10, 12, 14, 16 and 18 p.p.m. 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 p.p.m. solution to ensure that the sensivity of the flame photometer has not changed.

5·352 ANALYSIS OF SAMPLE

Reset the instrument at 100 scale divisions (full scale deflection) with 20 p.p.m. 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 p.p.m. more and 1 p.p.m. 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

SEVENTH SCHEDULE—*contd.*

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 p.p.m. more and 1 p.p.m. 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 FREE ACID IN SULPHATE OF AMMONIA

6·1 REAGENTS

Methyl red indicator solution.—Dissolve 25 mg. of methyl red in 5 ml. of 90% industrial methylated spirit with the aid of 0·5 ml. of 0·1 N sodium hydroxide solution. Dilute to 250 ml. with 50% industrial methylated spirit. If desired a screened methyl red indicator may be used.

0·1 *N sodium hydroxide solution*—carbonate free.

6·2 PROCEDURE

Weigh to the nearest centigram about 20 g. of the sample and dissolve in about 50 ml. of water. Filter, wash any insoluble matter and the filter paper free from sulphate and dilute the combined filtrate and washings to about 250 ml. Add 2 or 3 drops of the indicator solution and titrate with 0·1 N sodium hydroxide solution. Express the result as percentage by weight of sulphuric acid (H_2SO_4). 1 ml. 0·1 N sodium hydroxide solution = 0·0049 g. sulphuric acid (H_2SO_4).

7· DETERMINATION OF NEUTRALISING VALUE IN LIMING MATERIALS

7·1 REAGENTS

0·5 *N hydrochloric acid.*

Phenolphthalein indicator solution.—Dissolve 250 mg. of phenolphthalein in 150 ml. of industrial methylated spirit and dilute with water to 250 ml.

0·5 *N sodium hydroxide solution*—carbonate free.

7·2 PREPARATION OF THE SAMPLE

Prepare a portion of at least 50 g. of the sample for analysis as described in paragraph 1·11.

7·3 PROCEDURE

Weigh to the nearest mg. about 500 mg. of the sample prepared according to paragraph 7·2 and transfer to a 300 ml. flask. Add 50 ml. of 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 phenolphthalein 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).

8· DETERMINATION OF MAGNESIUM IN LIME AND GROUND LIMESTONE

8·1 REAGENTS

Standard calcium solution.—Dissolve 2·5 g. of calcium carbonate in 120 ml. of 0·5 N hydrochloric acid and dilute to 1 litre.

SEVENTH SCHEDULE—*contd.*

Buffer solution.—Dissolve 6·75 g. of ammonium chloride, 62 mg. of magnesium sulphate ($MgSO_4$. $7H_2O$), 93 mg. of disodium ethylenediamine tetracetate dihydrate and 57 ml. of ammonium hydroxide solution (sp. gr. 0·88) in water, and dilute to 100 ml.

Solochrome Black indicator.—Mix 200 mg. of Solochrome Black and 50 g. of sodium chloride uniformly together and grind to pass through a sieve having apertures of about 0·3 mm. square*.

Murexide indicator.—Mix 200 mg. of Murexide and 100 g. of sodium chloride uniformly together and grind to pass through a sieve having apertures of about 0·3 mm. square*. Protect this mixture from light.

0·5 N hydrochloric acid.

Dilute ammonia.—Dilute 240 ml. ammonium hydroxide solution (sp. gr. 0·88) to 1 litre.

N sodium hydroxide solution.

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.

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

Hydrogen peroxide solution—twenty volume strength.

0·05 N EDTA solution.—Dissolve 10 g. of disodium ethylenediamine tetracetate dihydrate in 800 ml. of water containing 55 ml. of N sodium hydroxide solution. Dilute 20 ml. of standard calcium solution with 30 ml. of water. Add 1 ml. of buffer solution and 200 mg. of Solochrome Black indicator; 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. of calcium carbonate ($CaCO_3$).

8·2 PROCEDURE

Weigh to the nearest mg. about 1 g. of finely ground sample and add 50 ml. of 0·5 N hydrochloric acid. Transfer to a conical flask, cover with a glass and boil for 3 minutes. Add 2 ml. of hydrogen peroxide solution, reboil, cool, add 1 ml. of ammonium chloride solution, a slight excess of dilute ammonia and 1 ml. of 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. of hot water. Wash the precipitate off the paper with not more than 50 ml. of water, and boil with 50 ml. of 0·5 N hydrochloric acid. Cool the solution, add 1 ml. of ammonium chloride solution, a slight excess of dilute ammonia and 1 ml. of 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. of 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. of 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. of dilute ammonia. Then add 200 mg. of Solochrome Black indicator and titrate with EDTA solution to a blue end point.

* British Standard Test Sieve, Mesh No. 52 is suitable (British Standards for Test Sieves 410:1943).

SEVENTH SCHEDULE—*contd.*

Dilute a further 20 ml. of the solution to 50 ml. and add 7 ml. of N sodium hydroxide solution. Then add 200 mg. of 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. of EDTA solution = 0·608 mg. of magnesium (Mg.).

9. THE PRESCRIBED SIEVE AND METHOD OF SIEVING

9·1 PURPOSE

Statement as to fineness of grinding of basic slag, phosphate rock, ground limestone and ground magnesian limestone. Implied definition of ground limestone and ground magnesian limestone in Part I of the Fourth Schedule to the Fertilisers and Feeding Stuffs Act, 1926.

9·2 THE SIEVE

British Standard Test Sieve, Mesh No. 100*.

9·3 METHOD OF SIEVING

Heat the sample 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. Shake the sieve for 5 minutes, frequently tapping the sides; then brush out the powder in the lower receiver and weigh. Replace the receiver and repeat the shaking and tapping procedure for 2 minutes. 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.

Disintegrate soft lumps such as can be caused to crumble by the application of the fibres of a soft brush after each shaking period, taking care that the hard parts of the brush do not make contact with the sieve, and that the brush is not used to brush particles through the sieve.

Calculate the fineness of grinding by expressing the weight of the material passing through the sieve as a percentage of the weight of the portion of the dried sample taken for sieving.

* British Standards for Test Sieves, 410:1943.

EIGHTH SCHEDULE

Methods of Analysis of Feeding Stuffs

(Sections 3(1), 4(3), 5(3), 6, 7(1), 13(2), 20(1), 26(4) and 28(1) and Regulation 14)

(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 Seventh Schedule.)

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 Phosphoric Acid.
6. Determination of Fibre.
7. Determination of Sugar.
8. Determination of Salt.
9. Determination of Ash.
10. Determination of Sand, Silicious Matter or Other Insoluble Mineral Matter.

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 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 a 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.

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*British Standard Test Sieve, Mesh No. 16 is suitable
† British Standard Test Sieve, Mesh Nos. 8, 7 or 6 is suitable } British Standards for Test Sieves 410:1943.

EIGHTH SCHEDULE—*contd.*

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

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 OIL

For the purposes of the Fertilisers and Feeding Stuffs Act, 1926, "oil" means the extract obtained as a result of treatment of a feeding stuff according to the method described in paragraph 3·21 or 3·22.

3·1 REAGENT

Petroleum spirit—light petroleum—boiling point 40°-60°C.

3·2 PROCEDURE

3·21 **For feeding stuffs not containing full cream dried milk**

Weigh to the nearest mg. about 3-5 g. of the sample; transfer to an extraction apparatus and extract with petroleum spirit 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 petroleum spirit 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 petroleum spirit 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

* British Standard Test Sieve, Mesh No. 16 is suitable (British Standards for Test Sieves 410:1943).

EIGHTH SCHEDULE—*contd.*

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 petroleum spirit extract as oil.

Where a sample is presumed to have an oil content in excess of 10%, 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 petroleum spirit 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 feeding stuffs containing full cream dried milk**

The procedure described in paragraph 3·21 above may give an incomplete extraction of oil from full cream dried milk and feeding stuffs containing full cream dried milk, and for these products the following modified procedure is prescribed. This modified procedure involves equilibrisation of the material with water vapour under conditions such that the moisture content is suitably increased but does not become excessive.

Weigh to the nearest mg. about 3-5 g. of the sample. Spread the weighed portion in a thin layer and place it in a suitable closed receptacle over a layer of water. Maintain at room temperature until the moisture content of the portion reaches approximately 10% and thereafter for a period of not less than 12 hours. The moisture content of the portion must not exceed 18% at the end of this time. Examine the portion so treated by the procedure described in paragraph 3·21.

4· DETERMINATION OF PROTEIN

4·1 Ascertain the percentage of total nitrogen by the method described in paragraph 4·2, and calculate the percentage of protein by multiplying the result by 6·25.

4·2 TOTAL NITROGEN

4·21 **Reagents**

Concentrated sulphuric acid—nitrogen free.
Mercury or mercuric oxide.
Anhydrous sodium sulphate or potassium sulphate.
Paraffin wax.
50% *sodium hydroxide solution.*—Dissolve 500 g. of sodium hydroxide in water and dilute to 1 litre.
Sodium thiosulphate.
0·2 *N sulphuric acid or hydrochloric acid.*
0·2 *N sodium hydroxide solution*—carbonate free.
Methyl red indicator solution.—Dissolve 25 mg. of methyl red in 5 ml. of 90% industrial methylated spirit with the aid of 0·5 ml. of 0·1 N sodium hydroxide solution. Dilute to 250 ml. with 50% industrial methylated spirit. If desired a screen methyl red indicator may be used.
Sucrose.

4·22 **Procedure**

Weigh to the nearest mg. about 2 g. of the sample (or such an amount as shall contain not more than 250 mg. of nitrogen) and transfer to a Kjeldahl flask. Add 25 ml. of concentrated sulphuric acid, 2 small globules of mercury (approximately 400 mg.) or approximately 0·5 g. of mercuric oxide, and 10 g. of anhydrous sodium sulphate or potassium sulphate. Heat gently over a small flame until frothing ceases and the

EIGHTH SCHEDULE—*contd.*

liquid is practically colourless. Continue to heat for a further two hours. Avoid local overheating. If frothing is excessive, add about 0·5 g. of paraffin wax.

Dissolve the cooled digest in water, and make up to a total volume of about 250 ml. Taking precautions against loss of ammonia, add sufficient 50% sodium hydroxide solution to neutralise the acid and 10 ml. in excess; then add 5 g. of 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. of 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 or nitric nitrogen, the appropriate determination should be made as described in paragraph 3·52 or paragraph 3·6 (Methods of Analysis of Fertilisers), and the amount so obtained deducted from the total nitrogen content.

5· DETERMINATION OF PHOSPHORIC ACID

For the purposes of the Fertilisers and Feeding Stuffs Act, 1926, "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.

5·1 QUINOLINIUM PHOSPHOMOLYBDATE METHOD

5·11 Reagents

Calcium oxide—finely ground.
Concentrated hydrochloric acid.
Concentrated nitric acid.
5 N sodium hydroxide solution.

Dilute hydrochloric acid.—Dilute 240 ml. of concentrated hydrochloric acid with water to 1 litre.

Citric-molybdic acid solution (A).—Stir 54 g. of molybdic anhydride (MoO_3) with 200 ml. of water; add 11 g. of sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdic anhydride dissolves. Dissolve 60 g. of citric acid in about 250 to 300 ml. of water and add 140 ml. of 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%) solution of potassium bromate until the colour is discharged. This reagent should be kept in the dark.

Quinoline solution.—Measure 60 ml. of concentrated hydrochloric acid and 300 to 400 ml. of water into a 1 litre beaker and warm to 70°-80°C. Pour 50 ml. of quinoline in a thin stream into the

EIGHTH SCHEDULE—*contd.*

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.

0·5 *N sodium hydroxide solution*—carbonate free.

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

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

Phenolphthalein solution.—Dissolve 250 mg. phenolphthalein in 150 ml. of industrial methylated spirit and dilute with water to 250 ml.

0·5 *N hydrochloric acid.*

0·1 *N sodium hydroxide solution*—carbonate free.

0·1 *N hydrochloric acid.*

Surface active agent.—0·5% solution of sodium dodecyl benzene sulphonate is suitable.

5·12 **Dissolution of the sample**

Weigh to the nearest mg. about 5 g. of the sample into a capsule or dish; add 1 g. of 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. of water; then add slowly 12 ml. of 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.

5·13 **Procedure**

Transfer a volume of the filtrate prepared according to paragraph 5·12 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 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 dilute 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

EIGHTH SCHEDULE—*contd.*

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.

5·2 SPECTROPHOTOMETRIC (VANADIUM PHOSPHOMOLYBDATE) METHOD.

5·21 **Reagents**

Potassium dihydrogen phosphate—containing at least 99·8% monopotassium dihydrogen phosphate.
Ammonium molybdate.
Ammonium vanadate.
Calcium oxide—finely ground.
Concentrated hydrochloric acid.
Concentrated nitric acid.
Standard phosphate solution.—Dissolve in water 1·9173 g. of potassium dihydrogen phosphate previously dried at 105°C. for 1 hour and dilute to 1 litre. Make a 5 fold dilution (1 ml.=0·2 mg. phosphoric acid (P_2O_5)).
Vanadium molybdate reagent.—Dissolve separately 20 g. of ammonium molybdate and 1 g. of ammonium vanadate in water, mix, acidify with 140 ml. of concentrated nitric acid and dilute to 1 litre.

5·22 **Dissolution of the sample**

Weigh to the nearest mg. about 5 g. of the sample into a capsule or dish; add 1 g. of 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. of water; then add slowly 12 ml. of 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

EIGHTH SCHEDULE—*contd.*

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.

5·23 **Procedure**

5·231 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 vanadium 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, say 4,200 Å, fill two 1 cm. cells with the 5·0 mg. solution and check the optical density of the cells. If there is a small difference, select the cell with the smaller reading as the standard reference cell.

Determine the apparent optical density 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.

5·232 ANALYSIS OF SAMPLE

Successively dilute a portion of the solution prepared according to paragraph 5·22 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 vanadium 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 vanadium molybdate reagent (at 20°C.), dilute to the mark, mix, and allow to stand for 10 minutes.

Measure the difference in optical density 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.

6· DETERMINATION OF FIBRE

For the purposes of the Fertilisers and Feeding Stuffs Act, 1926, "fibre" means the organic matter calculated as the result of treatment of the feeding stuff according to the method described in paragraph 6·2.

6·1 REAGENTS

Petroleum spirit—light petroleum—boiling point 40°-60°C.

0·255 *N sulphuric acid.*

0·313 *N sodium hydroxide solution.*—This solution must be free or nearly free from sodium carbonate.

1% *hydrochloric acid.*—Dilute 10 ml. of concentrated hydrochloric acid with water to 1 litre.

Alcohol—industrial methylated spirit 95-96% v/v.

Ethyl ether.

EIGHTH SCHEDULE—*contd.*

6·2 PROCEDURE

Weigh to the nearest mg. about 2·7 to 3·0 g. of the sample, transfer to an extraction apparatus and extract with petroleum spirit. Alternatively, extract with petroleum spirit by stirring, settling and decanting three times. Air dry the extracted sample and transfer to a dry 1000 ml. conical flask.* Add 200 ml. of 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. of 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% 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 the paper due to the insoluble material, and report the difference as fibre.

*NOTE: In the event of the sample containing 3% or more of calcium carbonate (chalk or limestone flour), it will be necessary to remove the calcium carbonate before digesting the sample with acid. This can be done at the stage in the procedure when the portion taken for analysis has been extracted with petroleum spirit. 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% 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. of 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 6·2.

EIGHTH SCHEDULE—*contd.*

7· DETERMINATION OF SUGAR

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

Sugar is included in the Schedules to the Act only as molasses or treacle, or as the sweetening constituent of molassed beet pulp and molasses feeds. It is necessary, therefore, as the first procedure, to "clean" the sugar from impurities, or from its absorbent body. The sugar is then determined as invert sugar after inversion of the sucrose.

7·1 REAGENTS

Potassium oxalate solution.—Dissolve 50 g. of potassium oxalate in water and dilute to 1 litre.

Zinc acetate solution.—Dissolve 219 g. of crystallised zinc acetate and 30 ml. of glacial acetic acid in water and dilute to 1 litre.

Potassium ferrocyanide solution.—Dissolve 106 g. of crystallised potassium ferrocyanide in water and dilute to 1 litre.

N hydrochloric acid.

Phenolphthalein indicator solution.—Dissolve 250 mg. of phenolphthalein in 150 ml. of industrial methylated spirit and dilute with water to 250 ml.

10% *sodium hydroxide solution.*—Dissolve 100 g. of sodium hydroxide in water and dilute to 1 litre.

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. of copper sulphate ($CuSO_4 . 5H_2O$) in water and dilute to 1 litre.

Sodium potassium tartrate solution.—Dissolve 346 g. of sodium potassium tartrate and 100 g. of 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. of invert sugar. It should be checked by titrating with a solution of pure sucrose (inverted by the procedure described in the Note following paragraph 7·223) using the procedure described in paragraph 7·223.

Methylene blue solution.—Dissolve 2·5 g. of methylene blue in water and dilute to 250 ml.

7·2 PROCEDURE

7·21 **Preparation of the sample**

7·211 WHEN THE SUBSTANCE IS IN SOLID FORM

Weigh to the nearest centigram about 20 g. of the sample or a sufficient quantity to contain about 2 g. of sugar. Grind in a mortar with hot water (temperature not to exceed 60°C.) and transfer with the aid of water to a 250 ml. beaker using in all about 120 ml. of water. Stir well and decant through muslin into a 250 ml. volumetric flask, allowing to drain until the liquid is substantially removed, and then squeeze the residue on the muslin. Return the residue to the beaker, add about 50 ml. of water, mix, and decant through the muslin into the volumetric flask, again squeezing the residue after draining. Repeat this treatment with a further 50 ml. of water, and finally squeeze the residue on the muslin. Add 5 ml. of potassium oxalate solution to the contents of the volumetric flask followed by 5 ml. of zinc acetate solution; mix well and then add 5 ml. of potassium ferrocyanide solution, dilute to 250 ml., mix well and filter. Determine the sugar in 50 ml. of the filtrate by the method described in paragraph 7·22.

EIGHTH SCHEDULE—*contd.*

7·212 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. of water. To clear the solution add 5 ml. of zinc acetate solution. Mix, then add 5 ml. of 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 7·22.

7·22 **Determination of the sugar content**

7·221 Transfer the measured volume of filtrate obtained as described in paragraph 7·211 or paragraph 7·212 to a 300 ml. beaker, add 15 ml. of N hydrochloric acid, dilute to 150 ml. with water, cover with a 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% sodium hydroxide solution, transfer to a 200 ml. volumetric flask and dilute to 200 ml. Filter if necessary.

7·222 PRELIMINARY ESTIMATION

(This estimation is usually necessary where the percentage of sugar is unknown.)—Transfer exactly 10 ml. of Fehling's solution to a 250 ml. conical flask and add 20 ml. of water. Add from a burette approximately 10 ml. of the filtrate prepared as described in paragraph 7·221, 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.

7·223 EXACT DETERMINATION

To 10 ml. of Fehling's solution in a 250 ml. conical flask add from a burette (X—1) ml. of the filtrate prepared as described in paragraph 7·221, 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. of 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.

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

Dissolve 2·375 g. sucrose (dried at 100°C.) in about 100 ml. of water in a 300 ml. beaker, add 15 ml. of 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% sodium hydroxide solution, transfer to a 500 ml. volumetric flask and dilute to 500 ml. Then follow the procedure described in paragraph 7·223.

EIGHTH SCHEDULE—*contd.*

1 ml. of this solution = 0·00475 g. sucrose = 0·005 g. invert sugar, i.e. 10 ml. of Fehling's solution = 10·5 ml. of this standard invert sugar solution.

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

8· DETERMINATION OF SALT

8·1 REAGENT

Calcium oxide—finely ground.—This reagent must be free from chloride.

8·2 PROCEDURE

Weigh to the nearest mg. about 5 g. of the sample, mix with 1 g. of 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).

9· 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.

10· DETERMINATION OF SAND, SILICIOUS MATTER OR OTHER INSOLUBLE MINERAL MATTER

10·1 REAGENTS

Concentrated hydrochloric acid.

Dilute hydrochloric acid.—Dilute 240 ml. of concentrated hydrochloric acid with water to 1 litre.

10·2 PROCEDURE

Weigh to the nearest mg. from 2 to 5 g. of the sample and incinerate until all the carbon has been destroyed.* Moisten with concentrated hydrochloric acid, evaporate to dryness, bake to render the silica insoluble, and then extract repeatedly with hot dilute hydrochloric acid. Filter, wash the insoluble matter with hot water, incinerate the insoluble matter and weigh. Regard the quantity obtained as sand and silicious matter.

* The ash obtained from the procedure described in paragraph 9 may be used for this determination.

NINTH SCHEDULE

Limits of Variation

(Sections 2(5) and 26(5) and Regulation 15)

PART I

LIMITS OF VARIATION FOR FERTILISERS

Article	Limits of Variation (expressed as percentages of the *whole bulk*)				
	Nitrogen	Phosphoric acid soluble in water	Phosphoric acid insoluble in water	Phosphoric acid	Potash
1. Calcium cyanamide	0·5	—	—	—	—
2. 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	0·3	2·0	—	—	—
(*b*) If such excess is not less than 1, but is less than 1·5 ..	0·3	1·5	—	—	—
(*c*) If such excess is not less than 0·5 but is less than 1 ..	0·3	1·0	—	—	—
(ii) In all other cases ..	0·3	0·5	0·5	—	—
3. Dried blood for fertilising purposes	0·5	—	—	—	—
4. Hoofs	0·5	—	—	—	—
5. Hoofs and horns	0·5	—	—	—	—
6. Horns	0·5	—	—	—	—
7. Nitrate of lime	0·5	—	—	—	—
8. Nitrate of potash	0·5	—	—	—	2·0
9. Nitrate of soda	0·5	—	—	—	—
10. Oil seed fertilisers, as described in the First Schedule to the Act	0·5	—	—	—	—
11. Potassic nitrate of soda ..	0·5	—	—	—	0·75
12. Potassium salts used as fertilisers, as described in the First Schedule to the Act:—					
(*a*) If the percentage of potash stated does not exceed 15	—	—	—	—	1·0
(*b*) If such percentage exceeds 15	—	—	—	—	2·0

NINTH SCHEDULE—*contd.*

Article	Limits of Variation (percentages are percentages of the *whole bulk*)
13. A product not otherwise mentioned in Part I of the First Schedule to the Act, obtained by mixing one or more of the articles mentioned in Part I of the said First Schedule with any other such article or with any other substance or substances.	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%.
14. Ammonium nitrate and mixtures of ammonium nitrate with any article not mentioned elsewhere in the First Schedule to the Act.	Nitrogen, one-twentieth of the amount stated.
15. Basic slag	Total phosphoric acid, 1%; phosphoric acid soluble in citric acid, 1%; amount that will pass through a prescribed sieve, one-twentieth of the amount stated.
16. Bone meal or other bone product as described in Part I of the First Schedule to the Act.	Nitrogen, 0·5%; phosphoric acid, 1%, provided that these limits of variation shall not operate so as to permit the application of the name "bone meal" to any article containing less than 3·5% nitrogen or less than 20% phosphoric acid.
17. Dicalcium phosphate ..	Phosphoric acid soluble in citric acid, 1%.
18. Fish residues or other fish products as described in Part I of the First Schedule to the Act. 19. Meat and bone residues as described in Part I of the First Schedule to the Act.	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%.
20. Guano as described in the First Schedule to the Act.	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.
21. Phosphate rock, ground or otherwise.	Phosphoric acid, one-twentieth of the amount stated; amount that will pass through a prescribed sieve, one-twentieth of the amount stated.

NINTH SCHEDULE—*contd.*

Article	Limits of Variation (percentages are percentages of the *whole bulk*)
22. Precipitated bone phosphate; dicalcium bone phosphate.	Phosphoric acid soluble in citric acid, 1%.
23. Sulphate of ammonia ..	Nitrogen, 0·3%; free acid, one-fifth of the amount stated or 0·02% whichever is the greater.
24. Superphosphate ..	Phosphoric acid soluble in water, one-twentieth of the amount stated.
25. Triple superphosphate	
26. Concentrated superphosphate	
27. Burnt or quick lime, ground or otherwise.	Neutralising value, one-tenth of the amount stated.
28. Burnt magnesian lime, ground or otherwise.	
29. Calcium hydroxide; hydrated lime; slaked lime; slaked magnesian lime.	
30. Mixed lime	
31. Chalk, ground	Neutralising value, one-twentieth of the amount stated.
32. Chalk, screened	Neutralising value, one-eighth of the amount stated; amount that will pass through a declared British Standard Test Sieve, one-tenth of the amount stated.
33. Limestone, ground; magnesian limestone, ground.	Neutralising value, one-twentieth of the amount stated; amount that will pass through a prescribed sieve, one-twentieth of the amount stated.

PART II

LIMITS OF VARIATION FOR FEEDING STUFFS

1. Alfalfa (lucerne) meal ..	Protein, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
2. Clover meal	
3. 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.
4. Cotton cakes or meals not decorticated.	
5. Oil cakes or meals not otherwise specifically mentioned in the First Schedule to the Act which are the product of any one undecorticated substance or seed from which oil has been removed.	
6. Palm kernel cake or meal	

NINTH SCHEDULE—*contd.*

Article	Limits of Variation (percentages are percentages of the *whole bulk*)
7. Compound cakes or meals as described in the First Schedule to the Act.	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater; protein, one-tenth of the amount stated; 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.
8. 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.
9. Maize by-products, not otherwise specifically mentioned in the First Schedule to the Act.	
10. Oil cakes or meals not otherwise specifically mentioned in the First Schedule to the Act, which are the product of any one decorticated or partly decorticated substance or seed from which oil has been removed.	
11. Rice bran or rice meal, or the by-product produced in milling shelled rice.	
12. Dried brewery and distillery grains.	Oil, 0·75%, or one-fifth of the amount stated, whichever is the greater; protein, one-fifth of the amount stated.
13. Dried Grass	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 article containing less than 10% protein.
14. Dried Grass (maintenance quality).	
15. Dried green fodder crops.	
16. Dried green roughage ..	
17. Dried plain beet pulp	Fibre, one-eighth of the amount stated.
18. Dried molassed beet pulp.	Sugar, one-tenth of the amount stated; fibre, one-eighth of the amount stated.
19. Molasses feeds, as described in the First Schedule to the Act.	
20. Dried yeast	Protein, one-twentieth of the amount stated.
21. Feeding dried blood ..	
22. Feeding bone flour ..	Phosphoric acid, one-twentieth of the amount stated; protein, one-fifth of the amount stated.

NINTH SCHEDULE—*contd.*

Article	Limits of Variation (percentages are percentages of the *whole bulk*)
23. Feeding bone meal, ground bone, or any other bone product for feeding purposes.	Phosphoric acid and protein, one-tenth of the respective amounts stated.
24. Feeding meat meal or any other product of meat for feeding purposes. 25. 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 articles containing less than 55% and less than 40% of protein respectively.
26. Fish meal, white fish meal, or 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 an article containing more than 6% of oil or 4% of salt.
27. Linseed cakes and the meals of such cakes; extracted linseed meal. 28. Maize, flaked .. .. 29. Maize germ cake or meal 30. Maize gluten feed .. . 31. Rape cake or meal .. 32. 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.
33. Linseed meal	Oil, 0·75%, or one-tenth of the amount stated, whichever is the greater.
34. Malt culms	Protein, one-fifth of the amount stated; fibre, one-eighth of the amount stated.
35. 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 article containing more than 27% of fibre.
36. Treacle or molasses	Sugar, one-twentieth of the amount stated.
37. 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.

TENTH SCHEDULE

Forms of Certificate of Analysis

(Sections 3(1), 13(5), (6) and (7) and 22(1) and Regulation 16)

PART I

CERTIFICATE OF ANALYSIS OF FERTILISER(1)

I, the undersigned, agricultural analyst appointed in pursuance of the provisions of the Fertilisers and Feeding Stuffs Act, 1926, hereby certify that I received on the day of , 19 , from (2) two parts of a sample of (3) for analysis; which parts were duly sealed and fastened up and marked (4) and were accompanied by a (5) , as follows:—(6)

and also by a signed statement that the sample was taken in the prescribed manner; and that one of the said parts has been analysed by me, or under my direction, and I declare the results of analysis to be as follows:—(7)

Nitrogen (N)		%
Phosphoric acid (P_2O_5)	Total	%
	Soluble in water	%
	Insoluble in water	%
	Soluble in citric acid	%
Potash (K_2O)		%
Neutralising value expressed in terms of calcium oxide (CaO)		%
Free acid, as sulphuric acid (H_2SO_4) in sulphate of ammonia		%
Amount that will pass through a prescribed sieve		%
Amount (of screened chalk) that will pass through the declared British Standard Test Sieve		%

(8)

and I am of opinion that (9) ..

The analysis was made in accordance with the Fertilisers and Feeding Stuffs Regulations (Northern Ireland), 1960.

As witness my hand this day of , 19 .

(Signature and address of analyst)

(1) Statements made in certificates are to be confined to matters which either are necessarily to be stated for the purposes of the Act or are voluntarily stated by the seller. They may extend to relevant matters of analysis, such as moisture content, but not to unrelated matters such as price.

TENTH SCHEDULE—*contd.*

(2) Here insert the name of the inspector or official sampler 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.

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

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

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

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

(7) Only the relevant items need be included.

(8) Here insert the names and percentages of other ingredients found in the sample, or particulars of the fineness of grinding, when any statement as to the amount of such ingredients or as to the fineness of grinding is made in any written documents (other than the statutory statement).

(9) Here enter information as follows:—

(*a*) If the article was sold under a name mentioned in the first column of the Fourth Schedule, state whether it accords with the definition contained in the second column; and if not, in what respect.

(*b*) If the composition of the articles agrees with or does not differ by more than the limits of variation from the statement of particulars contained in the statutory statement or warranty, or the particulars marked on or indicated by a mark applied to the article, or as the case may be, state that the particulars are correct within the limits of variation.

(*c*) If the composition of the article differs by more than the limits of variation from the particulars contained in the statutory statement, or warranty, or the particulars marked on or indicated by a mark applied to the article, or as the case may be, state the difference between the amount found and the amount stated, and that the difference is in excess of 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.)

TENTH SCHEDULE—*contd.*

PART II

CERTIFICATE OF ANALYSIS OF FEEDING STUFF(1)

I, the undersigned agricultural analyst appointed in pursuance of the provisions of the Fertilisers and Feeding Stuffs Act, 1926, hereby certify that I received on the day of , 19 , from (2) two parts of a sample of (3) for analysis; which parts were duly sealed and fastened up and marked (4) and were accompanied by a (5) as follows:—(6)

and also by a signed statement that the sample was taken in the prescribed manner; and that one of the said parts has been analysed by me, or under my direction, and I declare the results of the analysis to be as follows:—(7)

Oil	%
Protein	%
Fibre	%
Sugar	%
Salt (NaCl)	%
Sand and other silicious matter	%
Phosphoric acid (P_2O_5)	%

(8)

and I am of opinion that (9)

The analysis was made in accordance with the Fertilisers and Feeding Stuffs Regulations (Northern Ireland). 1960.

As witness my hand this day of , 19 .

(Signature and address of analyst)

(1) Statements made in certificates are to be confined to matters which either are necessarily to be stated for the purposes of the Act or are voluntarily stated by the seller. They may extend to relevant matters of analysis, such as moisture content, but not to unrelated matters such as price.

(2) Here insert the name of the inspector or official sampler 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.

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

TENTH SCHEDULE—*contd.*

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

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

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

(7) Only the relevant items need be included.

(8) Here insert:—

(*a*) the names and percentages of other ingredients found in the sample, when any statement as to the amount of such ingredients is made in any written document (other than the statutory statement).

(*b*) the name and estimated percentage of any ingredient included in the Third Schedule to the Act which is found in the sample and not expressly stated in the statutory statement.

(*c*) the name and estimated percentage of any ingredient found in the sample, being an ingredient deleterious to cattle (as defined by the Act) or to poultry, having regard to Section 7(2) and the Fifth Schedule to the Act.

(9) Here enter information as follows:—

(*a*) If the article was sold under a name mentioned in the first column of the Fourth Schedule, state whether it accords with the definition contained in the second column; and if not, in what respect.

(*b*) If the composition of the article agrees with or does not differ by more than the limits of variation from the statement of particulars contained in the statutory statement or warranty, or the particulars marked on or indicated by a mark applied to the article, or as the case may be, state that the particulars are correct within the limits of variation.

(*c*) If the composition of the article differs by more than the limits of variation from the statement of particulars contained in the statutory statement, or warranty, or the particulars marked on or indicated by a mark applied to the article, or as the case may be, state the difference between the amount found and the amount stated, and that the difference is in excess of 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 article is not suitable for feeding purposes for cattle (as defined by the Act) or for poultry, 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 but is intended to indicate their general purport.)

These Regulations consolidate, with amendments, the Regulations made in 1955 and 1956 under the Fertilisers and Feeding Stuffs Act, 1926.

The Regulations prescribe the manner of marking parcels of fertilisers and feeding stuffs intended for sale, and the forms of registers to be kept by certain persons dealing with fertilisers and feeding stuffs. The five schedules