

# **THE AGRICULTURE (FARM FEED) REGULATIONS [ARRANGEMENT OF REGULATIONS]**

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**SECTION 52-THE AGRICULTURE (FARM FEED)  
REGULATIONS**  
*Regulations by the Minister*

*Statutory  
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**PART I**  
**PRELIMINARY**

1. These Regulations may be cited as the Agriculture (Farm Feed) Regulations. Title
2. In these Regulations, unless the context otherwise requires- Interpretation  
"the Act" means the Agriculture (Fertilisers and Feed) Act.  
"Minister" means the Minister responsible for Agriculture.
3. These Regulations shall apply in relation to any farm feed as defined in section *two* of the Act. Application

**PART II**  
**REGISTRATION**

4. (1) Applications under Part II of the Act for registration, transfer of registration or renewal of registration of plant shall be made in Form FERT 4 in the First Schedule to the Agriculture (Fertilisers) Regulations, 1969 (hereinafter in these Regulations referred to as "the Fertilisers Regulations") and such application shall be accompanied by the appropriate fees shown in the First and Second Schedules to the Act, and be given to the Registering Officer.

Registration of plant and fees.  
S.I. No. 476 of 1969

(2) The Registering Officer shall issue a certificate of registration in Form FERT 5 in the First Schedule to the Fertilisers Regulations.

(3) The Registering Officer shall issue a certificate of provisional registration in Form FERT 6 in the First Schedule to the Fertilisers Regulations.

5. The Registering Officer shall keep a register of plant as prescribed in Form FERT 1 in the First Schedule to the Fertilisers Regulations.

Register of plant

6. Any plant within the definition of section *two* of the Act shall be so equipped as to permit the adequate performance therein of the activities described in the application for registration of such plant, to the satisfaction of the Registering Officer.

Requirement of plant

## **PART III**

### **ANALYSTS AND LABORATORIES**

7. (1) For the purposes of the Act an analyst shall furnish proof to the satisfaction of the Minister that he has competent knowledge of chemistry and of chemical analyses, as applied to farm feed, and such proof shall in every case comprise documentary evidence that the analyst holds a certificate or diploma attesting his possession of the requisite knowledge and given by a recognised competent body, and shall be submitted to the Minister when applying for approval, so, however, that the Minister may call for further evidence if required in any particular case.

Approval of analysts

(2) Where the requirements referred to in sub-regulation (1) are satisfied, the Registering Officer shall issue a certificate of approval on Form FERT 7 in the First Schedule to the Fertilisers Regulations.

**8.** (1) An approved laboratory shall be so equipped as to enable approved analysts to perform accurately for the purposes of the Act all the analyses specified under the Seventh Schedule to these Regulations and such laboratories shall have been inspected by a duly authorised officer of the Ministry of Agriculture, Food and Fisheries before approval by the Minister and may be inspected from time to time as the Registering Officer may deem necessary:

Approval of laboratories

Provided that, in addition, other laboratories may be approved for certain analyses only, such analyses to be specified by the Registering Officer after inspection by a duly authorised officer of the Ministry of Agriculture, Food and Fisheries.

(2) Where the Minister approves a laboratory, the Registering Officer shall issue a certificate of approval on Form FERT 8 in the First Schedule to the Fertilisers Regulations.

**9.** The Registering Officer shall keep a roll of approved analysts in Form FERT 2 in the First Schedule to the Fertilisers Regulations.

Roll of analysts

**10.** The Registering Officer shall keep a roll of approved laboratories in Form FERT 3 in the First Schedule to the Fertilisers Regulations.

Roll of laboratories

## **PART IV**

### **SEARCHES AND SEIZURES**

**11.** The certificate of authority to be held by inspectors under section *twenty-five* of the Act shall be issued by the Registering Officer and shall be-

Certificate of authority for inspectors

(a) in the case of general authorisation, in Form FERT 11 in the First

Schedule to the Fertilisers Regulations; and

(b) in the case of limited authorisation, in Form FERT 12 in the First Schedule to the Fertilisers Regulations.

## **PART V**

### **SALE OF FARM FEED**

**12.** The statement of analysis for each class of farm feed specified in the Second Schedule to these Regulations shall appear in English in lettering both durable and legible on the bag or container containing the same or on a label securely attached thereto.

Statements of analysis for sales in bags, containers, etc.

**13.** Where any class of farm feed specified in the Second Schedule to these Regulations is sold in bulk, the statement of analysis for such class shall appear in English in lettering both durable and legible on a note which shall be given to the purchaser or his agent at the time of delivery of such farm feed.

Statements of analysis for sales in bulk

**14.** Where any class of farm feed specified in the Second Schedule to these Regulations is sold in a container or a package under a trade name, trade mark, trade label or trade brand, as provided by section *thirty* of the Act, there shall appear in English in lettering both durable and legible on the container or package, or on a label securely attached thereto, a statement of analysis in respect thereof and, in addition, the Registering Officer shall have access to such records relating thereto as are specified in the Third Schedule to these Regulations.

Statements of analysis and records to be kept for sales under trade names, etc.

**15.** Where any farm feed is manufactured to a farmer's own specifications, such farm feed shall be exempted from these Regulations, but only if the farm feed is for the sole use of the farmer supplying the specifications and is not intended for re-sale.

Exemption of farm feed made to specification

**16.** No class of farm feed shall contain any of the ingredients listed as deleterious in the Fourth Schedule to these Regulations.

Deleterious ingredients

- 17.** The presence in any farm feed of any of the materials specified in the Fifth Schedule to these Regulations shall be declared in writing to the purchaser of any farm feed whether sold in containers, in bulk or under any trade name, trade mark, trade label or trade brand.
- Declaration of presence of certain ingredients upon sale

## **PART VI**

### **SAMPLING, ANALYSIS AND LIMITS OF VARIATION**

- 18.** A report of analysis shall be issued by the analyst performing the analysis in respect of every sample taken under the Act, and any such report shall be in Form FERT 9 in the First Schedule to the Fertilisers Regulations.
- Form of report to be used
- 19.** A certificate of analysis shall not be issued unless the sample has been taken in accordance with the Sixth Schedule to these Regulations and such certificate shall be in the Form FERT 10 in the First Schedule to the Fertilisers Regulations.
- Form of certificate to be used
- 20.** Samples for analysis for the purposes of the Act shall be taken in the manner prescribed in the Sixth Schedule to these Regulations and certificates of sampling issued in relation thereto shall be in Form FERT 13 in the First Schedule to the Fertilisers Regulations.
- Method of taking samples
- 21.** Methods of analysis shall be as prescribed in the Seventh Schedule to these Regulations.
- Methods of analysis
- 22.** The limits of variation in respect of any prescribed analysis shall be as prescribed in the Eighth Schedule to these Regulations.
- Limits of variation



## FIRST SCHEDULE

### Agriculture (Fertilisers and Feed) Act

### Agriculture (Farm Feed) Regulations

(Section 2)

## DEFINITIONS IMPLIED ON THE SALE OF FARM FEED UNDER CERTAIN NAMES

<i>Name of Farm Feed or Class of Feed</i>	<i>Implied Definition</i>
Cereal brans cerealkernels.	By-products produced in the milling of maize, wheat or other
Oilseed cakes or meals removal of (undecorticated) seeds, soya	Meals or cakes produced from the residues resulting from the of oil from undecorticated groundnuts, cottonseeds, sunflower beans or other oilseeds.
Oilseed cakes or meals removal of (decorticated) cottonseeds	Meals or cakes produced from the residues resulting from the oil from decorticated or partly decorticated groundnuts, sunflower seeds, soya beans or other oilseeds.
Meat and bone meal not more carcasses or matter has been removal of oil or	The product, containing not less than 40 per cent of protein (and than 4 per cent of salt) obtained by drying and grinding animal portions thereof (excluding hoof and horn) to which no other added, but which may have been treated previously for the fat.
Bone meal ground or	Commercially pure bone, raw or degreased, which has been crushed.
Meat meal not more carcasses or	The product, containing not less than 55 per cent of protein (and than 4 per cent of salt) obtained by drying and grinding animal portions thereof (excluding hoof and horn) to which no other

matter has removal of	been added but which may have been treated previously for the oil or fat.
Fish meal fish or fish waste.	A product obtained by drying and grinding or otherwise treating
Molasses sugar from sugar	A concentrated syrup product obtained in the manufacture of cane, to which no other matter has been added.
Molasses feed absorbent material	Any mixture (containing not less than 10 per cent of sugar) of an and molasses.
Mixture of molasses absorbent material.	Any mixture of molasses and urea with or without any
Compound cakes and meals	A homogeneous mixture of two or more farm feeds.
Concentrated or high substances energy farm feed to livestock.	A homogeneous mixture of two or more farm feeds or other intended to be mixed with some other farm feed before feeding

NOTE:

"Commercially pure" means that no other matter has been added. In the case of every article mentioned in this Schedule the definition shall be deemed not to exclude the presence of a substance added to improve the keeping or handling properties of the farm feed or the presence of any coccidiostat, anti-blackhead remedy, natural or synthetic hormone.

"Synthetic hormone" means a synthetic compound which has similar properties to, or has the property of stimulating the production of, a natural hormone.

"Coccidiostat" means a substance used in the prevention or curative treatment of disease in poultry caused by protozoal organisms of the order coccidia.

"Anti-blackhead remedy" means a substance used in the prevention or curative treatment of infection in poultry due to *Histomonas Meleagridis*.

## SECOND SCHEDULE

### Agriculture (Fertilisers and Feed) Act

### Agriculture (Farm Feed) Regulations

*(Sections 28, 29 and 52K and regulations 12, 13 and 14)*

### STATEMENTS OF ANALYSIS REQUIRED FOR DIFFERENT CLASSES OF FARM FEED

<i>Class of Farm Feed</i>	<i>Statement of Analysis</i>
Cereal brans	Percentages of protein, oil and fibre.
Oilseed cakes or meals (undecorticated)	Percentages of protein and oil.
Oilseed cakes or meals (decorticated)	Percentages of protein, oil and fibre.
Meat and bone meal	Percentages of protein, oil and phosphorus.
Bone meal	Percentages of protein and phosphorus.
Meat meal	Percentages of protein, oil and phosphorus.
Fish meal salt.	Percentages of protein, oil, phosphorus and
Molasses	Percentage of sugar.
Molasses feed	Percentages of sugar and fibre.
Mixture of molasses urea. and urea	Percentages of sugar and protein equivalent of
Compound cakes and the protein meals	Percentages of oil, fibre and protein (including equivalent of urea, if any)
Concentrated or high the protein energy feed proportions in farm feed defined	Percentages of oil, fibre and protein (including equivalent of urea, if any); a statement of the which the feed should be mixed with other by name or class.

#### NOTES:

1. The amount of protein, except in the case of Compound Cakes and Concentrated or High Energy farm feed, means the amount of nitrogen other than ammoniacal, nitric or urea

nitrogen, multiplied by 6.25. In the case of Compound Cakes and High Energy farm feed the amount of protein means the amount of nitrogen other than ammoniacal and nitric nitrogen multiplied by 6.25. The amount of protein equivalent of urea means the amount of urea nitrogen multiplied by 6.25.

2. In all cases the names of any added vitamins, minerals, antibiotics and synthetic or natural hormones shall be declared in the statement of analysis.

3. In all cases the maximum percentage of urea present in any farm feed shall be declared.

## **THIRD SCHEDULE**

**Agriculture (Fertilisers and Feed) Act,**

**Agriculture (Farm Feed) Regulations.**

*(Section 30 and regulation 14)*

### **STATEMENTS OF ANALYSIS AND RECORDS TO BE KEPT FOR SALES UNDER TRADE NAMES, ETC.**

Manufacturers of farm feeds shall keep records of all ingredients used in manufacturing any farm feed which is sold under a trade name, trade mark, trade label or trade brand. Such records shall be available for inspection at all reasonable times by a duly authorised officer, and shall be kept in such a manner as to enable the Registering Officer or any duly authorised officer to ascertain the materials from which any lot or batch of farm feed have been manufactured, and the proportions of such materials contained in the farm feed.

## **FOURTH SCHEDULE**

**Agriculture (Fertilisers and Feed) Act,**

**Agriculture (Farm Feed) Regulations,**

*(Section 52 and regulation 16)*

### **DELETERIOUS INGREDIENTS IN FARM FEED**

1. Salts soluble in water, if present in a farm feed in proportion likely to be injurious to the health of livestock.
2. All poisonous substances in quantities likely to be injurious to the health of livestock for which the farm feed is intended whether or not such substances are naturally present in the farm feed or material(s) from which the farm feed was manufactured.
3. Sand, siliceous matter or other insoluble mineral matter not naturally associated with ingredients of the farm feed 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.
4. For the purposes of this paragraph the term "insoluble" shall imply insolubility as determined by the prescribed method; the term "natural association" shall be construed as applying to average commercial samples of the farm feed or its ingredients with which it may be claimed that a particular mineral ingredient is associated.

## **FIFTH SCHEDULE**

**Agriculture (Fertilisers and Feed) Act,**

**Agriculture (Farm Feed) Regulations.**

*(Section 52 and regulation 17)*

### **INGREDIENTS IN FARM FEED THE PRESENCE OF WHICH MUST BE DECLARED**

1. Husks, chaff, glumes, 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 farm feed.

Where the kernels naturally associated in seeds with one or other of the above materials are present in a farm feed along with material with which they are 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.

2. Peat, peat moss or sugar cane pith, treated or untreated, ground or otherwise.
3. Wheat, maize or sorghum straw, maize rachis, ground or otherwise.
4. Sawdust or any other form of wood, treated or untreated.

## **SIXTH SCHEDULE**

### **Agriculture (Fertilisers and Feed) Act.**

### **Agriculture (Farm Feed) Regulations.**

*(Section 52 (h) and regulations 19 and 20)*

## **METHOD OR TAKING SAMPLES**

NOTE-In this regulation "metric ton" is defined as 1,000 kg.

1. Where a farm feed is contained in packages the samples shall be taken from different parts of the whole quantity as follows:

- (a) if the quantity does not exceed one ton, from not less than two unopened packages per ton or part thereof;
- (b) if the quantity exceeds one ton but does not exceed two tons, from not less than four unopened packages;
- (c) if the quantity exceeds two tons but does not exceed three tons, from not less than six unopened packages;
- (d) if the quantity exceeds three tons, from one additional unopened package for every additional ton or part thereof, but in no case need samples be taken from more than fifteen packages.

2. Where a farm feed is not contained in packages, a proportion calculated in accordance with paragraph 1 shall be taken from different parts of the whole quantity.

3. The samples shall be taken by means of a suitable sampling probe or by any other means as will ensure, as far as is practicable, the taking of a representative sample.

4. When a farm feed consists of material uneven in character, bulky or likely to get matted together, portions shall be taken from the selected packages, or from different parts of the farm feed if in bulk, any matted portions torn up, and all the portions thoroughly mixed together.

5. The samples thus taken shall be thoroughly mixed and reduced in size to give a final sample not exceeding six pounds in weight. This final sample shall be mixed and divided into three parts and each of these parts shall be transferred to a clean, dry, noncorrodible container capable of being closed in such a manner as to preserve the contents of the container in their original condition. These three containers shall be so sealed that they cannot be opened without breaking the seal. Each of these parts shall be marked with the name of the farm feed, date and place of sampling and the sample number together with the name of the inspector taking the sample.

6. When the farm feed is in liquid condition it should be thoroughly mixed before



sampling. When in containers, samples shall be taken as follows:

Where the number of containers-	Portions shall be drawn from-
exceeds 1 but does not exceed 20	not less than 2 containers
exceed 20 but does not exceed 40	not less than 4 containers
exceed 40 but does not exceed 60	not less than 6 containers
exceed 60	one extra container for every 20 containers by which the total exceeds

60.

Where the farm feed is in liquid condition and is in bulk a representative sample shall be taken in accordance with the scale of sampling set out above.

The portions drawn shall be mixed together in a clean, dry container and a sample of 1 kg weight shall be taken. The sample shall be divided into three equal parts by pouring successive portions into each of three clear glass bottles or jars, preferably with wide mouths. The bottles or jars shall be provided with air-tight stoppers or with lids which shall be so fastened that spillage or evaporation of the contents is prevented. Each of the bottles or jars shall be so sealed that they cannot be opened without breaking the seal. Each of the bottles or jars shall be marked with the name of the farm feed, date and place of sampling and the sample number together with the name of the inspector taking the sample.

7. In the case of all samples the first part shall be given to the owner or seller of the farm feed or his agent, the second part shall be delivered to an approved analyst for analysis and the third part shall be retained by the inspector for a period of not less than six months after the date on which the report or certificate of analysis is issued.

8. A certificate of sampling (Form FERT 13 in the First Schedule to the Agriculture (Fertilisers) Regulations, 1969) shall be made out in triplicate at the time of sampling and the relevant copies as detailed in the form should accompany each part of the sample.

## **SEVENTH SCHEDULE**

### **Agriculture (Fertilisers and Feed) Act.**

### **Agriculture (Farm Feed) Regulations.**

*(Section 52 (i) and regulation 21)*

## **METHODS OF ANALYSIS**

### **NOTE:**

1. In this Schedule "water" means distilled or purified water except where stated.
2. *Reagents* should be of the appropriate analytical purity.

### **PREPARATION OF SAMPLE**

(a) If the sample is in a fine condition and passes through a sieve having apertures of one millimetre square, it shall be thoroughly mixed and a portion not less than 100 grams in weight shall be placed in a stoppered bottle. From this portion the quantities for analysis shall be taken.

(b) If the sample does not wholly pass through a sieve having apertures of one millimetre square and wholly passes through a sieve having apertures of three millimetres square, it shall be thoroughly mixed and a portion for the determination of the moisture content shall be taken at once.

(c) If the sample is in a coarse condition as, for example, pieces of cake, it shall be carefully pulverised until the whole passes through a sieve having apertures of three millimetres square. It shall then be thoroughly mixed and a portion for the determination of the moisture content shall be taken at once.

(d) From the mixed sample as in (b) above, or from the coarsely crushed sample as in (c) above, a portion not less than 100 grams in weight shall be taken and further powdered and passed through a sieve having apertures of one millimetre square. The portion of the sample so prepared shall be placed in a stoppered bottle and from it the quantities for analysis shall be taken.

(e) If the original sample is appreciably moist, or if for any reason the operations of pulverisation and mixing are likely to result in loss or gain of moisture, the moisture in the bottled portion shall be determined as well as in the portion taken for that purpose under (b) or (c) above in order that the results of the analysis may be corrected to correspond with the original sample as regards moisture.

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

### **DETERMINATION OF MOISTURE**

A weighed quantity of the sample shall be dried at 100°C and then be reweighed.

#### DETERMINATION OF OIL OR FAT (ETHER EXTRACT)

##### *Reagents:*

*Petroleum ether*-light petroleum-b.p. 40°C to 60°C.

NOTE-If ambient temperature of the laboratory demands then petroleum ether- light petroleum-of b.p. 60°C to 80°C may be used.

##### METHOD

Extract 2 to 5 grams of the sample, which has been dried for 1 hour at 100°C, in a Soxhlet extraction apparatus with petroleum ether for a period of at least 6 hours. After evaporation of the solvent, dry the oil or fat for 30 minutes at 100°C, cool in a desiccator and weigh. Calculate the oil or fat percentage as follows:

$$\frac{(A-B) \times 100}{C}$$

where *A* = weight in grams of the Soxhlet flask after extraction

*B* = weight in grams of the Soxhlet flask before extraction

*C* = weight in grams of the sample taken.

#### DETERMINATION OF FIBRE

##### *Reagents:*

*Sulphuric acid solution*-Prepare a solution containing 1.25 grams of sulphuric acid per 100 ml from chemically pure sulphuric acid. (0.255N).

*Sodium hydroxide solution*-Prepare a solution containing 1.25 grams of sodium hydroxide from carbonate-free sodium hydroxide. (0.313N).

*Dilute hydrochloric acid solution* (1:100).

*Ethyl alcohol* (95 per cent v/v).

##### METHOD

Transfer a 2 to 3 gram sample, from which the bulk of the oil or fat has been extracted, to a conical flask (1 litre). Measure 200 ml of the sulphuric acid solution and heat to boiling; add to the flask, connect the flask to a condenser and heat. Bring the contents of the flask to boiling within one minute and boil gently and continuously for exactly 30 minutes. Rotate the flask at intervals of about 5 minutes to mix the contents thoroughly; do not allow any material to adhere to the sides of the flask out of contact with the solution.

At the end of 30 minutes remove the flask and immediately filter with suction through a Whatman No. 54 or No. 541 filter paper (or equivalent) fitted to a Hartley funnel or Buchner funnel. The time of filtration of the bulk of the 200 ml of liquid should not exceed 10 minutes. Wash with boiling water until the washings are free from acid.

Wash the material back into the flask with 200 ml of sodium hydroxide solution (measured at ordinary temperature and heated to boiling point). Boil again for exactly 30 minutes, observing the precautions stated for the acid treatment.

At the end of 30 minutes, remove the flask and immediately either filter direct through a suitable filter crucible or through filter paper and then transfer the charge to a filter crucible

for washing. Wash thoroughly with boiling water, then with dilute hydrochloric acid solution, again with boiling water until free from acid and finally three times with ethyl alcohol.

Dry the crucible and residue for 2 hours at 100°C, cool in a desiccator and weigh. Ignite until free from carbonaceous matter at a temperature not exceeding 600°C, cool in a desiccator and weigh.

Calculate the percentage of fibre as follows:

$$\frac{(A-B) \times 100}{C}$$

Where *A* = weight in grams of residue after drying

*B* = weight in grams of residue after washing

*C* = weight in grams of the sample taken.

#### DETERMINATION OF ASH

Weigh out accurately 2 to 5 grams of the sample into a tared porcelain or silica dish, previously heated to a temperature of 500°C and cooled. Char carefully and ignite in a muffle furnace at a temperature of 500°C for 2 hours or until combustion is complete. Cool in a desiccator and weigh. Calculate the percentage of ash as follows:

$$\frac{A \times 100}{B}$$

Where *A* = weight in grams of ash

*B* = weight in grams of the sample taken.

#### DETERMINATION OF SALT

Mix 5 grams of the sample with 1 gram of pure sodium carbonate or calcium oxide and thoroughly wet the mixture with a little water. Dry the mixture and heat at a temperature not exceeding 500°C in order to destroy organic matter. Extract the residue with hot water, make up the volume to 250 ml and filter. Determine the chlorine in an aliquot part of the filtrate and express the result in terms of sodium chloride (NaCl).

#### DETERMINATION OF PHOSPHORUS

Phosphorus may be determined by either of the matter described below.

#### QUINOLINIUM PHOSPHOMOLYBDATE METHOD

*Reagents:*

*Calcium oxide*-finely ground.

*Citric-molybdate acid solution*-Stir 54 g of molybdenum trioxide (MoO<sub>3</sub>) with 200 ml of water; add 11 g sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdenum trioxide dissolves. Dissolve 60 g citric acid in about 250 to 300 ml water and add 140 ml concentrated hydrochloric acid. Pour the molybdate solution into the acid solution, which is stirred throughout the addition. Then cool and, if necessary, filter the solution through a paper pulp pad./ Dilute the solution to 1 litre. If the solution is slightly green or blue in colour add dropwise a dilute (0.5 or 1 per cent) solution of potassium bromate until the colour is discharged. This reagent should be kept in the dark.

*Hydrochloric Acid*, concentrated (S.G. 1.18).

*Hydrochloric Acid* 25 per cent v/v-Dilute 25 ml of concentrated hydrochloric acid with water

to 100 ml.

*Hydrochloric Acid*, 0.5N.

*Hydrochloric Acid*, 0.1N.

*Indicator Solution*-Mix 3 volumes of thymol blue solution and 2 volumes of phenolphthalein solution prepared as follows:

Thymol blue solution-Dissolve 0.25 g thymol blue in 5.5 ml of 0.1N sodium hydroxide solution and 125 ml industrial methylated spirit. Dilute with water to 250 ml.

Phenolphthalein solution-Dissolve 0.25 g phenolphthalein in 150 ml of industrial methylated spirit and dilute with water to 250 ml.

*Reagents:*

*Nitric Acid*, concentrated (S.G. 1.42).

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

*Sodium Hydroxide*, 5N.

*Sodium Hydroxide*, 0.5N-carbonate free.

*Sodium Hydroxide*, 0.1N-carbonate free.

*Surface Active Agent*-0.5 per cent solution of sodium dodecylbenzene-sulphonate is suitable.

#### 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 water; then slowly add 12 ml of concentrated hydrochloric acid, taking suitable precautions to avoid loss by effervescence, and finally add 5 ml concentrated nitric acid. Heat to incipient boiling and keep at this temperature for 10 minutes. Dilute with about 10 ml water, filter, transfer the insoluble matter to the filter paper with a minimum amount of water and wash twice with small volumes of water. Then transfer the filter paper and insoluble matter to the original capsule or dish and incinerate until all the carbon is destroyed. Combine the ash with the filtrate and heat to boiling point. Cool, transfer to a 250 ml volumetric flask, dilute to the mark, mix well and filter. Discard the first 10 or 20 ml of the filtrate.

#### METHOD

Transfer a volume of the filtrate containing less than 30 mg phosphorus (P) and preferably about 22 mg phosphorus (P) to 500 ml stoppered conical flask marked at 150 ml. Dilute solution with water to 100 ml and 5N sodium hydroxide solution until a faint permanent turbidity or precipitate is formed. Dissolve the precipitate by the dropwise addition of 25 per cent hydrochloric acid, but avoid an excess.

Dilute to 150 ml, add 50 ml of the citric-molybdate solution, heat to incipient ebullition, maintain it at this temperature for 3 minutes and then bring it to 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 (tap) water for 5 minutes, then cool it to 15°C in running tap 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 0.5N 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 the addition of the 0.5N 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 0.5N 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 0.5N hydrochloric acid used from the number of ml of 0.5N sodium hydroxide to ascertain the volume of 0.5N sodium hydroxide equivalent to the phosphorus.

Carry out a blank determination on all the reagents, omitting only the sample, and using 0.1N standard alkali and acid instead of 0.5N for the titration. Calculate the amount of phosphorus in the portion taken for analysis from the factor 1.0 ml of 0.5N sodium hydroxide = 0.597 mg P (1.366 mg P<sub>2</sub>O<sub>5</sub>).

*Reagents:*

*Calcium Oxide*-finely ground.

*Hydrochloric Acid*, concentrated (S.G. 1.18).

*Nitric Acid*, concentrated (S.G. 1.42).

*Potassium Dihydrogen Phosphate Solution* (stock phosphate solution)-Dissolve in water 1.917 g of potassium dihydrogen phosphate previously dried at 105°C for 1 hour and dilute to 1 litre.

*Potassium Dihydrogen Phosphate Solution* (standard phosphate solution)-Dilute 50 ml of stock solution to 250 ml with water. 1 ml of this solution = 0.0874 mg phosphorus (0.2 mg P<sub>2</sub>O<sub>5</sub>).

*Vanado-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.

## STANDARDISATION OF INSTRUMENT

From a burette measure into a series of 100 ml volumetric flasks 25.0, 26.0, 27.0, 28.0, 30.0 and 31.0 ml of the standard phosphate solution (i.e. 0.219, 0.227, 0.236, 0.245, 0.254, 0.262 and 0.271 mg phosphorus, corresponding to 5.0, 5.2, 5.4, 5.6, 5.8, 6.0 and 6.2 mg P<sub>2</sub>O<sub>5</sub>). Add 25 ml of the vanado-molybdate reagent to each flask and dilute to 100 ml with water. Shake and allow to stand for 10 minutes.

Set the spectrophotometer to the correct wavelength, about 4,200  $\text{m}\mu$ , fill two 1 cm cells

with the 0.219 mg solution and check the extinction of the cells. If there is a small difference select the cell with the smaller reading as the standard reference cell.

Determine the apparent extinction (corrected for cell differences) of the 0.227, 0.236, 0.245, 0.254, 0.262 and 0.271 mg phosphorus solutions referred to the 0.219 mg phosphorus solution as standard.

Plot a calibration graph of scale readings against known phosphorus content.

#### ANALYSIS OF THE SAMPLE

Successively dilute a portion of the solution prepared as described under "Dissolution of the Sample" so that the final volume of about 25 ml contains between 2.404 and 2.709 mg of phosphorus (corresponding to 5.5 and 6.2 mg  $P_2O_5$  respectively).

Transfer this final volume to a 100 ml volumetric flask, add 25 ml of the vanado-molybdate reagent, dilute to the mark, mix, and allow to stand for 10 minutes. At the same time transfer 25 ml of the standard phosphorus solution into a second 100 ml volumetric flask. Add 25 ml of the vanado-molybdate reagent, dilute to the mark, mix, and allow to stand for 10 minutes.

Measure the difference in extinction between the two solutions and estimate the phosphorus content of the volume of the unknown solution from the calibration graph. Calculate the phosphorus content of the sample from the known dilution factors and the weight of the sample.

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

#### DETERMINATION OF PROTEIN

The percentage of protein, except in the case of compound cakes or meals and concentrated or high energy farm feed, shall be ascertained by multiplying the percentage of nitrogen, other than nitrogen present as ammoniacal, nitric or urea nitrogen, by 6.25. The presence of nitrogen in these latter forms shall be tested for and the quantity, if any, shall be determined and deducted from the total nitrogen.

In the case of compound cakes or meals and concentrated or high energy farm feed the percentage of protein shall be ascertained by multiplying the percentage of nitrogen, other than nitrogen present as ammoniacal or nitric nitrogen, by 6.25. The presence of nitrogen in these latter forms shall be tested for and the quantity, if any, shall be determined and deducted from the total nitrogen.

#### NITROGEN

##### *Reagents:*

*Mixed Indicator Solution*-Grind together in an agate mortar 0.6 g of methyl red and 0.6 g of methylene blue. Dissolve the mixture in 500 ml of 95 per cent ethanol. Shake, filter and store in a dark glass bottle. *2 per cent Boric acid solution*-Dissolve 20 g of boric acid in water, dilute to 1 litre and add 5 ml of the mixed indicator solution. *Concentrated sulphuric acid* S.G. 1.84.

*Anhydrous sodium sulphate.*

*Cupric sulphate.*

*Paraffin wax.*

*Granulated zinc.*

*Hydrochloric acid 0.1N.*

*Sodium Hydroxide solution-50 per cent w/v.*

#### METHOD

Weigh accurately 1 to 2 g of the sample (or such an amount as shall not contain more than 250 mg of nitrogen) and transfer to a kjeldahl flask. Add 25 ml of concentrated sulphuric acid and approximately 10 g of anhydrous sodium sulphate containing 0.4 g of cupric sulphate. Heat gently until frothing ceases, then strongly until the solution becomes clear and almost colourless. Continue heating for at least another hour. Avoid local overheating. If frothing is excessive, add about 0.5 g paraffin wax.

Dissolve the cooled digest in water and make up to a total volume of about 250 ml. Taking precautions against loss of ammonia add some zinc granules and sufficient 50 per cent sodium hydroxide solution to neutralise the acid and to give about 10 ml in excess, mix well and immediately connect to a distillation apparatus. Distil into an appropriate volume of 2 per cent boric acid solution. Titrate the contents of the receiving flask with 0.1N hydrochloric acid. Carry out a blank test using 2 g of sucrose in the place of the sample, and subtract the titration value of the blank from that of the sample. Express the result in terms of nitrogen.

1 ml of 0.1N hydrochloric acid = 0.0014 g nitrogen.

#### UREA NITROGEN

*Reagents:*

*Activated charcoal.*

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

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

*p-Dimethylaminobenzaldehyde solution*-Dissolve 2 g of p-Dimethylaminobenzaldehyde in 10 ml of concentrated hydrochloric acid and dilute to 100 ml with propan-2-ol.

*Hydrochloric acid-0.02N.*

*Sodium acetate solution*-Dissolve 136 g sodium acetate ( $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ ) in water and dilute to 1 litre.

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

#### METHOD

Weight to the nearest mg about 5 g of the sample (or such an amount as shall contain not more than 250 mg urea) and transfer to a 250 ml volumetric flask. Add 150 ml of 0.02N hydrochloric acid, shake for 30 minutes then add 10 ml of sodium acetate solution and mix well. Add 1 g of activated charcoal (*see Note*) to the flask and shake well, and stand for a further 15 minutes. Add 5 ml of Carrez solution 1, followed by 5 ml of Carrez solution 2 mixing well between additions. Dilute to volume with water and mix well. Filter a portion through a suitable dry filter paper into a dry clean 250 ml beaker. Transfer a 10 ml aliquot of the filtrate to a 50 ml flask, add 10.0 ml of p-dimethylaminobenzaldehyde solution, dilute to 50 ml with water, mix well and allow to stand for 10 minutes. Determine the extinction of the solution at 4,350 using a 1 cm cell against a blank of 10 ml of p-dimethylaminobenzaldehyde reagent diluted to 50 ml with water. Calculate the urea content of the sample by reference to a calibration graph prepared at the same time as the



test sample. (mg urea multiplied by 0.4665 = mg urea nitrogen.)

Establish the calibration graph as follows:

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

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

## DETERMINATION OF SUGAR

*Reagents:*

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

*Copper sulphate solution*-Dissolve 69.28 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) 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 invert sugar. It should be checked by titrating with a solution of pure sucrose (inverted by the procedure described in the note following the paragraph on "Exact Determination" below) using the procedure described in the paragraph.

*Hydrochloric acid N.*

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

*Phenolphthalein indicator solution*-Dissolve 0.25 g of phenolphthalein in 150 ml of industrial methylated spirit and dilute with water to 250 ml.

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

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

*Sodium hydroxide, 10 per cent w/v*-Dissolve 100 g of sodium hydroxide in water and dilute to 1 litre.

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

## PROCEDURE

Preparation of sample.

### WHEN THE SUBSTANCE IS IN SOLID FORM

Weigh to the nearest centigram about 10 g of the sample or a sufficient quantity to contain about 2 g of sugar. Grind in a mortar with hot water (temperature not to exceed 60°C) and transfer to a 500 ml volumetric flask using in all about 400 ml of water. Shake the flask at intervals during 30 minutes. Add 5 ml of potassium oxalate solution to the contents of the flask, followed by 5 ml of zinc acetate solution; mix well and then add 5 ml of potassium ferrocyanide solution, make up with water to 500 ml at the correct temperature, mix well and filter. Determine the sugar in 100 ml of the filtrate by the method described below.

## 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 below.

## DETERMINATION OF THE SUGAR CONTENT

Transfer the measured volume of filtrate obtained as described above to a suitable beaker, add 15 ml of N hydrochloric acid, dilute to 150 ml with water, cover with watch glass and heat to boiling point. Continue to boil for 2 minutes, cool, add 2 or 3 drops of phenolphthalein indicator solution, just neutralise with 10 per cent sodium hydroxide solution, transfer to a 200 ml volumetric flask and dilute to 200 ml. Filter if necessary.

## 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 above, 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.

## EXACT DETERMINATION

To 10 ml of Fehling's solution in a 250 ml conical flask add from burette ( $x-1$ ) ml of the filtrate, together with sufficient water to make a total volume of 60 ml. Heat to boiling point, boil briskly for 1 1/2 minutes and add 3 drops of methylene blue solution. Titrate from the burette at a 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 1 1/2 minutes. Then the total number of ml used in the determination equals the sugar equivalent of 10 ml of Fehling's solution.

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

Not more than 1 ml of filtrate should be required for completion of the titration. If more than 1 ml is required then the determination should be repeated using a more closely calculated volume of filtrate for the original addition. The time taken from the initial boiling point until the end of the titration should be about 3 minutes. If this time is exceeded by more than about 20 seconds, the titration should be repeated.

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

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 suitable 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 per cent sodium hydroxide solution, transfer to a 500 ml volumetric flask, dilute to 50 ml. then follow the procedure described in "Exact Determination" above.

1 ml of the solution = 0.00475 invert sugar, i.e., 10 ml of Fehling's solution = 10.5 ml of this standard invert sugar solution.

## DETERMINATION OF SAND, SILICEOUS MATTER OR OTHER INSOLUBLE MINERAL MATTER

*Reagents:*

*Hydrochloric acid*, concentrated, S.G. 1.18.

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

## PROCEDURE

Weigh to the nearest mg from 2-5 g of the sample and incinerate until all the carbon has been destroyed. (The ash obtained from the method for "Determination of Ash" may be used for this determination.) Moisten with concentrated hydrochloric acid, evaporate to dryness, bake to render the silica insoluble, and then extract repeatedly with hot 25 per cent hydrochloric acid. Filter, wash the insoluble matter and weigh. Regard the quantity obtained as sand and siliceous matter.

## **EIGHT SCHEDULE**

### **Agriculture (Fertilisers and Feed) Act.**

### **Agriculture (Farm Feed) Regulations.**

*(Section 52 (g) and regulation 22)*

### **LIMITS OF VARIATION**

<i>Farm Feed</i>	<i>Limits of Variation</i>
Cereal brans. . . . . per cent  the	Protein, one tenth of the amount stated. Oil, 0.75 or one tenth of the amount stated, whichever is greater. Fibre, one eighth of the amount stated.
Oilseed meals or cakes (undecorticated) . . . . . stated. Oil, 0.75 per cent  the	Protein, one tenth of the amount or one tenth of the amount stated, whichever is greater.
Oilseed meals or cakes (decorticated). . . . . 0.75 per cent  the	Protein, one tenth of the amount stated. Oil, or one tenth of the amount stated, whichever is greater. Fibre, one eighth of the amount stated.
Meat and bone meal . . . . . 0.75 per cent  the  stated.	Protein, one tenth of the amount stated. Oil, or one tenth of the amount stated, whichever is greater. Phosphorus, one tenth of the amount
Bone meal . . . . .	Protein, one tenth of the amount stated.
Meat meal . . . . . Protein, one  one tenth	Phosphorus, one tenth of the amount stated. tenth of the amount stated. Oil, 0.75 per cent or of the amount stated, whichever is the greater.
Fish meal . . . . . per cent	Protein, one tenth of the amount stated. Oil, 0.75 or one tenth of the amount stated, whichever is

the

stated. Salt, 0.75

Molasses . . . . .

Molasses feed . . . . .  
one eighth of the

Mixtures of molasses and urea . .  
equivalent of

Compound cakes and meals, and . .  
stated. Protein, concentrated or

cent or one feed,

one tenth of the

one eighth of

greater. Phosphorus, one tenth of the amount

per cent.

Sugar, one twentieth of the amount stated.

. . Sugar, one tenth of the amount stated. Fibre,  
amount stated.

. . Sugar, one tenth of the amount stated. Protein  
urea, one fifth of the amount stated.

. . . . Protein, one tenth of the amount

high energy farm equivalent of urea, 1.25 per

fifth of the amount stated. Oil, 0.75 per cent or

amount stated, whichever is the greater. Fibre,

the amount stated.