FERTILISERS AND FEEDING STUFFS

1956 No. 1853

The Fertilisers and Feeding Stuffs (Amendment) Regulations, 1956

Made - - - -	*21st November*, 1956
Laid before Parliament	*27th November*, 1956
Coming into Operation	*1st January*, 1957

The Minister of Agriculture, Fisheries and Food and the Secretary of State for Scotland, acting jointly, in exercise of the powers vested in them by Section 23 of the Fertilisers and Feeding Stuffs Act, 1926(a) and of all other powers enabling them in that behalf, and after consultation with the Advisory Committee constituted under the said Section, hereby make the following Regulations:—

Citation and Commencement

1.—(1) These Regulations may be cited as the Fertilisers and Feeding Stuffs (Amendment) Regulations, 1956. The Fertilisers and Feeding Stuffs Regulations, 1955(b), (hereinafter referred to as "the principal Regulations"), and these Regulations may be cited together as the Fertilisers and Feeding Stuffs Regulations, 1955 and 1956.

(2) These Regulations shall come into operation on the first day of January, 1957.

Amendment of Regulations 14 *and* 15 *of the principal Regulations*

2.—(1) Regulation 14 of the principal Regulations shall be amended as follows:—

(*a*) The heading of paragraph (4), namely, "*Determination of phosphoric acid*", shall be followed by the words "Phosphoric acid shall be determined by one or other of the following methods:—

(A) *First method*"

(*b*) Immediately after paragraph (4) there shall be added the following sub-paragraph:

"(B) *Second method*

(*a*) *Soluble phosphoric acid*

The water soluble phosphoric acid shall be extracted by the method described in paragraph (4) (A) (*a*) and the phosphoric acid shall be determined in an aliquot part of the water solution by the method described in paragraph (4) (B) (*e*).

(*b*) *Insoluble phosphoric acid*

The insoluble phosphoric acid shall be determined as a difference figure employing the procedure described in paragraph (4) (A) (*b*), the total phosphoric acid being determined by the method described in paragraph (4) (B) (*f*).

(*c*) *Total phosphoric acid*

(i) *In the absence of organic matter*

5 grams of the finely ground sample (if the phosphoric acid content does not exceed 15 per cent) or 2·5 grams (if the

(a) 16 & 17 Geo. 5. c. 45. (b) S.I. 1955/1673 (1955 I, p. 835).

phosphoric acid content exceeds 15 per cent) shall be placed in a 300 millilitre beaker and shall be mixed with 10 millilitres of water. The beaker shall be covered by a watch glass and 10 millilitres of concentrated hydrochloric acid shall be added slowly and this shall be followed by 5 millilitres of concentrated nitric acid. The beaker and contents shall be heated to incipient ebullition on a hot plate and kept at this temperature for 10 minutes. The contents of the beaker shall be diluted with 100 millilitres of water and shall be boiled for 10 minutes. The solution shall be cooled and transferred to a 500 millilitre volumetric flask and the volume adjusted to the mark. The contents of the flask shall be thoroughly shaken and shall be filtered into a dry beaker through a dry paper. The first 20 millilitres of the filtrate shall be rejected. The phosphoric acid shall be determined in an aliquot part by the method described in paragraph (4) (B) (*f*).

(ii) *In the presence of organic matter*

5 grams of the finely ground sample (if the phosphoric acid content does not exceed 15 per cent) or 2·5 grams (if the phosphoric acid content exceeds 15 per cent) shall be placed in a platinum dish, intimately mixed with 1 gram of calcium oxide, and thoroughly wetted with a little water. The mixture shall be dried and then heated to a temperature not exceeding 500°C until the bulk of the organic matter is destroyed. The residue in the dish after cooling shall be transferred to a 300 millilitre beaker and shall be well mixed with 10 millilitres of water. The beaker shall be covered by a watch glass and 12 millilitres of concentrated hydrochloric acid shall be added slowly and this shall be followed by 5 millilitres of concentrated nitric acid. The beaker and contents shall be heated to incipient ebullition on a hot plate and kept at this temperature for 10 minutes. The contents of the beaker shall be diluted with 100 millilitres of water and shall be boiled for 10 minutes. The solution shall be filtered into a 500 millilitre flask, the insoluble matter washed with hot water, and the insoluble matter and filter paper then transferred to a dish and incinerated to a carbon-free ash. The ash shall be heated for 5 minutes with dilute hydrochloric acid (one part of concentrated acid to four parts of water), then transferred to the filtrate in the 500 millilitre flask, well mixed, cooled, and the volume adjusted to the mark. The contents of the flask shall be thoroughly shaken and shall be filtered into a dry beaker through a dry paper. The first 20 millilitres of the filtrate shall be rejected. The phosphoric acid shall be determined in an aliquot part by the method described in paragraph (4) (B) (*f*).

(*d*) *Citric acid soluble phosphoric acid*

The phosphoric acid soluble in citric acid shall be extracted as described in paragraph (4) (A) (*d*) (except that the sample may not be moistened with alcohol or methylated spirits, but shall be dispersed by vigorous stirring with about 50 millilitres of the citric acid solution for a few minutes before adding the remainder), and the phosphoric acid shall be determined in an aliquot part of the citric acid solution as described below.

The determination shall be carried out on 50 millilitres or 25 millilitres (according to the amount of phosphoric acid (P_2O_5) present) as described in paragraph (4) (B) (*j*) beginning at the words "According to the expected phosphoric acid (P_2O_5)", except that no solid citric acid shall be added before 50 millilitres of the citric molybdate solution are added.

(*e*) *Quinoline molybdate method for determination of water soluble phosphoric acid*

An aliquot part (chosen so as to contain less than 70 milligrams of phosphoric acid (P_2O_5) and preferably about 50 milligrams phosphoric acid (P_2O_5)) of the solution obtained as above described in paragraph (4) (B) (*a*) shall be transferred to a conical flask of about 500 millilitres capacity fitted with a stopper or a rubber bung and marked at 150 millilitres. The aliquot part shall be diluted to 150 millilitres, and 50 millilitres of the citric molybdate reagent (1) (paragraph (4) (B) (*g*)) shall be added. The solution shall be heated to incipient ebullition and kept at this temperature for 3 minutes, then heated to the boiling point. 25 millilitres of the quinoline reagent (paragraph (4) (B) (*h*)) shall be slowly run in from a burette, with constant swirling throughout, the first few millilitres being added dropwise, and the rest in a slow stream keeping the solution gently boiling throughout. The flask shall then be allowed to stand in a bath of boiling water for five minutes, after which it shall be cooled in running water to 15°C. The contents of the flask shall be filtered through a pad of filter paper pulp, and the flask, precipitate and filter shall be washed with successive small volumes of cold water until they are free from acid. The filter-pad and precipitate shall then be transferred to the original flask, using not more than 100 millilitres of water. A few drops of a dilute neutral solution of a surface active agent (e.g. a 0·5 per cent solution of sodium dodecyl benzene sulphonate) may be added if desired to facilitate the dispersal of the precipitate. The flask shall then be stoppered and shaken vigorously until the pulp and precipitate are thoroughly dispersed. The stopper shall be removed and washed with distilled water, the washings being returned to the flask, then an accurately measured volume of 0·5 normal sodium hydroxide solution (free from carbon dioxide) shall be added: the volume added being such that it contains an excess of sodium hydroxide beyond that necessary to dissolve the precipitate. The stopper or bung shall be replaced, and the flask shaken until the precipitate is completely dissolved. The excess of sodium hydroxide shall be titrated with 0·5 normal hydrochloric acid solution, about 0·5 to 1 millilitre of indicator solution (paragraph (4) (B) (*i*)) being added and the end point being taken as the sharp change from green-blue to yellow.

The volume of 0·5 normal hydrochloric acid used shall be deducted from the volume of 0·5 normal sodium hydroxide, previously added, in order to ascertain the volume of 0·5 normal sodium hydroxide equivalent to the phosphoric acid (P_2O_5).

1 millilitre 0·5 normal sodium hydroxide solution is equivalent to 1·366 milligrams of phosphoric acid (P_2O_5). A "blank" determination shall be carried out on all the reagents, omitting only the sample, and using 0·1 normal solutions of acid and

alkali for the titration. The "blank" shall be calculated in terms of 0·5 normal sodium hydroxide and the result first obtained corrected accordingly.

(*f*) *Quinoline molybdate method for the determination of total phosphoric acid in all substances except basic slag*

50 millilitres or 25 millilitres (chosen to contain less than 70 milligrams of phosphoric acid (P_2O_5) and preferably about 50 milligrams phosphoric acid (P_2O_5)) of the solution obtained as above described in paragraph (4) (B) (*c*) shall be transferred to a conical flask of about 500 millilitres capacity fitted with a stopper or a rubber bung and marked at 150 millilitres. The solution shall be diluted to about 100 millilitres and if the sample does not contain calcium, 100 to 200 milligrams of pure calcium carbonate shall be added, then sodium hydroxide solution until a faint permanent precipitate or turbidity is formed. Dilute hydrochloric acid shall be added dropwise until the precipitate just dissolves. The solution shall then be diluted to 150 millilitres and the phosphoric acid determined as described above in paragraph (4) (B) (*e*) beginning at the words "50 millilitres of the citric molybdate reagent (1) (paragraph (4) (B) (*g*)) shall be added".

(*g*) *Preparation of citric molybdate reagent* (1)

54 grams of molybdic anhydride (MoO_3) shall be stirred with 200 millilitres of water and 11 grams of sodium hydroxide until the molybdic anhydride dissolves: the mixture shall be heated to assist solution. 60 grams of citric acid shall be dissolved in a further 300 millilitres of water and 140 millilitres of pure hydrochloric acid (Specific Gravity 1·18) added to it. The molybdate solution shall then be poured into the acid solution, which shall be stirred throughout the addition. The whole shall be cooled and if necessary filtered through a pad of filter paper pulp. The filtrate shall be diluted to a litre and the slight green or blue colour discharged by the dropwise addition of a 1 per cent solution of potassium bromate. This reagent shall be stored in the dark.

(*h*) *Preparation of quinoline reagent*

60 millilitres of concentrated hydrochloric acid shall be diluted to 400 millilitres with water and warmed to between 70 and 80°C. 50 millilitres of quinoline (laboratory reagent quality) shall be poured into the diluted acid in a thin stream with continuous stirring. After the quinoline has dissolved the solution shall be cooled, diluted to one litre with water, and filtered through a pad of filter paper pulp. The pulp shall not be washed.

(*i*) *Preparation of indicator*

0·10 gram of thymol blue shall be dissolved in 2·2 millilitres of 0·1 normal sodium hydroxide solution, 50 millilitres of methylated spirits shall be added and the whole diluted to 100 millilitres with water and mixed well. Three volumes of this solution shall then be mixed with one volume of 0·1 per cent. solution of phenolphthalein in 60 per cent. ethyl alcohol.

(*j*) *Quinoline molybdate method for determination of total phosphoric acid in basic slag*

2·5 grams of the finely powdered sample shall be transferred to a 300 millilitre beaker, thoroughly mixed with 30 millilitres

of water after which a further 70 millilitres of water shall be added with continuous stirring. The mixture shall be warmed, and during stirring, 10 millilitres of concentrated hydrochloric acid shall be added dropwise followed by 5 millilitres of concentrated nitric acid. The whole shall be gently boiled for 10 minutes, cooled, transferred to a 250 millilitre volumetric flask, diluted to the mark and well mixed. This solution shall be filtered through a dry paper into a dry beaker, the first 20 or 30 millilitres of filtrate being rejected.

According to the expected phosphoric acid (P_2O_5) content 50 millilitres or 25 millilitres shall be transferred to a conical flask of about 500 millilitres capacity fitted with a stopper or a rubber bung and marked at 150 millilitres. The solution shall be diluted to about 100 millilitres with water heated almost to boiling and a 20 per cent. solution of sodium hydroxide added drop by drop until there is a faint permanent precipitate. A few drops of hydrochloric acid shall be added to the now boiling solution to redissolve the precipitate. The solution shall be diluted to 150 millilitres, 1 gram of citric acid added and then 50 millilitres of the citric molybdate reagent (2) (paragraph (4) (B) (*k*)). The solution shall be gently boiled for 3 minutes and then 25 millilitres of the quinoline reagent (paragraph (4) (B) (*h*)) shall be slowly added from a burette, the flask being swirled continuously during the addition. The solution shall again be heated to boiling, and boiled gently for 2 minutes, after which it shall be kept hot but not boiling for a further 5 minutes, then cooled to 15°C.

The contents of the flask shall be filtered through a pad of filter paper pulp, and the flask, precipitate and filter shall be washed with successive small volumes of cold water until they are free from acid. The filter pad and precipitate shall be transferred to the original flask, not more than 100 millilitres of water being used in effecting the transfer. A few drops of a dilute neutral solution of a surface active agent (e.g., a 0·5 per cent. solution of sodium dodecyl benzene sulphonate) may be added if desired to facilitate the dispersal of the precipitate. The flask shall be stoppered and well shaken until the pulp and precipitate are thoroughly dispersed. The stopper shall be removed and washed with distilled water, the washings being returned to the flask, then an accurately measured volume of 0·5 normal sodium hydroxide solution (free from carbon dioxide) shall be added, the volume added being such that it contains an excess of sodium hydroxide beyond that necessary to dissolve the precipitate. The flask shall be shaken until the precipitate is completely dissolved, and the excess of sodium hydroxide titrated with 0·5 normal hydrochloric acid solution, about 0·5 to 1 millilitre of indicator solution (paragraph (4) (B) (*i*)) being added and the end point being taken as the sharp change from green-blue to yellow.

The volume of 0·5 normal hydrochloric acid used shall be deducted from the volume of 0·5 normal sodium hydroxide previously added, in order to ascertain the volume of 0·5 normal sodium hydroxide equivalent to the phosphoric acid (P_2O_5).

1 millilitre 0·5 normal sodium hydroxide solution is equivalent to 1·366 milligrams of phosphoric acid (P_2O_5). A "blank" determination shall be carried out on all the reagents, omitting only the sample, and using 0·1 normal solutions of acid and

alkali for the titration. The "blank" shall be calculated in terms of 0·5 normal sodium hydroxide, and the result first obtained corrected accordingly.

(*k*) *Preparation of citric molybdate reagent* (2)

This reagent shall be prepared in the manner described in paragraph (4) (B) (*g*) except that 120 grams of pure citric acid shall be used instead of 60 grams."

(*c*) At the end of sub-paragraph (1) (*b*) (i) of paragraph (5) for the expression "(5) (1) (*d*) (i)" shall be substituted the expression "(5) (1) (*b*) (i)".

(*d*) In the last sentence of the second of the two paragraphs headed "*Preparation and Standardisation of one twentieth normal disodium ethylenediamine tetracetate solution*" in paragraph (8) the figures "2·5" shall be substituted for the figures "2·4".

(2) Regulation 15 of the principal Regulations shall be amended as follows:—

For paragraph (5) there shall be substituted the following paragraph:

"(5) *Determination of phosphoric acid*

Either

(i) A weighed portion of the sample shall be heated with concentrated sulphuric acid until all organic matter is destroyed and the phosphoric acid is completely in solution. After dilution, the solution shall be filtered, the insoluble matter thoroughly washed and the filtrate made up to a definite bulk. The phosphoric acid shall be determined by the method described in Methods of Analysis of Fertilisers, paragraph (4) (A) (*e*), in an aliquot part of the solution, which shall first be nearly neutralised and then acidified with nitric acid.

Or, alternatively,

(ii) A weighed portion of the sample shall be treated as described in Methods of Analysis of Fertilisers, paragraph (4) (B) (*c*) (ii), and the phosphoric acid in an aliquot part of the solution obtained thereby shall be determined by the method described in paragraph (4) (B) (*f*)".

In Witness whereof the Official Seal of the Minister of Agriculture, Fisheries and Food is hereunto affixed this twenty-first day of November, nineteen hundred and fifty-six.

(L.S.) *D. Heathcoat Amory.*

Given under the Seal of the Secretary of State for Scotland, this twenty-first day of November, nineteen hundred and fifty-six.

(L.S.) *James Stuart.*

EXPLANATORY NOTE

(This Note is not part of the Regulations, but is intended to indicate their general purport.)

The Fertilisers and Feeding Stuffs Regulations, 1955, prescribe methods for the analysis of fertilisers and feeding stuffs. These amending Regulations provide an alternative method for the determination of phosphoric acid. They also correct two typographical errors in the earlier Regulations.