SRI LANKA STANDARD 645 : PART 5 : 1985

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# METHOD OF TEST FOR FERTILIZERS

## PART 5 - DETERMINATION OF PHOSPHOROUS CONTENT

SRI LANKA STANDARDS INSTITUTION

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SLS 645:Part 5:1985

Gr.5

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# SRI LANKA STANDARD
# METHODS OF TEST FOR FERTILIZERS
## PART 5 : DETERMINATION OF PHOSPHOROUS CONTNET

## FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1985-10-11, after the draft, finalized by the Drafting Committee on Fertilizers, had been approved by the Agricultural and Food Products Divisional Committee.

This part is one of a series of standards on testing of fertilizers. Other parts covering the determination of nitrogen, moisture, biuret and potassium are being issued. A complete list of standards may be obtained from the Sri Lanka Standards Institution.

The values used in this standard are given in SI units.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with CS 102.

In the preparation of this standard, the assistance derived from the Fertilizers and Feeding Stuffs Regulations of the United Kingdom (1968), is gratefully acknowledged.

## 1 SCOPE

This part of the standard prescribes methods for the determination of phosphorous in its various forms in fertilizers including fertilizer mixtures.

## 2 REFERENCES

| | |
|---|---|
| CS 102 | Presentation of numerical values |
| SLS 559 | Sampling of fertilizers |

## 3 PRINCIPLE

The acid solution containing orthophosphate is treated with an acid reagent containing molybdic acid and vanadic acid. A stable orange-yellow coloured complex of vanadomolybdiphosphoric acid ($H_3PO_4 \cdot VO_3 \cdot 11\ MoO_3 \cdot n.H_2O$) is formed. The maximum absorption of monochromatic light is measured at a wavelength of 420 nm.

## 4 PREPARATION OF TEST SAMPLE

Reduce the test sample as specified in Clause 6 of SLS 559:1972, to a quantity sufficient for analysis and grind not less than 225 g of the reduced sample without previously sieving. For fertilizer materials and moist fertilizer mixtures, grind to pass through a sieve of 1-mm aperture size. For dry mixtures that tend to segregate, grind to pass through a sieve of 350-µm aperture size. Grind as rapidly as possible to avoid loss or gain of moisture during the operation. Mix thoroughly and store in tightly stoppered bottles.

## 5 REAGENTS

Unless specified otherwise, chemicals of a recognised analytical grade and distilled water shall be employed in tests.

5.1 *Calcium oxide*, finely ground.

5.2 *Citric acid*, monohydrate.

5.3 *Hydrochloric acid*, concentrated, (d = 1.18).

5.4 *Nitric acid*, concentrated (d = 1.42).

5.5 *Stock phosphate solution*. Dissolve in water 1.9173 g of potassium dihydrogen phosphate previously dried at 105 °C for two hours and dilute to 1 litre. (1 ml of this solution contains 1 mg $P_2O_5$).

5.6 *Standard phosphate solution*. Dilute 50 ml of stock phosphate solution to 250 ml with water. (1 ml of this solution is equivalent to 0.2 mg $P_2O_5$).

5.7 *Sodium hydroxide*, c(NaOH) = 1 mol/litre.

5.8 *Vanado-molybdate reagent*. Dissolve separately 20 g of ammonium molybdate and 1 g of ammonium vanadate in hot water, cool, mix and acidify with 140 ml concentrated nitric acid and dilute to 1 litre.

## 6 APPARATUS

6.1 A spectrophotometer with matched 1-cm cells and a monochromator giving a source of light with a wavelength of 420 nm is required.

## 7 DETERMINATION OF TOTAL PHOSPHOROUS CONTENT (As $P_2O_5$)

### 7.1 Standardization of instrument

Measure from a burette, into a series of 100-ml volumetric flasks, 25.0 ml, 26.0 ml, 27.0 ml, 28.0 ml, 29.0 ml, 30.0 ml and 31.0 ml of the standard phosphate solution (see 5.6) containing 5.0 mg, 5.2 mg, 5.4 mg, 5.6 mg, 5.8 mg, 6.0 mg and 6.2 mg of $P_2O_5$ respectively. Within 5 minutes for the entire series, add 25 ml of the vanadomolybdate reagent (see 5.8) into each flask and dilute to 100 ml, ensuring that the temperature of the reagent and dilution water is maintained at 27±2 °C. Mix and allow to stand for 10 min.

If a single beam spectrophotometer is used, set the spectrophotometer to a wavelength of 420 nm, fill two 1-cm cells with the standard solution containing 5.0 mg $P_2O_5$ to which the vanadomolybdate reagent has been added as given above and check absorbance of the cells. If there is a small difference, select the cell with the lower reading as the standard reference cell.

Using the other cell (sample cell), determine the apparent absorbance at 27±2 °C (corrected for cell differences) of the other standard solutions containing 5.2 mg, 5.4 mg, 5.6 mg, 5.8 mg, 6.0 mg and 6.2 mg $P_2O_5$ referred to the solution containing 5.0 mg $P_2O_5$ as the standard. After each determination, empty and refill the cell containing the 5 mg standard solution and readjust to zero absorbance to avoid errors that might arise from temperature changes. Plot a calibration graph of absorbance against concentration in mg $P_2O_5$ per 100 ml of standard solution.

### 7.2 Dissolution of sample

#### 7.2.1 *In the case of fertilizers and fertilizer mixtures other than basic slag*

#### 7.2.1.1 In the absence of organic matter

Weigh, to the nearest milligram, about 5 g of the sample prepared as in 3 (mass $m$ grams) 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 (volume $V_0$ ml). Mix well and filter the solution through a dry filter paper into a dry flask, discarding the first 20 ml. Retain the rest of the filterate.

#### 7.2.1.2 In the presence of organic matter

Weigh, to the nearest milligram, about 5 g of the sample prepared as in 3 (mass $m$ grams) into a dish of about 50 mm in diameter; add 1 g of calcium oxide and mix well with a thin glass rod. Calcine the mixture at a temperature not exceeding 500 °C to destroy the organic matter. Allow to cool, transfer the contents to a 400-ml beaker, add 100 ml water, stir thoroughly and heat to boiling point. Add slowly to the boiling solution 10 ml of hydrochloric acid and then 10 ml of concentrated nitric acid, and boil gently. If the solution is clear, continue to boil gently for 10 min, cool, transfer to a 500-ml volumetric flask and dilute to the mark with water (volume $V_0$ ml).

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 into a dish and calcine until all the carbon is destroyed. Allow to cool and transfer the contents to the filterate; heat to boiling and gently boil for 10 minutes; cool, transfer to a 500-ml volumetric flask, dilute to mark and filter (volume $V_0$ ml).

7.2.2 *In the case of basic slag.*

Weigh, to the nearest milligram, about 2.5 g of the sample prepared as in 3 (mass *m* grams) into a 400-ml beaker, wet the solid thoroughly with about 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 and 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 (volume $V_0$ ml). Mix well and filter the solution through a dry medium-fine filter paper into a dry beaker, rejecting the first 30 ml of the filterate.

### 7.3 Procedure

7.3.1 Successively dilute a known volume ($V_1$ ml) of the solution prepared as in **7.2** so that the final volume of about 25 ml contains between 5.5 mg and 6.2 mg $P_2O_5$, taking care that the water used for dilution is at a temperature of 27±2 °C.

7.3.2 Transfer this final volume to a 100-ml volumetric flask, add 25 ml of the vanadomolybdate reagent (at a temperature of 27±2 °C); dilute to mark; mix and allow to stand for 10 minutes. At the same time, transfer 25 ml of the standard phosphate solution (see 5.6) at 27 ± 2 °C into a second volumetric flask. Add 25 ml of the vanadomolybdate reagent (at 27±2 °C) dilute to mark, mix and allow to stand for 10 minutes. (This solution contains 5.0 mg of $P_2O_5$).

7.3.3 Measure the difference in absorbance at 27±2 °C between the two solutions and estimate the $P_2O_5$ content of the sample solution from the calibration graph constructed as in 7.1.

### 7.4 Calculation

$$P_2O_5 \text{ content, per cent by mass} = \frac{y}{1000} \times \frac{V_0}{V_1} \times \frac{100}{m}$$

where,

$m$ is mass, in g, of sample;

$V_0$ is volume, in ml, of initial sample solution;

$V_1$ is volume, in ml, of sample solution diluted for test; and

$y$ is graph reading, in mg $P_2O_5$ per 100 ml, of sample solution.

## 8 DETERMINATION OF WATER SOLUBLE PHOSPHOROUS CONTENT

### 8.1 Dissolution of sample

Weigh, to the nearest 0.01 g, about 10 g (mass $m$ grams) of the sample prepared as in 3 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 with water, mix well and filter (volume $V_0$ ml).

### 8.2 Procedure

To 25 ml of the solution (volume $V_1$ ml) prepared as in 8.1, add 1 ml of concentrated nitric acid; heat to incipient ebullition on a hotplate and maintain the solution at this temperature for 10 minutes. Cool, neutralize with sodium hydroxide (see 5.7) solution. Successively dilute this solution so that a final volume of about 25 ml contains between 5.5 mg $P_2O_5$ and 6.2 mg $P_2O_5$. Continue determination as in 7.3.2.

### 8.3 Calculation

Calculate the water soluble $P_2O_5$ content as given in 7.4.

## 9 DETERMINATION OF WATER INSOLUBLE PHOSPHOROUS CONTENT

Determine the water insoluble phosphorous content as the difference between the total phosphorous content determined as in 7 and the water soluble phosphorous content determined as in 8.

## 10 DETERMINATION OF CITRIC ACID SOLUBLE PHOSPHOROUS CONTENT (As $P_2O_5$)

### 10.1 Standardization of instrument

Standardize the instrument as given in 7.1.

*NOTE - In the preparation of the standard phosphate solutions, add to each flask, a volume of citric acid equal to the amount contained in the diluted sample solution mentioned in* 10.3.1 *prior to the addition of the vanadomolybdate reagent.*

## 10.2 Dissolution of sample

Weigh, to the nearest milligram, about 1 g of the sample prepared as in 4 into a 250-ml conical flask, add 100 ml of freshly prepared 2 per cent citric acid, stopper and shake for 30 minutes using a mechanical shaker (Set at a shaking speed of twice per second), filter immediately.

## 10.3 Precedure

**10.3.1** Successively dilute a portion of the solution (volume $V_1$ ml) prepared as in **10.2**, so that a final volume of about 25 ml contains between 5.5 mg $P_2O_5$, and 6.2 mg $P_2O_5$, taking care that the dilution water is at 27±2 °C.

**10.3.2** Transfer this solution to a 100-ml volumetric flask, add 25 ml of the vanadomolybdate reagent (at a temperature of 27±2 °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 27±2 °C) into a second 100-ml volumetric flask . Add sufficient citric acid such that when the solution is made up to 100 ml, its concentration is equal to that of the diluted sample solution. Add 25 ml of the vanadomolybdate reagent (at 27±2 °C), dilute to mark, mix and allow to stand for 10 minutes.

**10.3.3** Measure the difference in absorbance at 27±2 °C between the two solutions and estimate the $P_2O_5$ content of the sample solution using the calibration graph.

## 10.4 Calculation

$$\text{Citric acid soluble } P_2O_5 \text{ content, per cent by mass} = \frac{y}{1000} \times \frac{100}{V_1} \times \frac{100}{m}$$

where,

$y$ is graph reading, in mg/100 ml;

$V_1$ is volume, in ml, of sample solution diluted for test (see **10.3.1**); and

$m$ is mass, in g, of the sample.

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