

SRI LANKA STANDARD 645 : PART 4 : 1989

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**METHODS OF TEST FOR
FERTILIZERS**

PART 4 - DETERMINATION OF POTASSIUM CONTENT

SRI LANKA STANDARDS INSTITUTION

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SLS 645:Part 4:1989

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SRI LANKA STANDARD
METHODS OF TEST FOR FERTILIZERS
PART 4 - DETERMINATION OF POTASSIUM CONTENT

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1989-12-14, after the draft, finalized by the Drafting Committee on Fertilizers, had been approved by the Chemicals 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 phosphorus have been issued.

This part of the standard consists of two sections as follows:

Section 1 : Determination of potassium content - Flame photometric method.

Section 2 : Determination of potassium content - Potassium tetraphenylborate gravimetric method.

Section 2 of this standard is technically identical to the corresponding method published by the International Organization for Standardization. The text of this International Standard has been accepted as suitable for publication without deviation as a Sri Lanka Standard. Certain terminology and conventions are not however identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

- a) Wherever the words *International Standard* appear referring to a particular standard, they should be interpreted as *Sri Lanka Standard*.
- (b) The comma has been used throughout as a decimal marker in International Standards. In Sri Lanka Standards, it is the current practice to use a full point on the base line as the decimal marker.

All standard values given in this standard are in SI units.

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

In the preparation of this standard, the assistance obtained from the publications of the International Organization for Standardization and the Fertilizers and Feeding Stuffs Regulation of United Kingdom (1978) is gratefully acknowledged.

1 SCOPE

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

2 REFERENCES

- CS 102 Presentation of numerical values.
- CS 124 Test sieves.
- SLS 559 Sampling of fertilizers.

3 DEFINITIONS

For the purpose of this standard, the following definitions shall apply:

3.1 repeatability : A quantitative measure of the variability associated with a single operator in a given laboratory obtaining successive repeat results on the same apparatus. It is defined as that difference between two such single results that would only be exceeded in the long run in one case in twenty in the normal and correct operation of the test method.

3.2 reproducibility : A quantitative measure of variability associated with operators working in different laboratories, each obtaining single result on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

4 PREPARATION OF THE TEST SAMPLE

Reduce the test sample specified as in Clause 6 of SLS 559:1982, to a quantity sufficient for analysis. 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- μ m 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.

SECTION 1 : DETERMINATION OF POTASSIUM CONTENT -
FLAME PHOTOMETRIC METHOD

1 PRINCIPLE

The sample is ashed and dissolved in dilute hydrochloric acid or, if it does not contain organic substances, it is dissolved directly in dilute hydrochloric acid. The solution is diluted and the potassium content of the extract is determined by flame photometry.

2 REAGENTS

Unless specified otherwise, reagents of analytical grades and distilled water or water of equivalent purity shall be used.

2.1 *Hydrochloric acid*, concentrated (rel.den.=1.18).

2.2 *Ammonium oxalate*, saturated solution.

2.3 *Ammonia*, 30 per cent (V/V) solution, prepared by using concentrated ammonia solution (rel. den. = 0.88).

2.4 *Potassium stock solution*

Dissolve 3.4807 g of potassium dihydrogen phosphate, previously dried for one hour at 105 °C, in water and dilute to 1 litre.
One millilitre of this solution contains 1 mg of potassium.

2.4.1 *Potassium working solution*

Dilute 50 ml of stock solution (2.4) to 1 litre.
One millilitre of this solution contains 0.05 mg of potassium.

3 APPARATUS

Flame photometer

4 PROCEDURE

4.1 Preparation of solutions

4.1.1 *Test solution*

4.1.1.1 Fertilizers containing little or no organic matter

Weigh, to the nearest 0.001 g, 2.5 g of the prepared test sample and transfer to a 400-ml beaker. Add 50 ml of water and 5 ml of hydrochloric acid (2.1). Evaporate to dryness on a steam bath. Add to the residue, 125 ml of water and 50 ml of ammonium oxalate solution (2.2). Boil for 30 minutes. If necessary, add a small quantity of potassium free antifoaming agent. Cool the mixture, add a slight excess of ammonia solution (2.3) and allow to cool. Transfer to a 250-ml graduated flask, dilute to the mark with water, mix and filter through a fast medium filter paper.

4.1.1.2 Fertilizers containing organic matter

Weigh, to the nearest 0.01 g, 10 g of the prepared test sample into a suitable crucible and place in a muffle furnace. Gradually raise the temperature to about 475 °C (not exceeding 500 °C). Maintain at this temperature for at least 16 hours and then open the furnace and allow the crucible to cool. Grind the residue to eliminate any lumps. Add 50 ml of water and 10 ml of hydrochloric acid (2.1). Evaporate to dryness on a steam bath. Add to the residue, 125 ml of water and 50 ml of ammonium oxalate solution (2.2). Boil for 30 minutes. Cool the mixture, add a slight excess of ammonia solution (2.3) and allow to cool. Transfer to a 500-ml graduated flask, dilute to the mark with water, mix and filter through a medium fast paper.

4.1.2 *Blank solution*

Prepare a blank solution omitting the sample.

4.1.3 *Dilute sample and blank solutions*

Dilute, test solutions (4.1.1.1 or 4.1.1.2) and the blank solution (4.1.2) to a concentration within the optimal measuring range of the flame photometer.

4.1.4 *Standards solutions*

Dilute, the potassium working solution (2.4.1) to obtain at least five standard solutions of increasing concentration within the optimal measuring range of the flame photometer.

4.2 Measurement

Set the flame photometer to measure the potassium emission according to the manufacturer's instructions. Aspirate successively, in triplicate, the standard solutions (4.1.4) the sample solution and the blank solution (4.1.3), washing with water between each aspiration. Plot the calibration curve using the median emissions of the standard solutions against concentrations of potassium. Determine the concentration of potassium in the sample solution and the blank solution by reference to the calibration curve.

NOTE

The concentration of potassium in the sample solution may be confirmed as follows.

Prepare two further standard potassium solutions to contain 1 mg/l more and 1 mg/l less than the estimated potassium content of the dilute sample solution. Aspirate successively, the standard solution containing 1 mg/l less, the dilute sample solution and the standard solution containing 1 mg/l more. Take the median result of each of the three readings and calculate the potassium content of the sample solution.

5 CALCULATION

Calculate the percentage potassium content of the sample as K taking into account, the mass of the test sample, the dilution carried out in the course of the analysis and the results of the blank test.

NOTE

Conversion factor for K to K_2O is 1.204.

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ISO 5318:1983

SECTION 2 : DETERMINATION OF POTASSIUM CONTENT -
POTASSIUM TETRAPHENYLBORATE GRAVIMETRIC METHOD
(Reference method)

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Further particulars of the terms and conditions of the permit may be obtained from the Sri Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.



SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science & Technology.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.