

SRI LANKA STANDARD 645 : PART 7 : 1994

UDC 631.8 : 543 . 217.5

METHOD OF TEST FOR FERTILIZERS
PART 7 : DETERMINATION OF SODIUM CONTENT

SRI LANKA STANDARDS INSTITUTION

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Gr. 5

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SRI LANKA STANDARD
METHODS OF TEST FOR FERTILIZERS
PART 7 : DETERMINATION OF SODIUM CONTENT

FOREWORD

This standard was approved by the Sectoral Committee on Chemical and Chemical Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1994-12-22.

This part is one of the series of standards on testing of fertilizers. Other parts which have already been issued cover the determination of nitrogen, moisture, biuret, potassium, phosphorous, calcium and magnesium.

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

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

Section 2 : Determination of sodium content - Atomic absorption spectrophotometric method.

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 SLS 102.

In the preparation of this standard, the valuable assistance derived from the following publications is gratefully acknowledged:

(i) Association of Official Analytical Chemists

1 SCOPE

This standard prescribes methods for the determination of sodium content in fertilizers including fertilizer mixtures.

2 REFERENCES

- | | |
|---------|----------------------------------|
| SLS 102 | Presentation of numerical values |
| SLS 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 a 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 prior sieving. For fertilizer materials and moist fertilizer mixtures, grind to pass through a sieve of 1mm 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 SODIUM CONTENT-FLAME PHOTOMETRIC METHOD

1 PRINCIPLE

The sample is boiled with water and ammonium oxalate and filtered. The filtrate is diluted and the sodium content 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 Ammonium oxalate solution

Dissolve 40 g of ammonium oxalate in water and dilute to 1 litre.

2.2 Sodium chloride, dried at 105 °C to a constant mass

3 APPARATUS

3.1 *Flame photometer*

4 PROCEDURE

4.1 Preparation of the sample solution

Weigh, to the nearest milligram, 2.5 g (if sample contains less than 4 per cent sodium) or 1.25 g (if sample contains 4 per cent to 20 per cent sodium) of the prepared test sample into 250-ml beaker. Add 125 ml of water and 50 ml of ammonium oxalate solution (2.1) and boil for 30 minutes. Cool the mixture and transfer the content into 250-ml volumetric flask. Dilute to the mark with water, mix and filter through a dry filter paper. (Whatman No. 42 or equivalent).

4.2 Sodium stock solution

Dissolve 1.2711 g of dried sodium chloride (2.2) in water and dilute to 500 ml.

4.3 Sodium standard solution series

Depending on the sodium content of the sample prepare the dilution series as follows:

(a) 1 per cent or more sodium content prepare standard solutions by diluting to cover the range 0 ppm to 40 ppm sodium at intervals of less than or equal to 5 ppm.

(b) Less than 1 per cent sodium content prepare standard solutions by diluting to cover the range of 0 ppm to 10 ppm sodium at intervals of 2 ppm.

4.4 Preparation of standard curve

Atomize portions of standard solutions until readings for series of standard solutions are reproducible. Prepare standard curve of emission against concentrations of sodium.

NOTE

Add appropriate amount of lithium nitrate, if internal standard instrument is to be used.

4.5 Determination

Transfer 25 ml (less than 4 per cent sodium) or 10 ml (4 per cent to 20 per cent sodium) sample solutions to 250-ml volumetric flask. Dilute to volume with water and mix. (if internal standard instrument is used, add required amount of lithium nitrate before diluting to volume). Atomize portions of sample several times to obtain reliable average readings for each solution. Determine the sodium content from standard curves (a) or (b).

4.6 Calculation

4.6.1 If sodium content is 0 per cent to 4 per cent;

$$\text{Sodium content as Na, per cent by mass} = \frac{250 A}{m}$$

4.6.2 If sodium content is 4 per cent to 20 per cent;

$$\text{Sodium content as Na, per cent by mass} = \frac{625 A}{m}$$

Where,

m is the mass of sample, in mg

A is the sodium content in the sample, in mg per litre

4.6.3 Sodium content as NaCl, per cent by mass = Na per cent x 2.543

NOTE

Conversion factor for Na to NaCl is 2.543

SECTION 2 : DETERMINATION OF SODIUM CONTENT-ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

1 PRINCIPLE

The sample is boiled with water and ammonium oxalate and filtered. The filtrate is used to determine the sodium content by atomic absorption spectrophotometry.

2 REAGENTS

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

2.1 Ammonium oxalate solution

Dissolve 40 g of ammonium oxalate in water and dilute to 1 litre.

2.2 Sodium chloride, dried at 105°C to a constant mass.

3 APPARATUS

3.1 Atomic absorption spectrophotometer, with air-acetylene flame, wavelength 330.3 nm.

4 PROCEDURE

4.1 Preparation of the sample solution

Weigh, to the nearest milligram 2.5 g (if sample contains less than 4 per cent sodium) or 1.25 g (if sample contains 4 per cent to 20 per cent sodium) of the prepared test sample into 250-ml breaker. Add 125 ml of water and 50 ml of ammonium oxalate solution (2.1) and boil for 30 minutes. Cool the mixture and transfer the content into 250-ml volumetric flask. Dilute to the mark with water, mix and filter through a dry filter paper. (Whatman No. 42 or equivalent)

If the solution contains less than 1 per cent sodium, use the same solution for the determination. In the case of solutions containing 1 per cent to 20 per cent sodium, place 20 ml in 100 ml volumetric flask. Dilute to volume with water and mix. Use this solution for the determination.

4.2 Preparation of the standard curve

Dissolve 2.5421g of dried sodium chloride (2.2) in water and dilute to 1 litre. Prepare a series of standard solutions to cover the range of 0 ppm to 200 ppm at intervals of less than or equal to 20 ppm sodium.

4.3 Determination

Set the spectrophotometer (3.1) and aspirate standard solutions and sample solution.

Plot the standard curve of absorbance against concentrations of the standard solutions. Determine the sodium content from the standard curve.

4.4 Calculation

4.4.1 If sodium content in the solution is less than 1 per cent;

$$\text{Sodium content as Na, per cent by mass} = \frac{25 A}{m}$$

4.4.2 If sodium content in the solution is 1 per cent to 20 per cent;

$$\text{Sodium content as Na, per cent by mass} = \frac{125 A}{m}$$

Where,

m is the mass of sample, in mg.

A is the sodium content in the sample, in mg per litre.

4.4.3 Sodium content as NaCl, per cent by mass = Na per cent x 2.543

NOTE

Conversion factor for Na to NaCl is 2.543

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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.



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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.

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