SRI LANKA STANDARD 645 : PART 3 : 2009
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METHODS OF TEST FOR FERTILIZERS

PART 3 : DETERMINATION OF BIURET CONTENT

(First Revision)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 645 : Part 3 : 2009

Gr. 5

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This standard does not purport to include all the necessary provisions of a contract.

Sri Lanka Standard
METHODS OF TEST FOR FERTILIZERS
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FOREWORD

This standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2009-11-30.

This standard was first published in 1986. In this First Revision, test methods specified in IS 6092 : Part 5 : 1985 (2001) had been considered to identify the test methods for the determination of biuret content of fertilizers.

This part is one of a series of standards on testing of fertilizers. Other parts cover the determination of nitrogen, moisture, potassium, calcium, magnesium, sodium and phosphorus.

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

Method 1: Determination of biuret content- Atomic Absorption Spectrophotometric method

Method 2 : Determination of biuret content - Colourimetric method

Method 1 is recommended for use as reference method.

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

In reporting the result of a test as 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 **SLS 102**.

In the preparation of this standard the assistance derived from the following publication is gratefully acknowledged :

IS 6092 : Part 5 : 1985 (2001) Indian Standard Methods of sampling and test for fertilizer
Part 5: Determination of secondary elements and micronutrients

1 SCOPE

This part of the standard prescribes the methods for the determination of biuret in fertilizers including fertilizer mixtures.

2 REFERENCES

SLS 102 Rules for rounding off numerical values
SLS 559 Sampling of fertilizers

3 QUALITY OF REAGENTS

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4 PREPARATION OF TEST SAMPLE

Reduce the test sample specified as in Clause **6** of **SLS 559: 1982** 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 METHODS OF TEST

5.1 Method 1 - Determination of biuret content- Atomic Absorption spectrophotometric method

5.1.1 *Field of application*

This method is intended for determining biuret content in urea and mixed fertilizers.

5.1.2 *Apparatus*

5.1.2.1 Atomic absorption spectrophotometer

5.1.3 *Reagents*

5.1.3.1 Copper sulphate solution, dissolve 15 g of copper sulphate pentahydrate in water and dilute it to 1 litre. The solution should be capable of hydrolyzing 0.1 g urea per 200 ml solution.

5.1.3.2 Buffer solution, dissolve 24.6 g of potassium hydroxide and 30 g of potassium chloride in water and dilute it to 1 litre. Check the pH and adjust to 13.4.

5.1.3.3 Starch solution, take 1 g of starch (soluble) and add 10 ml cold water, triturate to a thin paste, and pour gradually into 150 ml boiling water containing 1 g oxalic acid. Boil until solution clears, cool, and dilute it to 200 ml. Prepare fresh solution every week.

5.1.3.4 Bromocresol purple indicator, dissolve 0.1 g bromocresol purple in 19 ml of 0.1 mol dm^{-3} sodium hydroxide and dilute to 250 ml with water.

5.1.3.5 Biuret -Pure biuret with guaranteed composition if not available to recrystallize, weigh approximately 10 g reagent grade biuret, transfer it to 1-litre beaker, add 500 ml of water, and heat on hot plate with occasional stirring until dissolved. Boil slowly until volume decreases to about 250 ml. Remove, and let it cool gradually to room temperature. Filter through fritted-glass funnel, transfer it to evaporating dish, place in an oven at 105^0 C to 110^0 C and dry for 1 hour. Remove it form oven, place it into a desiccator, and cool to room temperature.

NOTE : *If the biuret is contaminated with cyanuric acid and/ or urea, it should be purified, as follows. Transfer 5* g *of biuret to a 100-* ml *beaker, add 15* ml *of ammonia (10 per cent,* m/m*) and stir for 15* minutes*.(Cyanuric acid and urea dissolve. Biuret is practically insoluble). Pass the solution through a 1 G 3- filter crucible and wash the crucible twice with 5* ml *of water and subsequently, three times with 10* ml *acetone. Finally, dry for 3* h *at 105* ^{0}C *and keep the biuret in a brown wide mouth bottle with a ground-in stopper.*

5.1.3.6 Biuret standard solution (0.4 mg/ml), dissolve 0.4000 g recrystalized biuret in warm water, cool, transfer it to 1-litre flask, and dilute it up to the mark.

5.1.3.7 Copper stock solution (1 000 µg Cu/ml), dissolve 1.000 g pure copper metal in minimum amount of concentrated nitric acid and add 5 ml of concentrated hydrochloric acid. Evaporate it almost to dryness and dilute it to 1 litre with 0.1 mol dm^{-3} hydrochloric acid.

5.1.3.8 Copper standard solution, dilute aliquots of copper stock solution as prepared **5.1.3.7** with water to obtain more than equal to 4 standard solutions within range of determination, 1 µg to 4 µg, copper/ml in the final solution.

5.1.3.9 Hydrochloric acid, 1 $moldm^{-3}$

5.1.4 *Determination of calibration factor*

5.1.4.1 Transfer aliquots of biuret standard solution containing 4 mg, 8 mg and 12 mg biuret separately to 100-ml volumetric flasks, dilute to about 30 ml with water, and add 25 ml of ethanol to each.

5.1.4.2 While stirring with magnetic stirrer, add 20 ml starch solution, 10 ml of copper sulphate solution, and 20 ml of buffer solution. Remove stirring bar, rinse, dilute it to the mark, mix thoroughly and let it stand for 10 minutes. Filter under vacuum about 50 ml through dry 150 ml medium porosity fritted glass funnel into a dry flask. Transfer 25 ml aliquots of each filtrate to 250-ml volumetric flasks, acidify with 5 ml of 1 mol dm^{-3} hydrochloric acid, and dilute it to the volume with water. Proceed as given in Appendix **A** using copper standard solution to determine complexed copper in solution by atomic absorption spectrophotometer after adding equivalent amounts of ethanol potassium hydroxide solution, buffer solution, and 1 mol dm^{-3} of hydrochloric acid. Take more than three readings of each solution. From mean value of copper concern, calculate factor relating milligram of copper found to milligram biuret added.

NOTE : *This determination should be done daily or each time when the estimation is taken up.*

5.1.5 *Procedure*

5.1.51 In urea, weigh accurately sample containing less than 10 mg biuret, dissolve in water transfer it to 100-ml volumetric flask, add 25 ml of ethyl alcohol and proceed as given in **5.1.4.2**.

5.1.5.2 In mix fertilizers, transfer accurately weighed sample not exceeding 5 g, containing less than 40 mg biuret to 250-ml beaker and add 1 ml of water for each gram of sample. Warm, add 65 ml alcohol and 7 drops bromocresol purple, and adjust pH to first blue colour (pH 6 to7) with 10 per cent of potassium hydroxide. Place on hot plate, heat it to boiling, cool, and if pH has changed, make final adjustment to first blue with vacuum, filter through paper pulp pad which has been washed with alcohol, into 100-ml volumetric flask (if filtrate is not clear, improper pH adjustment has been made. Add hydrochloric acid and readjust to pH 6 to 7 . Wash pad and precipitate with ethanol and further dilute it to volume. Transfer 25 ml aliquot to 100-ml volumetric flask and proceed as given in **5.1.4.2**.

5.1.6 *Calculation*

From copper found, calculate biuret concentration using factor and appropriate dilution factors.

NOTE : *Final aliquot should be varied to give copper concentration between* 1 µg/ml *and* 4 µg/ml .

5.2 Method 2 - Determination of Biuret content - Colourimetric method

5.2.1 *Field of application* **-** This method is applicable to urea samples only, and not for complex/ mixed fertilizers.

5.2.2 *Reagents*

5.2.2.1 Alkaline tartrate solution, dissolve 40 g of sodium hydroxide in 50 ml of water, cool, add 50 g of sodium potassium tartrate and dilute to 1 litre. Let it stand for at least one day before use.

5.2.2.2 Copper sulphate solution, dissolve 15 g of copper sulphate pentahydrate in distilled water and dilute to 1 litre.

5.2.2.3 Standard biuret solution 1 mg/ml, dissolve 100 mg of biuret in carbon dioxide – free water and make up to 100 ml.

5.2.3 *Preparation of standard curve*

Pipette out a series of aliquots, 2 ml to 50 ml, of standard biuret solution in 100–ml volumetric flasks. Bring the volume to approximately 50 ml with carbon dioxide-free water. Add one drop of methyl red and neutralize with 0.1 mol dm $^{-3}$ sulphuric acid to pink colour. Add with swirling 20 ml of alkaline tartrate solution and then 20 ml of copper sulphate solution. Dilute to mark. Shake for 10 seconds and place in a water bath for 15 minutes at 30 ± 5 ^{0}C.

Prepare the reagent blank simultaneously, determine absorbance of each solution against blank at 555 nm with 2 cm to 4 cm cell and plot the standard curve.

5.2.4 *Procedure*

5.2.4.1 In urea, dissolve (with stirring) 5 g of the sample in a 100 ml of water (see Note). Filter and wash into 250-ml volumetric flask and dilute to volume. Transfer 25 ml aliquot to a 100-ml volumetric flask and proceed as given in **5.2.3**.

NOTE : *If it is not completely dissolved heat the solution at temperature approximately 50* ^{0}C *for 30 minutes.*

5.2.5 *Calculation*

From the standard curve, determine the concentration of biuret in final dilution. Then calculate as given below :

$$Biuret,\ per\ cent\ by\ mass\ =\ \frac{C_1 x\ 100}{C_2}$$

where,

C_1 is the concentration of biuret in final dilution obtained from standard curve in mg/ml and ;
C_2 is the concentration of original sample in final dilution expressed in mg/ml.

APPENDIX A
DETERMINATION OF CALIBRATION FACTOR

A.1 APPARATUS

A.1.1 Atomic Absorption Spectrophotometer

A.2 REAGENTS

A.2.1 Standard solution

Prepare standard solutions in 0 µg to 20 µg range fresh daily. Automatic dilution apparatus may be used.

A.2.2 Copper stock solution (1 000 µg copper per ml)

Dissolve 1 000 g of pure copper metal in minimum amount of nitric acid and add 5 ml of hydrochloric acid. Evaporate almost to dryness and dilute to 1 litre with hydrochloric acid 0.1 mol dm^{-3}.

A.2.3 Dilute aliquots of solution prepared in **A.2.2** with hydrochloric acid (0.5 mol dm^{-3}) to make four or more standard solution of copper within the range of determination.

A.3 PREPARATION OF SAMPLE SOLUTIONS

A.3.1 For inorganic materials and mixed fertilizers

Dissolve 1.0 g of the well ground sample in 10 ml of hydrochloric acid in 100 ml beaker. Boil and evaporate the solution nearly to dryness on hot plate.

NOTE : *Heating of the sample should be controlled to avoid baking of the residue.*

Re dissolve the residue in 20 ml of hydrochloric acid (2 mol dm^{-3}), boiling gently if necessary. Filter through fast paper into 100-ml volumetric flask, and wash the paper and residue thoroughly with water. Measure the absorption of the solution directly, or dilute with hydrochloric acid (0.5 mol dm^{-3}) to obtain solutions within ranges of instrument.

A.3.2 For fertilizers containing organic matter (Tankage, Corncobs, Cotton-seed Hulls, etc*)*

Place 1.0 g of the sample in 100-ml beaker. Char on hot plate and ignited for 1 hour at 500^{o}C with muffle door propped open to allow free access of air. Break up cake with stirring rod and dissolve in 10 ml of hydrochloric acid as in **A.3.1.**

A.3.3 For fertilizer containing fritted trace elements

Dissolve less than or equal to 1.0 g of the well ground sample in 5 ml of perchloric acid and 5 ml hydrofluoric acid. Boil and evaporate to dense perchloric acid fumes. Dilute carefully with water filter and proceed as in **A.3.1.**

Alternatively dissolve the sample in 10 ml of hydrochloric acid, 5 ml of hydrofluoric acid and 10 ml of methyl alcohol. Evaporate to dryness. Add 5 ml of hydrochloric acid and evaporate. Repeat hydrochloric acid addition and evaporation. Dissolve residue as in **A.3.1.**

NOTE : *Normally platinum ware should be used; borosilicate or other glassware may be used if sodium, potassium, calcium and iron are not to be determined.*

A.4 DETERMINATION

A. 4.1 Set up instrument as in **A.1.** Read four or more standard solutions prepared within analytical range before and after each group of 6 to 12 samples. Flush burner with water between samples and re-establish "0" absorption point each time. Prepare calibration curve from average of each standard before and after sample group. Read concentration of samples from plot of absorption against µg/ml.

A.5 CALCULATION

Per cent copper = (µg/ml) x (*F* / *sample mass*) x 10^{-4}

where,

F is the ml original dilution x ml final dilution/ml aliquot; if original 100 ml volume is diluted.

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