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भारतीय मानक मसौदा

चेलेटेड जिंक (Zn-EDTA), कृषि ग्रेड - विशिष्टि

(आइ एस 13921 का पहला पुनरीक्षण)

Draft Indian Standard

CHELATED ZINC (Zn-EDTA), AGRICULTURAL GRADE – SPECIFICATION

(*First Revision of IS 13921***)**

ICS No. 65.080

Soil Quality and Fertilizers Sectional Committee, FAD 07

Last Date of Comments: 1 July 2024

FOREWORD

(*Formal clauses would be added later*)

Chelated fertilizers improves the bioavailability of micronutrients such as Iron, Copper, Zinc and in turn contribute to the productivity and profitability of commercial crop production.

This standard was first published in 1994 to provide uniformity in the production of quality chelated zinc (Zn-EDTA) to the users. In this revision, the limits of cadmium and arsenic along with their test methods have been incorporated to align it with Fertilizer Control Order. Also, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates one amendment issued to the previous version of this standard.

In the preparation of this standard, due consideration has been given to the provisions under *Fertilizer Control Order*, 1985. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for chelated zinc for agricultural use.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 5985 : 1985	Code of practice for handling and storage of bagged fertilizers (*first revision*)
IS 6092 (Part 1) : 1985	Methods of sampling and test for fertilizers: Part 1 Sampling (*first revision*)
IS 6092 (Part 5) : 1985	Methods of sampling and test for fertilizers: Part 5 Determination of secondary elements and micronutrients (*first revision*)
IS 6092 (Part 6) : 1985	Methods of sampling and test for fertilizers: Part 6 Determination of moisture and impurities (*first revision*)

3 REQUIREMENTS

3.1 Description

This material shall be in the form of free-flowing crystalline powder.

3.2 The material shall also comply with the requirements specified in Table 1.

Table 1 Requirements for Chelated Zinc (Zn-EDTA), Agricultural Grade

(*Clause* 3.2)

Sl No.	**Characteristic**	**Requirement**	**Method of test (Ref to)**
(1)	(2)	(3)	(4)
i)	Zinc content (expressed as Zn), percent by mass, *Min* in the form of Zn-EDTA	12.0	Annex A
ii)	Lead (as Pb) percent by mass, *Max*	0.003	IS 6092 (Part 5)
iii)	*p*H	6.0 – 6.5	Annex B
iv)	Cadmium (as Cd), percent by mass, *Max*	0.0025	Annex C
v)	Arsenic (as As), percent by mass, *Max*	0.01	IS 6092 (Part 6)

4 PACKING

4.1 The packing should be capable of providing adequate protection to the contents from absorption of moisture by the use of inner plastics liner. Further the packing should be

physically strong enough to withstand the normal stresses of handling in stacking, transport and storage.

4.2 It is recommended that the material is packed in 100 g to 500 g packings or as agreed to between the purchaser and the supplier.

5 MARKING

5.1 The packing shall be securely closed and marked with the following information:

a) Name of the fertilizer;
b) Indication of source of manufacture;
c) Percentage of zinc by mass;
d) Gross and net quantity in kg;
e) Batch number, in code or otherwise, to enable the lot of manufacture to be traced back from records;
f) Month and year of packing; and
g) Any other requirements as specified under the *Fertilizer Control Order*, 1985 and the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 HANDLING AND STORAGE

Factors to be borne in view in the handling and storage of the material shall be as prescribed in IS 5985.

7 SAMPLING

7.1 The method for drawing representative samples of the material shall be as prescribed in IS 6092 (Part 1).

7.2 Number of Tests

7.2.1 Zinc shall be tested on each of the individual samples. The lot shall be considered to have satisfied the requirement for zinc, if test results on each of the individual samples meet the corresponding requirement given in Table 1.

7.2.2 The remaining characteristics given in Table 1 shall be tested on the composite sample. The lot shall be considered to have met the remaining requirements given in Table 1. If each of the test results on the composite sample satisfies the corresponding requirement given in Table 1

7.2.3 The lot shall be declared as conforming to the requirements of the specification if **7.2.1** and **7.2.2** are satisfied.

8 TESTS METHODS

Tests shall be carried out according to methods prescribed in co1 (4) of Table 1.

9 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A
[*Table* 1, *Sl No.* (i)]
DETERMINATION OF ZINC

A-1 REAGENTS

A-1.1 EDTA Solution – 0.05 M**.**

Dissolve 18.612 g of disodium ethylene diamine tetra-acetate dihydrate (EDTA) in distilled water and make up the volume to 1 litre.

A-1.2 Standard Zinc Solution (1000 ppm)

Weigh accurately 1.0 g of zinc metal in a beaker. Add 200 ml HCI (1:1), keep it for few hours and allow it to dissolve completely. Transfer the solution to 1 litre volumetric flask. Make up the volume to the mark.

A-1.3 Concentrated Ammonia Solution - (sp gr 0.88).

A-1.4 Ammonium Nitrate – AR Grade salt.

A-1.5 Buffer Solution (*p*H 10)

Dissolve 8.0 g AR grade ammonium nitrate in 65 ml of water and add 35 ml of concentrated ammonia solution (sp gr 0.88).

A-1.6 Eriochrome Black (T) Indicator Mixture

Mix thoroughly 1 g of Eriochrome black (T) indicator with 100 g of AR grade potassium nitrate.

A-1.7 Hydroxylamine Hydrochloride - AR Grade.

A-1.8 Potassium Cyanide - 15 percent aqueous solution.

NOTE- Potassium cyanide is extremely poisonous, should be used with extreme care.

A-1.9 Manganese Sulphate Solution

Dissolve 11.15 g of manganese sulphate in 1 litre of distilled water.

A-1.10 Sodium Fluoride - AR Grade**.**

A-2 PREPARATION OF SAMPLE SOLUTION

Weigh accurately 1.0 g of the sample and transfer it to 100 ml volumetric flask. Make up the volume with distilled water. Keep it overnight.

A-3 PROCEDURE

A-3.1 Standardization of EDTA Solution

Take 10 ml of standard zinc solution. Dilute it by adding 30 ml distilled water. Add 10 ml of buffer solution and 30-40 mg of indicator mixture. Titrate with EDTA solution till clear blue end point is obtained. Note the volume of EDTA used as V_1 ml.

A-3.2 Standardization of Manganese Sulphate Solution

Take 25 ml of manganese sulphate solution. Dilute it by adding 100 ml distilled water. Add 0.25 g of hydroxylamine hydrochloride and 10 ml of buffer solution. Add 0.25 g of

hydroxylamine hydrochloride and 10 ml of buffer solution. Add 30-40 mg of indicator mixture. Titrate with EDTA solution till clear blue end point is obtained. Note volume of EDTA used as V_2 ml.

A-3.3 Determination of EDTA Content of Zn-EDTA Fertilizer

Take 10 ml of sample solution. Dilute it by adding 100 ml of distilled water. Add 0.25 g of hydroxylamine hydrochloride. Add 10 ml of buffer solution and 30-40 mg of indicator mixture. Warm to 40 °C and titrate with standard EDTA solution (preferably stirring magnetically) to clear blue end point. Note the volume of EDTA used as V_3 ml. After the end point, add 2.5 g of sodium fluoride and stir for 1 min. Titrate the solution with standard manganese sulphate solution, slowly, till a permanent red colour is obtained. Note the volume of manganese sulphate added as V_4 ml. Stir for 1 min. Titrate the excess of manganese ions with EDTA solution until the colour changes to pure blue. Note the volume of EDTA used as V_5 ml. After the second end point add 4-5 ml 15 percent aqueous potassium cyanide solution. Titrate it with manganese sulphate solution till colour changes sharply from blue to red. Note the volume of manganese sulphate solution added as V_6 ml.

A-4 CALCULATION

Molarity of EDTA solution (M_1) =

$$\frac{Molarity\ of\ standard\ zinc\ solution\ \times\ \text{Volume of standard zinc solution taken}}{\text{Volume of EDTA used } (V_1)}$$

Molarity of standard manganese sulphate solution =

$$\frac{M_1 \times V_2}{\text{Volume of standard manganese sulphate solution taken}}$$

No. of millimoles of EDTA used in titrating Zn + other metals (A) = $M_1 \times V_3$

No. of millimoles of EDTA liberated by sodium fluoride (B) = $M_2 \times V_4 - M_1 \times V_5$

Hence, No. of millimoles of EDTA used for titrating zinc (C) = $A - B$

But, No. of millimoles of EDTA liberated by KCN (D) = $M_2 \times V_6$

Hence, No. of millimoles EDTA contained by Zn-EDTA sample = $D - C$

EDTA percent = $372.24 \times (D - C)$

Percent of magnesium in the sample = $24.31 \times B$

Percent of free zinc = $65.38 \times C$

Percent zinc chelated with EDTA = $65.38 \times (D - C)$

ANNEX B
[*Table* 1, *Sl No.* (iii)]
DETERMINATION OF *p*H

B-1 PROCEDURE

Dissolve 5 g of the material in freshly boiled and cooled water, dilute to 100 ml and mix. Determine the *p*H value of the solution with a *p*H meter.

ANNEX C
[*Table* 1, *Sl No.* (iv)]
DETERMINATION OF CADMIUM

C-1 REAGENTS

C-1.1 Standard Cadmium Solution

Weigh out 1 g of pure cadmium metal and transfer it to a 250 ml beaker. Add 50 ml of water and 10 ml of concentrated nitric acid to dissolve the metal completely. Transfer the cadmium solution to a one litre flask with necessary washing. Make up the volume up to the mark. Shake well. This is a 1000 ppm solution of cadmium, (hereinafter called standard A). Dilute 1 ml of standard A to 100 ml in a volumetric flask. This is a 10 ppm solution of cadmium, (hereinafter called standard B).

C-1.2 Glass distilled water of *p*H 2.5 + 0.5

Dilute 1 ml of 10 percent sulphuric acid to one litre with glass distilled water and adjust the *p*H to 2.5 + 0.5 with a pH meter using sulphuric acid or sodium hydroxide solution. The water so obtained is called acidified water.

C-2 PREPARATION OF WORKING STANDARDS

Pipette out the following volume of standard B in 100 ml of numbered volumetric flask and make up the volume with acidified water. Stopper the flask and shake them well. The same acidified water should be used for the preparation of the sample solution. Fresh standards should be prepared and used every time.

Flask No.	Volume of standard (B) taken in ml	Concentration of cadmium after making volume to 100 ml (in ppm)
1	0	0.0
2	2.0	0.2
3	4.0	0.4
4	8.0	0.8
5	12.0	1.2
6	16.0	1.6
7	20.0	2.0

C-3 PROCEDURE

C-3.1 Preparation of Sample Solution

Weigh 2 g of chelated zinc and transfer it to a 100 ml volumetric flask giving repeated washings with acidified water. Dissolve the material by shaking well, make up the volume and mix thoroughly. Filter a portion if necessary. For higher concentration of cadmium adjust the weight and dilution such that the absorbance of final flaming solution is not more than a 2 ppm solution of cadmium.

Aspirate the standards as well as the sample solution in an atomic absorption spectrophotometer at a wave length of 228.8 nm using air acetylene flame and note the corresponding absorbance value for each solution.

C-4 CALCULATION

Draw a graph using concentration (ppm) as the X-axis and absorbance as the Y-axis. Determine the concentration of cadmium in ppm in the sample solution from the graph.

$$\text{Cadmium (as Cd), concentration in ppm} = \frac{C \times F}{M}$$

where,

C = concentration in ppm of final sample solution
M = mass of the sample
F = dilution factor.