



DRAFT EAST AFRICAN STANDARD

Fertilizer — Urea — Specification

EAST AFRICAN COMMUNITY

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

In order to achieve this objective, the Community established an East African Standards Committee mandated to develop and issue East African Standards.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

Fertilizer — Urea — Specification

1 Scope

This Draft East African Standard specifies the requirements, methods of test and sampling for urea fertilizer.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies

ISO 8157, *Fertilizers and Soil Conditioners – Vocabulary*

DEAS 913, *Fertilizers - Methods of Sampling*

ISO 17318, *Fertilizers and soil conditioners -- Determination of arsenic, cadmium, chromium, lead and mercury contents*

ISO 8397, *Solid Fertilizers and Soil Conditioners – Test sieving*

ISO 5315, *Fertilizers – Determination of Total Nitrogen Content – Titrimetric method after distillation*

3 Terms and definitions

For the purpose of this standard, terms and definitions given in ISO 8157 shall apply.

4 Requirements

4.1 Physical

Fertilizer shall be in the form of a free-flowing granules or prills, free from foreign matter and impurities. The colour of the fertilizer shall be uniform. When tested by ISO 8397, not less than 90 percent by weight of the material shall be of particles in the size range of 1 mm to 4 mm for prills and 2mm to 5mm for granules.

4.2 Chemical

The fertilizer shall comply with the requirements specified in Table 1

Table 1 - Chemical requirements for urea fertilizer

Characteristic	Requirement	Method of test
Total nitrogen, % by mass, min.	46	ISO 5315
Biuret, % by mass, max.	1.5	Annex A
Moisture, % by mass, Max.	1.0	Annex B

5 Heavy metal contaminants

Heavy metal contaminants in the fertilizers shall conform to the limits given in Table 2 when tested with the method specified therein.

Table 2 — Requirements for heavy metal contaminants

Heavy metal	Requirement	Method of test
Arsenic, mg/kg, max	20	ISO 17318
Cadmium, mg/kg, max.	7	
Mercury, mg/kg, max	0.1	
Lead, mg/kg, max.	30	
Chromium, mg/kg, max.	500	

6.0 Sampling

Sampling of fertilizer shall be carried out as prescribed in DEAS 913.

7.0 Packaging and labelling

7.1 Packaging

The fertilizer shall be packaged in materials that are clean and non-defective that protect the product from physical, chemical and moisture contamination and withstand multiple stages of handling (transportation and storage).

7.2 Labelling

Each package shall be indelibly labeled in English and/or any other language with the following information:

- a) name of the fertilizer i.e. "urea fertilizer;
- b) name and address of the manufacturer and importer;
- c) nutrient content;
- d) net content by mass in kg;
- e) handling instructions – including the words "Use No hooks";
- f) production date and expiry date;
- g) country of origin
- h) batch number
- i) storage conditions

Annex A (normative)

Determination of biuret

A.1 Reagents

A.1.1 Alkaline tartarate solution – Dissolve 40 g of NaOH in 500 ml of H₂O, add 50 g potassium sodium tartrate (NaKC₄ H₄ O₆ .4H₂O), and dilute to 1 litre. Let the solution stand for a day before use.

A.1.2 Copper sulphate solution (CuSO₄) – Dissolve 15 g CuSO₄.5H₂O in carbon dioxide (CO₂) free water (H₂O), and dilute to 1 litre.

A.1.3 Biuret – To recrystallize, weigh approximately 10 g reagent grade biuret, transfer to 1 litre beaker, add 1l absolute alcohol, and dissolve. Concentrate by gentle heating to approximately 250 ml. Cool at 5 °C and filter through fritted glass funnel. Repeat crystallization and dry final product for 1 hour at 105 °C – 110 °C in an oven. Remove from oven, place in desiccator and cool to room temperature.

A.1.4 Biuret standard solution – 1 mg / ml - Dissolve 1. 0000 g recrystallized biuret in carbon dioxide (CO₂) free water and dilute to 1 litre.

A.2 Preparation of the standard curve

Transfer series of aliquots, 2 ml – 50 ml of standard biuret solution to 100 ml volumetric flasks. Adjust volume to approximately 50 ml with CO₂ free H₂O. Add 1 drop methyl red, and neutralize with 0.05 M H₂SO₄ to pink colour. Add, with swirling 20 ml alkaline tartrate solution and then 20 ml CuSO₄ solution. Dilute to volume, shake for 10 s, and place in H₂O bath for 15 minutes at 30 °C ± 5 °C. Also, prepare reagent blank. Determine the absorbance *A* of each solution against blank at 555 nm (instrument with 500nm to 570 nm filter is also satisfactory) with 1 cm - 5cm cell. Plot the standard curve.

A.3 Procedure

Continuously, stir approximately 10g of the sample containing 30 mg to 125 mg biuret in 150 ml of water at 50 °C for 30 minutes. Filter and wash into 250 ml volumetric flask, and dilute to volume. Transfer 50 ml aliquot to 100 ml volumetric flask and proceed as in A.2 above. Calculate biuret concentration using standard curve.

Annex B (normative)

Determination of moisture content

B.1 Oven dry method

B.1.1 General

The method does not apply to fertilizers that yield volatile substances other than water at drying temperature.

B.1.2 Procedure

B.1.2.1 Weigh accurately 2 g of the prepared sample in a pre-weighed, clean and dry weighing bottle or petridish.

B.1.2.2 Heat in an oven for about 5 hours at $105^{\circ}\text{C} + 2\text{ C}$ to constant weight. Cool in a desiccator and weigh. For urea, heat at $70^{\circ}\text{C} + 5^{\circ}\text{C}$ for five hours to constant weight.

B.1.3 Calculation

$$\text{Moisture per cent by weight (\%)} = 100 \times \frac{B - C}{B - A}$$

where,

A is the weight in gram of the empty bottle;

B is the weight of the bottle plus the material in gram, before drying;

C is the weight of the bottle plus the material in gram, after drying

B.2 Vacuum desiccator method

B.2.1 General

The method is applicable to Ammonium Chloride, Calcium Ammonium Nitrate (CAN), Di-Ammonium Phosphate (DAP) and all types of complex and mixtures of NPK fertilizers.

B.2.2 Procedure

Weigh accurately in duplicate 5g of prepared sample in a weighed shallow porcelain dish. Put the sample in a desiccator over concentrated sulphuric acid, close and introduce vacuum for about 10 minutes, then stop the vacuum pump and leave the sample for 24 hours, then release vacuum, remove the sample from the desiccator and weigh.

B.2.3 Calculation

$$\text{Moisture per cent by weight} = 100 \times \frac{(W_2 - W_3)}{(W_2 - W_1)}$$

where,

W_1 is the weight in gram of empty porcelain dish;

W_2 is the weight in gram of porcelain dish with sample before putting the sample for 24 hours in the desiccator;

W_3 is the weight in gram of porcelain dish with sample after putting the sample for 24 hours in the desiccator.

B.3 Karl Fischer method

B.3.1 General This method is applicable to fertilizers like CAN, Urea and urea based complexes. This method is not suitable for phosphate rock based fertilizers and fertilizers containing monocalcium phosphate, calcium sulphate, alkali carbonates as well as aldehydes and ketone groups.

B.3.2 Apparatus

Karl Fischer titrator

B.3.3 Reagents

B.3.3.1 **Karl Fischer reagent(KF)** – Karl Fischer solution (pyridine free) (single solution)

B.3.3.2 **Di-sodium tartarate dihydrate** ($\text{Na}_2\text{C}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$) analytical grade

B.3.3.3 **Methanol-KF grade/spectroscopy grade** containing less than 0.05 % water

B.3.4 Procedure

B.3.4.1 Standardization of KF reagent

B.3.4.1.1 Set up the instrument as per manufacturer's manual.

B.3.4.1.2 Add methanol to the titration vessel until the electrodes are dipped and titrate with Karl-Fischer reagent to a pre-set end point persists for 30 seconds.

B.3.4.1.3 Add 100mg of the disodium tartarate dehydrate to the titration vessel carefully and titrate with Karl Fischer reagent to a pre-set end point (the pre-set end point should persist for 30 seconds). Note the volume of KF reagent used as V_1 mL.

B.3.4.2 Determination of moisture of sample

B.3.4.2.1 Weigh accurately 1 g of the prepared sample and transfer to the titration vessel carefully and stir until dispersed.

B.3.4.2.2 Titrate with KF reagent to the same pre-set end point as above and note the volume of KF reagent used as V_2 mL.

B.3.5 Calculation

$$\text{Factor (F)} (\text{mgH}_2\text{O}/1 \text{ ml of KF reagent}) = \frac{0.1566 \times \text{mg of sodium tartarate dihydrate added}}{V_1}$$

$$\text{Moisture per cent by weight} = \frac{F \times V_2 \times 100}{\text{Weight of sample (gram)} \times 1000}$$