



DRAFT EAST AFRICAN STANDARD

Fertilizer — Triple Superphosphate — 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 — Triple Superphosphate — Specification

1 Scope

This Draft East African standard specifies requirements, methods of sampling and test for Triple Superphosphate (TSP) 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 5316, *Fertilizers – Extraction of Water-Soluble Phosphate*

ISO 6598, *Fertilizers- Determination of Phosphorous Content- Quinolinephosphomolybdate- Gravimetric method*

ISO 8397 Solid Fertilizers and Soil Conditioners – Test sieving

3 Terms and Definitions

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

4.0 Requirements

4.1 Physical

4.1.1 The fertilizer shall be in granular or prilled forms free flowing, uniform in colour and free from foreign matter.

4.1.2 The particle size of the granular material, shall be such that not less than 90 % by mass of fertilizer, shall be of particles size range of 2 mm – 5 mm and for prilled material shall be of particles size range of 1mm - 4mm when tested in accordance with ISO 8397

4.2 Chemical

The Triple Superphosphate fertilizer shall comply with the requirements given in Table 1

Table 1 — Requirements for Triple Superphosphate fertilizer

| Characteristic | Requirement | Method of test |
|---|-------------|----------------|
| Total phosphates, as P ₂ O ₅ , % by mass, min | 46 | ISO 6598 |
| Water soluble phosphates, as P ₂ O ₅ , % by mass, min | 42.5 | ISO 5316 |
| Free phosphoric acid as P ₂ O ₅ , % by mass max. | 3.0 | Annex B |

| | | |
|---------------------------|-----|---------|
| Moisture, % by mass, max. | 5.0 | Annex A |
|---------------------------|-----|---------|

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 P_2O_5 , max. | 60 | |
| Mercury, mg/kg, max. | 0.1 | |
| Lead, mg/kg, max. | 30 | |
| Chromium, mg/kg, max. | 500 | |

7 Sampling

Sampling of fertilizer shall be carried out in accordance with DEAS 913.

8 Packaging and labelling

8.1 Packaging

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

8.2 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. "Triple Superphosphate (TSP) 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; and
- i) storage conditions.

Annex A (normative)

Determination of moisture content

A.1 Oven dry method

A.1.1 General

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

A.1.2 Procedure

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

A.1.2.2 Heat in an oven for about 5 hours at 105 °C + 2 °C to constant weight. Cool in a desiccator and weigh. For urea, heat at 70 °C + 5 °C for five hours to constant weight.

A.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.

A.2 Vacuum desiccator method

A.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.

A.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.

A.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.

A.3 Karl Fischer method

A.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.

A.3.2 Apparatus

Karl Fischer titrator

A.3.3 Reagents

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

A.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

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

A.3.4 Procedure

A.3.4.1 Standardization of KF reagent.

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

A.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.

A.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.

A.3.4.2 Determination of moisture of sample

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

A.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.

A.3.5 Calculation

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

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

Annex B (normative)

Determination of free acidity

B.1 Part 1: Determination of free acidity as H₂SO₄

B.1.1 Reagent

B.1.1.1 Standard sodium hydroxide solution – 0.02 N

B.1.1.2 Methyl red-methylene blue mixed indicator solution – prepared by mixing equal volumes of 0.2 % solution in rectified spirit of methyl red and 0.1 % solution in rectified spirit of methylene blue.

B.1.2 Procedure

B.1.2.1 Dissolve 20 g of the sample in about 50 ml of cold neutral water.

B.1.2.2 Filter and make up the volume to about 200 ml. The filtering media shall be neutral and shall not contain any alkaline material which would neutralize the free acidity.

B.1.2.3 Titrate the solution with standard NaOH solution using one or two drops of methyl red-methylene blue mixed indicator and a microburette.

B.1.3 Calculation

$$\text{Free sulphuric acid (as H}_2\text{SO}_4\text{) \% by mass} = \frac{4.904 \times N \times V}{W}$$

where.

N is the normality of standard NaOH solution;

V is the volume in milliliter (ml) of standard NaOH solution;

W is the weight in gram (g) of sample taken for the test.

B.2 Part 2 :Determination of free acidity as HNO₃

B.2.1 Reagent

B.2.1.1 Standard sodium hydroxide solution – 0.02 N

B.2.1.2 Methyl red-methylene blue indicator solution – prepared by mixing equal volumes of 0.2 % methyl red solution in alcohol and 0.1 % methyl blue solution in alcohol

B.2.2 Procedure

B.2.2.1 Dissolve 10 g of the sample in 100 ml of neutral water at room temperature in a beaker or conical flask.

B.2.2.2 Filter and add 2-3 drops of indicator solution

B.2.2.3 Titrate the solution with standard NaOH solution until the colour changes from violet to green

B.2.3 Calculation

$$\text{Free Nitric acid (as H)(HNO}_3\text{) \% by mass} = \frac{4.904 \times N \times V}{W}$$

where;

N is the normality of standard NaOH solution;

V is the volume in milliliter (ml) of standard NaOH solution;

W is the weight in gram (g) of sample taken for the test.

B.3 Part 3: Determination of free acidity as P₂O₅

B.3.1 Reagents

B.3.1.1 Acetone

B.3.1.2 Standard sodium hydroxide solution – 0.1 N

B.3.1.3 Bromo cresol green indicator solution – Dissolve 0.1 g bromocresol green in 100mL of rectified spirit

B.3.2 Procedure

B.3.2.1 Weigh accurately about 2.5 g of the prepared sample in a 250mL Erlenmeyer flask

B.3.2.2 Add 100 ml neutral acetone. Shake for one hour.

B.3.2.3 Filter rapidly through whatman filter paper No.1 in to 250 ml Erlenmeyer flask. Wash four times with 10mL portion of acetone

B.3.2.4 Evaporate acetone as far as possible

B.3.2.5 Add about 50 ml water and drops of bromocresol green indicator

B.3.2.6 Titrate with standard NaOH solution, until the colour changes from yellow to blue

B.3.3 Calculation

$$\text{Free phosphoric acid (as P}_2\text{O}_5\text{) \% by mass} = \frac{7.1 \times N \times V}{W}$$

where;

N is the normality of standard NaOH solution;

V is the volume in milliliter (mL) of standard NaOH solution;

W is the mass in gram (g) of sample taken for the test.