



DRAFT EAST AFRICAN STANDARD

Fertilizer — Potassium sulphate (sulphate of potash) — 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 — Potassium sulphate (sulphate of potash) — Specification

1 Scope

This Draft East African Standard specifies requirements and methods of sampling and test for potassium sulphate (sulphate of potash) 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 10084, *Solid fertilizers – Determination of mineral – acid- soluble sulphate content- Gravimetric method*

ISO 17319, *Fertilizers and soil conditioners — Determination of water-soluble potassium content — Potassium tetraphenylborate gravimetric method*

AOAC 928.02, *Chloine (Water-soluble) in fertilizers*

AOAC 983.04, *Sodium in fertilizers. Atomic absorption spect*

3 Terms and Definitions

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

4 Requirements

4.1 Physical

The material shall be crystalline, prills or powder, uniform in colour and free from any foreign matter.

Not less than 90 % by weight of the material shall be of particles in the size range of 1 mm to 4 mm for prills or not less than 65 % particles in the size range of 0.25 mm to 1.7 mm for powdered crystals. The fertilizer material shall be tested in accordance with ISO 8397.

4.2 Chemical

The potassium sulphate (sulphate of potash) fertilizer shall comply with the requirements given in Table 1.

Table 1 — Chemical requirements for potassium sulphate fertilizer

Characteristic	Requirement	Method of Test
Potash contents (as K ₂ O) % by mass, min.	50	ISO 17319
Sulphur (as S) % by mass, min.	17.5	ISO 10084

Total chloride (as Cl), % by mass, max.	2.5	AOAC 928.02
Sodium content (as NaCl), % by mass, max.	2.0	AOAC 983.04
Moisture %, by mass max.	1.5	Annex D

5 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 Sampling

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

8 Packaging and labelling

8.1 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).

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. "potassium sulphate (sulphate of potash" fertilizer;
- b) nutrient content;
- c) name and address of the manufacturer and importer;
- 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 potash content (flame photometric method)

A.1 Reagents

Potassium nitrate (KNO₃) or potassium chloride (KCl). Recrystallise reagent grade salt twice from water and dry for 5 hours at 105 °C.

A.2 Preparation of solution

Dissolve 1.5058 g of sample in water (H₂O) and dilute to 500 ml.

A.3 Preparation of standard curve

Dissolve 1.2931 g of KNO₃ (or 0.9535 g KCl) in water and dilute to 500 ml (1 000 ppm K). Prepare standard solutions by dilution to cover a range of 0 ppm–80 ppm K at intervals of 10 ppm, adding appropriate amounts of lithium nitrate (LiNO₃) if internal standard is to be used. Prepare standard curve of emission against concentration, adjusting instrument so that 50 ppm K gives reading near mid-scale. Atomize portions of standard solutions until readings for the series are reproducible.

A. 4 Procedure

Transfer 10 ml aliquot of sample solution to 250 ml beaker. Dilute to 100 ml volume and mix (if internal standard instrument is used, add required LiNO₃ before diluting to volume). Atomize portions of sample several times to obtain reliable average reading for each solution.

Determine ppm K from standard curve (temperature of standard and sample solutions must not differ by more than 2 °C).

Calculate % K₂O as follows:

0% – 30%, ppm K/2 = % K₂O

> 30%. ppm K/1 = % K₂O

Annex B (normative)

Determination of sodium content

B.1 Reagents

B.1.1 Concentrated hydrochloric acid, analytical grade.

B.1.2 Barium chloride solution, 12 per cent.

B.1.3 Ammonium carbonate solution, 1 M.

B.1.4 Perchloric acid.

B.1.5 Ethanol, 96 per cent.

B.1.6 Washing alcohol, prepared by mixing ethanol with 0.2 per cent (v/v) of perchloric acid.

B.1.7 Ammonium hydroxide, 2 M.

B.1.8 Magnesium uranyl acetate solution – Dissolve 90.0 g of crystallized uranyl acetate in 60 ml of glacial acetic acid and sufficient water by stirring and warming it to 70°C. Dilute the solution to 1 litre. Dissolve 600 g of crystallized magnesium acetate in 60 ml of glacial acetic acid and sufficient water by stirring and warming to 70°C. Dilute the solution to 1 litre. Mix the two solutions. Allow to stand for several hours. Filter off any residue. The final solution should be kept at 20°C in flasks made of glass with very low sodium content. The solution should also be used at 20°C.

B.2 Procedure

Weigh accurately about 10 g of the prepared sample, add a few milliliters of concentrated hydrochloric acid and 100 ml of water and heat to boiling in a beaker. To the boiling solution, add slowly in small quantities barium chloride solution. Heat for sometime and then cool. Transfer to a 250-ml volumetric flask and dilute to the mark with water, stir the solution and filter. Reject the first few milliliters of filtrate. Take exactly 10 ml of the filtrate and add a few milliliters of ammonium carbonate solution. Filter off the precipitated carbonates and wash the precipitate with water, the washings being added to the filtrate. Evaporate the filtrate and washings to dryness in a porcelain dish of 10 cm diameter and calcine gently. Add to the residue a small quantity of water and 6 ml of perchloric acid and evaporate almost to dryness in a water bath. Repeat the evaporation with perchloric acid twice again.

Cool the residue, add a few milliliters of ethanol and crush the moist mass to a fine state by using a glass pestle. Decant off the liquid. Repeat the crushing of the residue and decantation with further additions of washing alcohol, collecting all the decanted liquid. Transfer the precipitate to a small filter and wash thoroughly with ethanol adding these washings also to the decanted liquid. Neutralize the filtrate and washings with ammonium hydroxide and heat to dryness. Transfer the residue to a beaker with about 5 ml of water and add an excess of magnesium uranyl acetate, maintaining the temperature at 20°C. Stir the precipitation and allow it to stand for half an hour at 20°C. Decant off the clear solution through a sintered glass crucible which has been previously washed with ethanol, dried at 120°C ± 5°C and weighed. Again, add a small quantity of ethanol and repeat the decantation twice. Finally wash the precipitate with ethanol on the filter. Dry the crucible with the precipitate for half an hour at 120°C ± 5°C, cool in desiccator and weigh.

B.3 Calculation

Sodium (as NaCl), per cent by mass (on dry basis)

$$= \frac{97.05 M_1}{M}$$

where

M_1 = mass in g of the precipitate and

M = mass in g of the prepared sample taken for the test.

Annex C (normative)

Determination of chlorides other than ammonium chloride

C.1 Reagents

C.1.1 Standard silver nitrate solution – 0.1N

C.1.2 Concentrated nitric acid

C.1.3 Ferric ammonium sulphate solution - saturated in water and stabilized by addition of 50ml nitric acid.

C.2. Procedure

C.2.1 Dissolve 0.2g of the prepared sample in 40ml.

C.2.2 Add exactly 50ml of standard silver nitrate solution and 5ml of concentrated nitric acid.

C.2.3 Add 0.5ml of nitrobenzene and make up the volume of the mixture to exactly 100ml with water.

C.2.4 Take exactly 50ml of the solution and add 2ml of ferric ammonium sulphate solution.

C.2.5 Titrate the excess of silver nitrate in this portion with standard ammonium thiocyanate solution.

C.2.6 Carry out a blank test following the procedure given as above but without using the material.

C.3 Calculation

Total chloride (as Cl), per cent by mass (on dry basis)

$$= \frac{7.094 (V_1 - V_2) N}{W}$$

where

V₁ = Volume in ml of standard ammonium thiocyanate used in the blank determination

V₂ = Volume in ml of standard ammonium thiocyanate solution used in the test with the material.

N = Normality of standard ammonium thiocyanate solution

W = Weight in gram, of the prepared sample taken for the test.

Annex D (normative)

Determination of moisture content

D.1 Oven dry method

D.1.1 General

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

D.1.2 Procedure

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

D.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.

D.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.

D.2 Vacuum desiccator method

D.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.

D.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.

D.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.

D.3 Karl Fischer method

D.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.

D.3.2 Apparatus

Karl Fischer titrator

D.3.3 Reagents

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

D.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

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

D.3.4 Procedure

D.3.4.1 Standardization of KF reagent.

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

D.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.

D.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.

D.3.4.2 Determination of moisture of sample

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

D.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.

D.3.5 Calculation

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

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