DUS DEAS 904

DRAFT UGANDA STANDARD

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Fertilizer — Phosphate rock powder — Specification

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National foreword

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(a) a member of International Organisation for Standardisation (ISO) and

(b) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and

(c) the National Enquiry Point on TBT Agreement of the World Trade Organisation (WTO).

The work of preparing Uganda Standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of representatives of consumers, traders, academicians, manufacturers, government and other stakeholders.

Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council.

This Draft Uganda Standard, DUS DEAS 904: 2018, *Fertilizer — Phosphate rock powder — Specification,* is identical with and has been reproduced from a Draft East African Standard, DEAS 904: 2018*, Fertilizer — Phosphate rock powder — Specification,* and is being proposed for adoption as a Uganda Standard.

This standard was developed by the Food and agriculture Standards Technical Committee (UNBS/TC 2).

Wherever the words, “East African Standard" appear, they should be replaced by "Uganda Standard."

DEAS 904:2018

DRAFT EAST AFRICAN STANDARD

Fertilizer — Phosphate rock powder — Specification

EAST AFRICAN COMMUNITY

First Edition 2018

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 — Phosphate rock powder — Specification

1 Scope

This Draft East African Standard specifies requirements, methods of sampling and test for phosphate rock fertilizers in powder form of biogenic sedimentary origin.

2 Normative References

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

ISO 8157, *Fertilizer – Terminology and Vocabullary*

DEAS 913, Fertilizer – *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*

3 Terms and definitions

For the purpose of this East African standard, the following terms and definition shall apply together with those given in ISO 8157;

3.1

biogenic sedimentation

biogenic sedimentation – is the deposition by settling of a suspended material which may be either constituents, or secretions of plants or animals produced by life processes.

4 Requirements

4.1 General requirements

4.1.1 Physical

The fertilizer material shall consist of phosphate rock from natural deposits in the form of free-flowing powder. The colour of the phosphate rock powder fertilizer shall be uniform.

4.1.2 Particle size

The particle size shall be such that at least 90 % of the fertilizer materials pass through 0.15 mm sieve and not more than 10 % of the remaining material shall pass through 0.25 mm sieve. The fertilizer sample shall be determined by the method given in ISO 8397.

4.1 Specific requirements

Phosphate rock powder fertilizer shall comply with the requirements prescribed in Table 1.

Table 1 Requirements for Phosphate rock powder

Characteristic	Requirement	Method of test
Total phosphates, (as P_2O_5), % by mass, min	28.0	Annex A (I)
Citric acid soluble phosphorus (as P_2O_5) % by mass, min.	9	Annex A (II)
Calcium content (as CaO) % by mass, min.	35	Annex B
Moisture, percent by mass, max.	5	Annex C

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

5.2 The radioactive elements found in the fertilizer materials shall not exceed the limit set by the respective country.

6 Sampling

Sampling of Phosphate Rock Powder fertilizer shall be carried out as prescribed in DEAS 913

7 Packaging, and labelling

7.1 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).

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. common name and brand name;

b) name and address of the manufacturer/packer;

c) net content by mass, in kilogram (kg) or tonnes (weight or mass);

d) nutrient content, i.e. total phosphorus (% P_2O_5) by mass;

e) country of origin;

f) handling instructions, including the words such as “use no hooks”;

g) production date;

h) batch number; and

i) storage conditions.

Annex A
(normative)

Determination of total phosphorus

A.1 Reagents

For the purpose of this East African Standard, only analytical grade reagents and distilled water given below shall be used:

a) Concentrated hydrochloric acid;

b) Concentrated nitric acid;

c) Magnesium nitrate solution;

d) Citric molybdic acid reagent;

e) Quinoline solution; and

f) Quimociac reagent.

NOTE All the reagents above can be prepared as described below;

A. *Magnesium nitrate solution* – Dissolve 950 g of P-free Mg $(NO_3)_2.6H_2O$ in water and dilute to 1 L.

B. *Citric molybdic acid reagent* – Dissolve 54 g of 100% molybdic anhydride (MoO_3) and 12 g NaOH with stirring in 400 mL hot water and cool. Dissolve 60 g citric acid in mixture of 140 mL HCl and 300 mL water and cool. Gradually add molybdic solution to citric acid solution while stirring. Cool, filter and dilute to 1 L (solution may be green or blue colour, depending on exposure to light). If necessary, add 0.5 % $KBrO_3$ solution drop by drop until green colour becomes pale. Store in dark polyethylene bottle.

C. *Quinoline solution* – Dissolve 50 mL synthetic quinoline while stirring in mixture of 60 mL HCl and 300 mL water. Cool, dilute to 1 L and filter. Store in polyethylene bottle.

D. *Quimociac reagent* **–** Dissolve 70 g of sodium molybdate dihydrate in 150 mL water. Dissolve 60 g citric acid in mixture of 85 mL HNO_3 and 150 mL water and cool. Gradually add sodium molybdate solution to citric acid-nitric acid mixture while stirring (**A**). Dissolve 5 mL synthetic quinoline in mixture of 35 mL HNO_3 and 100 mL water (**B**). Gradually add this solution (**B**) to solution (**A**), mix and let it stand for 24 h. Filter, add 280 mL acetone, dilute to 1 L with water and mix well. Store in polyethylene bottle.

A.2 Procedure

For cases where preparation of reagents should be needed; the following procedures should be followed:

Weigh 1 g of sample, treat with 15-30 mL concentrated hydrochloric acid and 3-10 mL concentrated nitric acid and digest till brown fumes cease, then dilute to 200 mL. Pipette into 500 mL Erlenmeyer flask, aliquot containing not more than 25 mg P_2O_5, dilute to approximately 100 mL with water. Proceed with one of the following methods:

A. Add 30 mL citric molybdic acid reagent and boil gently for 3 minutes (solution must be precipitate free at this stage). Remove from heat and swirl carefully. Immediately add from burette 10 mL quinoline solution with continuous swirling (add first 3-4 mL drop wise and remainder in steady stream) or

B. Add 50 mL quimociac reagent, cover with watch glass, place on hot plate in well-ventilated hood and boil for one minute.

After treatment with either **A** or **B**, cool to room temperature; swirl carefully 3-4 times during cooling. Filter into crucible with glass fibre filter paper or sintered glass crucible grade 4 (30 mL capacity), previously dried at 250 ^{O}C and weighed and washed five times with 25 mL of water. Dry crucible and contents for 30 minutes at 250 ^{O}C. Cool in a desiccator to a constant weight and weigh.

A.3 Calculation

$$\text{Total phosphorus (as } P_2O_5\text{) \% by mass} = \frac{0.03207 \times (A - B) \times F \times 100}{W}$$

where,

A = Weight of sample precipitates in gram;
B = Weight of sample precipitates in gram for blank;
W = Weight of sample;
F = Dilution factor.

II. Determination of citric acid soluble phosphorus content

a) REAGENTS

For the purpose of this East African Standard, only analytical grade reagents and distilled water shall be used.

a) Standard phosphate solution – Dilute 50 mL of stock phosphate solution to 250 mL with water (1 mL of this solution is equivalent to 0.2 mg P_2O_5).

b) Vanado-molybdate reagent – Dissolve separately 20 g of ammonium molybdate and 1 g of ammonium vanadate in hot water. Cool, mix and acidify with 140 mL concentrated nitric acid and dilute to 1 L.

b) STANDARDIZATION OF INSTRUMENT

Measure from a burette, into a series of 100-mL volumetric flasks, 25.0 mL, 26.0 mL, 27.0 mL, 28.0 mL, 29.0 mL, 30.0 mL, and 31.0 mL of the standard phosphate solution containing 5.0 mg, 5.2 mg, 5.4 mg, 5.6 mg, 5.8 mg, 6.0 mg and 6.2 mg of P_2O_5 respectively. Within five minutes for the entire series, add 25 mL of the vanadomolybdate reagent into each flask and dilute to 100 mL, ensuring that the temperature of the reagent and dilution water is maintained at 27 + 2 ^{O}C. Mix and allow to stand for ten minutes.

If a single beam spectrophotometer is used, set the spectrophotometer to a wavelength of 420 nm, fill two 1-cm cells with the standard solution containing 5.0 mg P_2O_5 to which the vanadomolybdate reagent has been added as given above and check the absorbance of the cells. If there is a small difference, select the cell with the lower reading as the standard reference cell.

Using the other cell (sample cell), determine the apparent absorbance at 27 + 2 ^{O}C (corrected for cell differences) of the other standard solutions containing 5.2 mg, 5.4 mg, 5.6 mg, 5.8 mg, 6.0 mg and 6.2 mg of P_2O_5 referred to the solution containing 5.0 mg P_2O_5 as the standard. After each determination, empty and refill the cell containing the 5-mg standard solution and readjust to zero absorbance to avoid errors that might arise from temperature changes. Plot a calibration graph of absorbance against concentration in mg P_2O_5 per 100 mL of standard solution.

NOTE – In the preparation of the standard phosphate solution, add to each flask, a volume of citric acid equal to the amount contained in the diluted sample solution mentioned in **5.1 (a)** prior to the addition of the vanadomolybdate reagent.

c) DISSOLUTION OF SAMPLE

Weigh 1 g of prepared sample into a 250-mL conical flask, add 100 mL of freshly prepared 2 per cent citric acid, stopper and shake for 30 minutes using a mechanical shaker (set a -shaking speed of twice per second), filter immediately.

d) PROCEDURE

A. Successively dilute a portion of the solution (volume V1 mL) prepared as in **5.1.2**, so that a final volume of about 25 mL contains between 5.5 mg P_2O_5 and 6.2 mg P_2O_5, taking care that the dilution water is at 27 ± 2^{O}C.

B. Transfer this solution to a 100-mL volumetric flask, add 25 mL of the vanadomolybdate reagent (at a temperature of 27 + 2^{O}C), dilute to the mark, mix and allow to stand for 10 minutes. At the same time, transfer 25 mL of the standard phosphate solution (at 27 + 2^{O}C) into a second 100-mL volumetric flask. Add sufficient citric acid such that when the solution is made up to 100 mL, its concentration is equal to that of the diluted sample solution. Add 25 mL of vanadomolybdate reagent at 27 + 2^{O}C, dilute to mark, mix and allow to stand for 10 minutes. Measure the difference in absorbance at 27 + 2^{o}C between the two solutions and estimate the P2O5 content of the sample solution using the calibration graph.

e) CALCULATION

Citric acid soluble P_2O_5 content % by mass = $\frac{Y}{1000} \; \frac{100}{V1} \; \frac{100}{M}$

where,

Y = graph reading, in mg/100 mL;
V1 = volume (in mL) of sample solution diluted for test;
M = mass (in gram) of the sample.

Annex B
(normative)

Determination of calcium oxide

B.1 Reagents

For the purpose of this East African Standard, only analytical grade reagents and distilled water shall be used.

a) Concentrated hydrochloric acid.

b) Concentrated nitric acid.

c) Hydrofluoric acid – 40%

d) Sodium carbonate – solid

e) Dilute hydrochloric acid – 1:1 (v/v)

f) Ammonium chloride – solid

g) Dilute acetic acid – 1:1 (v/v)

h) Ammonium oxalate – solid

i) Oxalic acid solution – 1g/L (m/v)

j) Dilute sulphuric acid 1:4 (v/v)

k) Standard potassium permanganate solution (0.1 N) – Dissolve3.2g of potassium permanganate in 1 litre of water. Let stand in the dark overnight. Filter, without washing, through glass wool. Avoid contact with rubber or other organic material. Store in a dark-coloured glass-stoppered bottle. Standardized against standard sodium oxalate.

B.2 Procedure

For cases where preparation of reagents should be needed; the following procedures should be followed:

B.2.1 Transfer 0.5g of sample that has been dried at 105 ^{0}C to a 400-mL tall from beaker. Dissolve, by boiling gently with 20ml of concentrated hydrochloric acid and 10ml of concentrated nitric acid, until the volume is reduced to about 10ml. Covering the beaker by watch-glass throughout the process of dissolution, add 20ml of concentrated nitric acid and then evaporate to a small volume. Wash the lower surface of the watch-glass into beaker with hot water, filter and wash collecting the filtrate and washings in a 250ml measuring or volumetric flask. Preserve the filter and washings.

B.2.2 Dry the residue and burn off the filter paper by ignition in a platinum crucible. Moisten the cooled residue with a few drops of a concentrated nitric acid and add about 10ml of hydrofluoric acid. Evaporate continuously just to dryness, ignite and fuse the residue with about 2 to 3 g of sodium carbonate. Extract the melt in about 25-30ml of dilute hydrochloric acid in a beaker, evaporate to dryness and bake. Extract the baked mass with about 10ml of concentrated hydrochloric acid, dilute, filter and wash collecting the filtrate and washings in the same volumetric flask where in the first filtrate along with washings has been preserved under 5.1. Cool the combined filtrates and make up to the mark.

B.2.3 Pipette out exactly 100ml from the solution in the volumetric flask under 5.2 into 400ml-beaker. Add sufficient ammonium chloride (about 15ml) to hold magnesium in solution in presence of ammonium oxalate. Add 20ml of dilute acetic acid, heat to boiling point and add about 2g of ammonium oxalate while stirring. Boil and allow the precipitate to settle for 1 to 2 hours, cool and filter the solution through a close filter paper. Wash the precipitate with hot water until free from oxalate.

B.2.4 Dissolve the precipitate on the filter in about 50ml of hot dilute hydrochloric acid. Wash the filter thoroughly with hot water until the filter is free from acid and collect the washings also in the same beaker in which calcium was first precipitated. Reduce the bulk to about 100ml by evaporation and re-precipitate calcium as oxalate under 5.3

B.2.5 Wash the precipitate of calcium oxalate into same beaker in which precipitation was first carried out and treat the filter with about 25ml of warm (70 to 80^{0}C) dilute sulphuric acid for dissolving adhering precipitate of calcium oxalate. Wash the filter free from acid by means of hot water. Collect the washings also in the same beaker into which the precipitate was transferred. Add about 25ml more of dilute sulphuric acid and raise the temperature of contents to about 70^{0}C. When the precipitate has dissolved, titrate with standard potassium permanganate solution until a permanent pale pink colour persists in solution.

B.3 Calculation

$$\text{Calcium oxide \%} = \frac{A \times B \times F\,(0.028) \times 100}{C}$$

where,

A is the volume (in ml) of standard potassium permanganate solution;

B is the normality of the standard potassium permanganate solution;

C is the mass (in gram) of the sample represented by the aliquot taken;

F is the dilution factor.

Annex C
(normative)

Determination of moisture content

C.1 Oven dry method

C.1.1 General

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

C.1.2 Procedure

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

C.1.2.2 Heat in an oven for about 5 hours at 105^0C + 2^0C to constant weight. Cool in a desiccator and weigh. For urea, heat at 70^0C + 5^0C for five hours to constant weight.

C.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.

C.2 Vacuum desiccator method

C.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.

C.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.

C.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.

C.3 Karl Fischer method

C.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.

C.3.2 Apparatus

Karl Fischer titrator

C.3.3 Reagents

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

C.3.3.2 Di-sodium tartarate dihydrate (Na2C4O62H2O) analytical grade

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

C.3.4 Procedure

C.3.4.1 Standardization of KF reagent.

C.3.4.1.1 Set up the instrument as per manufacturer’s manual.

C.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.

C.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.

C.3.4.2 Determination of moisture of sample

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

C.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 V2 ml.

C.3.5 Calculation

$$\text{Factor (F)(mgH}_2\text{O/1 ml of KF reagent)} = \frac{0.1566 \times \text{mg of sodium tartarate dihydrate added}}{V1}$$

$$\text{Moisture per cent by weight} = \frac{F \times V_2 \times 100}{\text{Weight of sample (gram)} \times 1\,000}$$