Reference No. ARS 1489:2018(E) ICS 65.080

DARS 1489:2018

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This African Standard was prepared by the ARSO Technical Harmonization Committee Number 5 on Chemistry and Chemical Engineering (ARSO/THC 05).

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Single superphosphate fertilizer — Specification

1 Scope

This African Standard specifies the requirements, sampling and test methods for single superphosphate 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.

AOAC 2006.03, Arsenic, cadmium, cobalt, chromium, lead, molybdenum, nickel, and selenium in fertilizers — Microwave digestion and inductively coupled plasma-optical emission spectrometry

EN 12048, Solid fertilizers and liming materials — Determination of moisture content — Gravimetric method by drying at 105±2 °C

EN 13475, Liming materials — Determination of calcium content — Oxalate method

EN 16032, Fertilizers — Extraction and determination of elemental sulfur

ISO 5316, Fertilizers — Extraction of water-soluble phosphates

ISO 6598, Fertilizers — Determination of phosphorus content — Quinoline phosphomolybdate gravimetric method

ISO 7409, Fertilizers — Marking — Presentation and declarations

ISO 8157, Fertilizers and soil conditioners Vocabulary

ISO 8397, Solid fertilizers and soil conditioners — Test sieving

ISO 11047, Soil quality — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc — Flame and electrothermal atomic absorption spectrometric methods

ISO 14820-1, Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling

ISO 14820-2, Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation

ISO 15178, Soil quality — Determination of total sulfur by dry combustion

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

3 Terms and definitions

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

4 Requirements

4.1 General Description

The fertilizer shall be in the form of free flowing powder or granules. It shall be free from hard lumps, foreign matter and shall not cake on storage

4.2 Physical requirements

When test sieved in accordance with ISO 8397, the particle size for granular fertilizer shall be such that not less than 90 %, by mass of the fertilizer shall be of particles in the size range of 1 mm to 4 mm. Not more than 5% shall be below 1 mm.

4.3 Chemical requirements

Single superphosphate fertilizer shall comply with the following compositional limits.

Table 1 — Chemical requirements

Parameters	Requirements		Method of test
	Granules	Powder	AI.
Water soluble phosphate, P ₂ O ₅ , %, m/m, min.	15.8	15.8	ISO 5316
Free phosphoric acid, as P ₂ O ₅ , % m/m, max.	4	4	Annex A
Moisture content, % m/m, max.	5	512	EN 12048
Sulphur as S, % m/m, min.	11	110	ISO 15178/ EN 16032

4.4 Heavy metal contaminants

The presence of heavy metal contaminants shall not exceed the limits stipulated in Table 2.

Table 2 — Heavy metal contaminants

Parameter	Maximum limit, mg/kg	Test method
Arsenic, As	10.0	ISO 17318
Cadmium, Cd	7.0	ISO 17318
Mercury, Hg	0.1	ISO 17318
Selenium, Se	1.0	AOAC 2006.03
Lead, Pb	30.0	ISO 17318
Chromium, Cr, max.	50.0	ISO 17318/ ISO 11047

5 Sampling

Sampling shall be carried out in accordance with ISO 14820-1.

6 Tests

6.1 Methods of test

Samples of the fertilizer shall be prepared in accordance with ISO 14820-2 and tested in accordance with the methods of test indicated in Table 1 and Table 2.

6.2 Inspection

From the bulk samples obtained from ISO 14820-1, inspect the lot for the characteristics relating to the packing and marking of the product.

7 Compliance

The lot shall be deemed to comply with the standard if after inspection and testing it complies with the requirements of this standard.

8 Packaging and labelling

8.1 Packaging

The fertiliser shall be packed in clean, non-defective and strong containers. The material for which the container is made shall be such as to protect the contents from moisture and also not lead to easy rupture during handling, transportation and storage.

8.2 Labelling

- **8.2.1** Each container of the fertiliser shall bear a label in indelible marking in accordance with ISO 7409, the Globally Harmonized System (GHS) and with the following particulars:
- a) type of the fertilizer, as "Single superphosphate fertilizer"
- b) brand name
- c) name, address and physical location of the manufacturer/packer/importer;
- d) country of origin/manufacture;
- e) date of manufacture
- f) best before date
- g) the content net weight;
- h) batch/lot number;
- i) storage instructions;
- j) handling instructions;
- k) instructions for use;
- instructions for disposal of used containers.
- m) form of fertilizer (granular or powdered)

8.2.2 Bulk containers

Where the fertilizer is distributed in bulk, the marking information shall accompany the delivery notice to the purchaser.

9 Certificate of analysis

A certificate of analysis stating the minimum percentage levels of plant nutrient elements shall accompany every lot or consignment of the fertilizer.

10 Material safety

Each container must be accompanied by a Material Safety Data Sheet (MSDS) and Technical Data Sheet (TDS).

Annex A

(normative)

Determination of free phosphoric acid

A.1 Purpose

This method determines the percent of phosphoric acid (P₂O₅) in commercial fertilizers by colorimetry.

A.2 Reagents and apparatus

- 1) An Atomic Absorption Spectrophotometer or a suitable colorimeter.
- 2) Nitric Acid (HNO₃), 1.42 specific gravity.
- 3) Hydrochloric Acid (HCI), 1.19 specific gravity.
- 4) Potassium Phosphate, Monobasic (KH₂PO₄), Primary Standard Grade, dried at 105-110 °C for several hours prior to use.
- 5) Ammonium Molybdate ((NH₄)6MO₇O₂₄•4H₂O), Reagent Grade.
- 6) p-Methylaminophenol Sulfate, Reagent Grade.
- 7) Sodium Acid Sulfite (NaHSO₃), Reagent Grade.
- 8) Cuvets, optical glass, matched, 10mm path length.
 - NOTE If available, an optical glass, automatic flow-through cuvet may be used.

A.3 Preparation of standard solutions

Phosphate Stock Solution: Weigh 0.4394 g of dried, primary standard KH₂PO₄ into a 500- mL volumetric flask. Add 30 ml of HNO₃ and 5 ml of HCl and boil until brown fumes have been expelled. Dilute to volume with distilled water.

- **5 ppm Working Standard:** Pipette a 5-ml aliquot of the phosphate stock solution into a 200-ml volumetric flask and dilute to volume with distilled water. This solution is equivalent to 57.2% phosphoric acid.
- **4 ppm Working Standard:** Pipette a 4-mL aliquot of the phosphate stock solution into a 200-mL volumetric flask and dilute to volume with distilled water. This solution is equivalent to 45.8% phosphoric acid.

Blank Working Solution: Boil a mixture consisting of 5 ml of HCl and 30 ml of HNO₃ until brown fumes have been expelled, transfer to a 500-ml volumetric flask and dilute to volume with distilled water. Transfer a 4-ml aliquot to a 200-ml volumetric flask and dilute to volume with distilled water.

A.4 Preparation of colour development solutions

Acid Molybdate Solution: Pour, with stirring, a solution containing 16.62 g of ammonium molybdate in 156.9 ml of distilled water into a solution of 318.4 ml of HCl to which 29.6 ml of distilled water has been added.

Reducing Solution: Dissolve 1.0 g of p-methylaminophenol sulfate and 3.0 g of sodium acid sulfite in 100 ml distilled water.

A.5 Procedure

Weigh, to the nearest 0.1 mg, 0.50 ± 0.02 g of sample into a 500-ml volumetric flask, adding 30 ml of HNO₃ and 5 ml of HCl. Boil until brown fumes have been expelled, cool to room temperature and dilute to volume with distilled water. Transfer a 2-ml aliquot to a 100-ml volumetric flask and dilute to volume with distilled water. From this second dilution, transfer a 10-ml aliquot to a 20-ml scintillation vial and add a 2-ml aliquot of acid molybdate solution and a 2-ml aliquot of reducing solution. Allow to stand 1 hour for color development. At the same time that the sample vial is prepared, a blank vial, a 4 ppm phosphorus vial and a 5 ppm phosphorus vial must also be prepared and allowed to develop color along with the sample. The blank, 4 ppm phosphorus and 5 ppm phosphorus are prepared by the same procedure used for preparation of the sample, using 10-ml aliquots of the blank working solution, the 4 ppm phosphorus working standard and the 5 ppm phosphorus working standard.

NOTE When making determinations on fertilizers with low percentages of phosphoric acid, it is necessary to adjust the sample weight so that the sample working dilution contains between 4 ppm and 5 ppm phosphorus.

Calibrate the instrument using the blank solution, the 4 ppm phosphorus solution and the 5 ppm phosphorus solution, then determine the concentration of the sample solution.

NOTE When using an Atomic Absorption Spectrophotometer for colour measurements, the cuvet holder is placed over the burner in the light path and a Cu hollow cathode lamp is used with the instrument grating set at a wavelength of 640 nm.

A.6 Calculations

The method of calculating the % phosphoric acid will vary according to the make and model of instrument used. Report the results to the nearest $0.1\% P_2O_5$ as follows:

% Phosphoric Acid (P2O5)

% Phosphoric Acid (P2O5)

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