AFRICAN STANDARD

DARS
1482
Second Edition 2025

Granulated superphosphate fertilizers — Specification

Reference No. DARS 1482:2025(E)
ICS 65.080

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Introduction

Mineral fertilizers are materials, either natural or manufactured, containing nutrients essential to normal growth and development of plants. Various studies, including those conducted by FAO, have established beyond doubt the existence of a close relationship between fertilizer consumption level and agricultural productivity. Crop yield levels are generally higher in those countries where fertilizer consumption levels are also high. Amongst the various agricultural inputs, fertilizers, perhaps next only to water, contribute the maximum to increasing agricultural production. It has been estimated that about 50 percent of the increase in agricultural production witnessed during the last decade in developing countries is attributable to fertilizer use.

Most developing countries have assigned special priority to fertilizers in their efforts to modernize their agriculture. It has also been increasingly realized that it is relatively cheaper and more in a country's long-term interest to import fertilizer than food-grains. Many developing countries, within the limitations imposed by availability of raw materials, financial resources, technical manpower, etc, are also making serious efforts to set up fertilizer plants so as to meet part or all of their fertilizer requirements. This phenomenon has resulted in substantial growth in fertilizer consumption.

Phosphorus is an important constituent of the earth's crust, but has been concentrated over geological time in deposits of phosphate rock (formed mainly from the remains of aquatic organ isms), and is present in most natural and cultivated soils in insufficient quantity for full crop growth. The sources from which phosphorus is obtained for use in fertilizers are: (i) phosphate rock deposits, by far the most important source, and (ii) basic slag, a by-product of ·the steel industry.

Plants take phosphorus from the soil solution in the form of phosphate ions (HPO_4^{--} and H_2PO_4), so that for fertilizer phosphorus to be available it must be released in ionic form to the soil solution. The phosphorus in fertilizers occurs in various chemical and physical forms whose availability varies very much.

Granulated superphosphate fertilizers — Specification

1 Scope

This African Standard specifies requirements, sampling method and test methods for granulated superphosphate fertilizers.

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 969.02, *Phosphorus (total) in fertilizers — Alkalimetric quinolinium molybdophosphate method*

AOAC 993.31, *Phosphorus (available) in fertilizers — Direct extraction method*

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*

ISO 5316, *Fertilizers — Extraction of water-soluble phosphates*

ISO 7409, *Fertilizers — Marking — Presentation and declarations*

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

ISO 8189, *Solid fertilizers — Determination of moisture content — Gravimetric method by drying under reduced pressure*

ISO 8397, *Solid fertilizers and soil conditioners — Test sieving*

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 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 and the following apply.

3.1

superphosphate

The product obtained by treating phosphate rock with sulphuric acid. It is also known as single superphosphate.

3.2

concentrated superphosphate

The product obtained by treating phosphate rock with phosphoric acid or phosphoric acid and sulphuric acid mixture. It is also called ‘enriched superphosphate’ or ‘triple superphosphate’.

3.3

available phosphorus

the fraction of phosphorus available to plants. This is the sum of the water-soluble and citrate-soluble phosphorus.

3.4
total phosphorus
the sum of available and citrate-insoluble phosphorus

4 Requirements

4.1 General requirements

The fertilizer shall be homogenous, free-flowing in the form of granules, pellets, or crystals and free from foreign matter and hard lumps

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

Superphosphate fertilizer shall comply with the following compositional limits in Table 1

Table 1 —Chemical requirements for superphosphates

Sl. No.	Characteristic	Requirement, %, by mass			Method of test
		Grade 1 Single Superphosphate	Grade 2 Single Superphosphate	Triple/ Concentrated Superphosphate	
(i)	Water-soluble phosphate (as P_2O_5), min	18.0	14.0	40	ISO 5316
(ii)	Available phosphate (as P_2O_5), min	21.5	16.5.	46.0	AOAC 969.02 / AOAC 993.31
(iii)	Free phosphoric acid (as P_2O_5), Max	4.0	4.0	4.0	Annex A
(iv)	Moisture, max	5.0	5.0.	4.0	EN 12048/ ISO 8189
(v)	Sulfur (as S), min	11	11	11	ISO 15178/ EN 16032

4.4 Heavy metal contaminants

The heavy metal contaminants, if present, shall not exceed the limits stipulated in Table 2.

Table 2 — Heavy metal contaminants limits

Sl. No.	Parameter	Limits, mg/kg	Test method
i)	Arsenic, As, max.	40	AOAC 2006.03/ ISO 17318
ii)	Cadmium, Cd, max.	45	AOAC 2006.03/ ISO 17318
iii)	Mercury, Hg, max.	0.1	AOAC 2006.03/ ISO 17318
iv)	Selenium, Se, max.	1.0	AOAC 2006.03
v)	Lead, Pb, max.	30.0	AOAC 2006.03/ ISO 17318
vi)	Nickel	120	AOAC 2006.03/ ISO 17318
vii)	Chromium, Cr Max.	500	AOAC 2006.03/ ISO 17318

5 Sampling

Sampling and sample preparation for inspection and testing shall be carried out in accordance with ISO 14820 parts 1 and 2.

6 Compliance

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

7 Packaging and labelling

7.1 Packaging

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

7.2 Labelling

7.2.1 Each package of the fertiliser shall be legibly and indelibly marked in accordance with ISO 7409, and with the following particulars:

a) Name of the product as superphosphate fertilizer;

b) type;

c) grade

d) name and physical address of the manufacturer/packer;

e) minimum water-soluble phosphates stated as P_2O_5 content of the material;

f) net weight of the material in the package;

g) content of fluorine and sulfur;

h) manufacture and expiry or best before date;

i) lot or batch number;

j) handling, use and storage instructions; and

k) warning statement similar or closely similar in meaning to the statements in 1, 2 and 3 preceded by the words: “WARNING”:

   1) do not swallow. The dust from this product may act as an irritant, avoid inhalation or contact with eyes and skin;

   2) this product may contain fluorine as an impurity; do not feed this product to livestock or use in feedstock mixtures. If top dressing pastures do not graze for 3 weeks or until rain or irrigation is received; and

   3) this product may contain heavy metal as impurities; its repeated use may therefore lead to accumulation of the same in the soil. Depending on soil characteristics, irrigation water quality, plant species and variety, crop uptake of heavy metals may exceed the maximum limits allowed in local food standards; in pasture, the offal from grazing animal may also exceed these limits; therefore its application in accordance to local regulation is advised.

7.2.2 Bulk containers

Where the product is distributed in bulk, the marking information shall accompany the delivery note to the purchaser.

8 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.

9 Material safety

Each consignment must be accompanied by a Material Safety Data Sheet (MSDS) and a certificate of analysis (COA).

Annex A
(normative)

Determination of free phosphoric acid

A.1 Purpose

This method determines the percent of phosphoric acid (P_2O_5) in commercial fertilizers by colorimetry.

A.2 Reagents and apparatus

1) An Atomic Absorption Spectrophotometer or a suitable colorimeter.

2) Nitric Acid (HNO_3), 1.42 specific gravity.

3) Hydrochloric Acid (HCl), 1.19 specific gravity.

4) Potassium Phosphate, Monobasic (KH_2PO_4), Primary Standard Grade, dried at 105-110 °C for several hours prior to use.

5) Ammonium Molybdate ($(NH_4)6MO_7O_{24}{\bullet}4H_2O$), Reagent Grade.

6) p-Methylaminophenol Sulfate, Reagent Grade.

7) Sodium Acid Sulfite ($NaHSO_3$), 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 KH_2PO_4 into a 500-mL volumetric flask. Add 30 ml of HNO_3 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 HNO_3 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 HNO_3 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 colour 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 colour 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 (P_2O_5)

Bibliography

Working Group to identify and acknowledge useful literature used in the preparation of this standard.

KS 46:2011, *Granulated superphosphate fertilizer — Specification*