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Fertilizers, soil conditioners and beneficial substances — Determination of EDTA soluble phosphorus content in inorganic fertilizers

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 134, *Fertilizers, soil conditioners and beneficial substances.*

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Owing to the variation of basic phosphate component, character and processing technology, traditionally, different extraction solutions and methods have been utilized for the determination of the phosphorus content of phosphate fertilizers. Extraction solutions currently employed for phosphorus content determination include alkaline ammonium citrate (i.e. Petermann's solution), neutral ammonium citrate, citric acid, ethylene diamine tetraacetic acid (EDTA) and citric acid, or just EDTA. Each of these extractants is designed to target specific phosphate components of phosphorus-based fertilizers.

Due to rapid developments in the modern fertilizer industry, especially with the formulation of compound/complex fertilizers, many phosphate fertilizers may have multiple phosphorus-containing components. The co-existence of these various phosphorus sources in a compound/complex fertilizer can complicate the effective extraction and determination of the phosphorus content.

Fertilizers, soil conditioners and beneficial substances — Determination of EDTA soluble phosphorus content in inorganic fertilizers

1 Scope

This document specifies the method for the determination of the EDTA soluble phosphorus content of inorganic fertilizers. The method is applicable for fertilizers composed of or blended from multiple sources such as superphosphate, ammonium phosphate, triple superphosphate, and/or nitrophosphate. It is not suitable for fertilizers containing calcium magnesium phosphates.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this proposed standard. 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*

ISO 8358, *Solid fertilizers — Preparation of samples for chemical and physical analysis*

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

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this proposed standard, the terms and definitions given in ISO 8157 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

The EDTA soluble phosphorus content in inorganic fertilizers (calcium magnesium phosphate excluded) is extracted by ethylene diamine tetraacetic acid disodium salt solution. Phosphorus content in the extracting solution in the form of orthophosphate reacts with quimociac reagent in the acid medium to form yellow quinolinium molybdophosphate precipitate. The EDTA soluble phosphorus content is determined by gravimetric quinolinium molybdophosphate method.

5 Reagents

WARNING — Nitric acid is both corrosive and toxic. Refer to the applicable safety data sheet (SDS). Quinoline is irritating to the eyes, skin and respiratory system. Refer to the applicable SDS. The related operations shall be performed in the fume hood. This document does not point

out all possible safety problems, and the user shall bear the responsibility to take proper safety and health measures.

NOTE Related laws and regulations of corresponding countries and/or regions can apply to these operations.

Analytical grade reagent (AR) chemicals shall be used in all tests, unless otherwise indicated. The purity of water used throughout shall be understood to mean reagent water with electrical resistivity ≥ 18,2 MΩ·cm.

5.1 Ethylene diamine tetraacetic acid disodium salt dihydrate ($C_{10}H_{14}N_2Na_2O_8{\cdot}2H_2O$) solution (37,5 g/l, eq. 0,1 mol/l).

Dissolve 37,5 g of EDTA with a certain amount of deionized water in a 1 000 ml beaker. Dilute by adding deionized water to the 1 000 ml marking. Mix well.

5.2 Quimociac reagent.

The reagents used in the preparation of quimociac reagent:

— sodium molyddate ($Na_2MoO_4.2H_2O$, CAS: 10102-40-6);

— citric acid ($C_6H_8O_7.H_2O$, CAS: 5949-29-1);

— nitric acid (HNO_3, 65 – 68 %, CAS: 7697-37-2);

— quinoline (2-azabicyclo[4.4.0]deca-1(6),2,4,7,9-pentaene, C_9H_7N, CAS: 91-22-5);

— acetone (C_3H_6O, CAS: 67-64-1).

To make solution a, add 70g sodium molybdate ($Na_2MoO_4{\cdot}2H_2O$) into a 400 ml beaker. Add 100 ml water and stir to dissolve.

To make solution b, add 60 g citric acid ($C_6H_8O_7{\cdot}H_2O$) into a 1l beaker. Add 100 ml water and stir to dissolve, then add 85 ml nitric acid (HNO_3, 65 % to 68 %).

To make solution c, add solution a into solution b, and mix well.

To make solution d, mix 85 ml nitric acid (HNO_3, 65 % to 68 %) with 100 ml water in a 400 ml beaker, then add 5 ml quinoline (2-azabicyclo[4.4.0]deca-1(6),2,4,7,9-pentaene, C_9H_7N,CAS: 91-22-5). Mix well.

Add solution d into solution c, mix well and stand overnight. Filter the mixed solution with filter paper, add 280 ml of acetone into the filtrate, then dilute the solution by adding water to 1 l. The as-prepared quimociac reagent should be stored in a polyethylene bottle and preserved in a dark place to avoid light and heat.

NOTE If the colour of quimociac reagent turns to light blue (caused by light), an appropriate amount of potassium bromate solution (KBr, 10 g/l) can be added into the quimociac reagent until the colour disappears.

5.3 Nitric acid solution (HNO_3, 1 + 1).

Dilute a certain volume of 65 % to 68 % nitric acid (ρ = 1,39 g/ml to 1,40 g/ml) with equal volume of water.

6 Apparatus and materials

6.1 Ordinary laboratory apparatus.

6.2 Electric thermostatic drying oven, temperature can be maintained at 180 ± 2 °C.

6.3 Glass filtering crucible, type IV (pore size: 5μm to15 μm), volume of 30 ml.

6.4 Thermostatic water bath oscillator, equipped with reciprocating oscillator or rotating oscillator which temperature can be maintained at 60 ± 2 °C.

7 Test procedure

7.1 General

Replicate experiments shall be done for the determination.

7.2 Preparation of test sample

Prepare the test samples for analysis in accordance with ISO 8358 and ISO 14820-2.

7.3 Weigh of test portion

Weigh the appropriate amount of the test portion (accurately to 0,000 2 g) to obtain between 100 mg to 200 mg P_2O_5.

7.4 Extraction of EDTA soluble phosphorus

According to 7.3, weigh the appropriate amount of the test portion on the filter paper and wrap it, put it into a 250 ml volumetric flask. Add 150 ml EDTA solution (5.1), plug the flask tightly, shake the volumetric flask to break apart the filter paper and disperse the sample into the solution, then put it in the thermostatic water bath oscillator (6.4) pre-set at 60 °C ± 2 °C, oscillate for 1 h at constant temperature (set the oscillation frequency to make the sample in volumetric flask can flip over freely, and to keep the sample suspended in the extracting solution while avoiding collection of excessive amounts of sample on the upper portions of the interior of the volumetric flask). Then take the volumetric flask out, cool down to ambient temperature, dilute the solution by water to scale, mix well and dry filter. Discard the first part of the filtrate. Label the remaining filtrate as solution I for determining EDTA soluble phosphorus content.

7.5 Determination of the EDTA soluble phosphorus content

Draw the appropriate amount of solution I (volume of v_1) by single-line pipette, place into a 500 ml beaker, add 10 ml nitric acid solution (5.3), dilute by water to 100 ml, heat on the hot plate till boiling, and hold for 2 min to 3 min, remove and add 35 ml quimociac reagent (5.2), then cover with a watch glass. Continue heating on the hot plate for 1 min or place covered-beaker into water bath pre-set at close to boil until the precipitation is complete and the precipitate has settled to the bottom of the beaker. Remove the beaker from the hot plate or water bath and allow to cool to ambient temperature.

Filter it by glass filter crucible (6.3), which was pre-dried to constant weight in drying oven at 180 °C ± 2 °C beforehand. Filter the supernarant first, and then wash the precipitate by decantation 1 to 2 times, use 25 ml of water each time, then transfer the precipitate to the glass filter crucible with water and wash with an additional water, the total amount of water was 125 to 150 ml. Place the glass filter crucible containing the precipitate in the drying oven pre-set at 180 °C ± 2 °C, drying for 45 min after the temperature reach 180 °C, take it out and transfer it into a desiccator and cool it down to ambient temperature, weigh the precipitation together with the filter.

7.6 Blank test

Except for not adding samples, the blank test should be carried out exactly the same as the sample test using the same amount of reagent by identical analysis procedures.

8 Calculation and expression of results

8.1 General

The EDTA soluble phosphorus content (w_1) is expressed by a mass fraction percentage of P_2O_5 as given in Formula (1):

$$w_1 = \frac{(m_1 - m_2) \times 0{,}03207}{m_A \times \frac{v_1}{250}} \times 100 \quad (1)$$

where

m_1 is the weight of quinolinium molybdophosphate precipitation during the sample test, in g;

m_2 is the weight of quinolinium molybdophosphate precipitation during the blank test, in g;

m_A is the weight of test portion used during the sample test for determination of EDTA soluble phosphorus content, in g;

v_1 is the volume of test solution I used during the sample test for determination of EDTA soluble phosphorus content, in ml;

250 is the total volume of test solution I, in ml.

The reported value is the arithmetic average of two parallel determinations using separate solid sample aliquots, and shall be rounded off to two significant figures after the decimal point.

8.2 Precision

8.2.1 Ring test

Details of ring test on the precision of the method are summarized in Annex A.

8.2.2 Repeatability, *r*

For EDTA soluble phosphorus content of all levels, the repeatability limit *r* is 0,25, in a mass fraction percentage.

8.2.3 Reproducibility, *R*

For EDTA soluble phosphorus content of all levels, the reproducibility limit *R* is 0,50, in a mass fraction percentage.

9 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) test method used with reference to this document, i.e. ISO 22018:—;

c) test results obtained;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

All operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method, which might have influenced the test results.