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Fertilizers, soil conditioners and beneficial substances — Determination of ammonium citrate, disodium-EDTA soluble phosphorus and potassium by ICP-OES in inorganic fertilizers

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Fertilizers, soil conditioners and beneficial substances — Determination of ammonium citrate, disodium-EDTA soluble phosphorus and potassium by ICP-OES in inorganic fertilizers

Engrais, amendements et substances bénéfiques – Détermination de la teneur en phosphore et potassium solubles dans le citrate d'ammonium et l'EDTA disodique par ICP-OES dans les engrais inorganiques

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Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms and definitions ... **1**
4 Principle ... **1**
5 Reagents ... **2**
6 Apparatus ... **3**
7 Sampling and sample preparation ... **4**
8 Procedure ... **4**
8.1 Extraction of P and K ... 4
8.2 Instrument conditions ... 4
8.3 Preparation of standards ... 5
8.3.1 Standards from reagent salts ... 5
8.3.2 Standards from commercial stock solutions ... 5
8.3.3 Calibration and wavelength splitting ... 7
8.3.4 Analysis ... 7
8.4 Quality control ... 7
8.4.1 Reference material ... 7
8.4.2 Duplicates ... 7
9 Calculations and expression of results ... **7**
9.1 Calculations ... 7
9.2 Precision ... 8
9.2.1 General ... 8
9.2.2 Repeatability, *r* ... 9
9.2.3 Reproducibility, *R* ... 9
10 Test report ... **10**
Annex A (informative) **Ring test background and results** ... **11**
Annex B (informative) **Critical factors** ... **24**
Bibliography ... **26**

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Foreword

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

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Introduction

Inductively coupled plasma-optical emission spectrometer (ICP-OES) instrumentation is becoming increasingly popular in fertilizer testing laboratories (see References [1], [2] and [3]). Because these instruments are automated and can determine multiple elements simultaneously, they offer significant productivity gains. Also, the method does not generate hazardous wastes. The ammonium citrate disodium etheylenediamine tetraacetic acid (EDTA) organic solvent and samples containing high phosphorus and/or potassium concentrations pose some analytical challenges for the instrumentation, which must be addressed. Due to the increasing use of this technology, this document was created.

For organic fertilizers and for liquid fertilizers containing phosphorous acid (H_3PO_3 or phosphite), an alternative method that specifically measures PO_4 is preferred since the ICP-OES measures all elemental phosphorus, which can result in a high bias for these types of fertilizer materials (see Reference [1]).

Due to the inability of the ICP-OES to differentiate between phosphorus species, under normal operating conditions, this method is primarily intended for fertilizer materials in which the source(s) of phosphorus is known. While this document can be used with minimal bias, the repeatability and reproducibility is not consistent with other standards routinely used for inspection at this time, such as EN 15959[4], EN 15477[5] or ISO 22018[6].

As a result, this document is intended for screening purposes, where a large number of fertilizer phosphorus and potassium results must be obtained as efficiently as possible.

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Fertilizers, soil conditioners and beneficial substances — Determination of ammonium citrate, disodium-EDTA soluble phosphorus and potassium by ICP-OES in inorganic fertilizers

1 Scope

This document specifies an inductively coupled plasma-optical emission spectrometer (ICP-OES) method for the determination of the ammonium citrate, disodium-etheylenediamine tetraacetic acid (EDTA) soluble phosphorus and potassium content in fertilizer extract solutions.

This document is applicable to all inorganic fertilizer products, whether compound or complex fertilizer products or blends thereof, and straight concentrated fertilizer products.

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 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, soil conditioners and beneficial substances — Vocabulary*

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*

3 Terms and definitions

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

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

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

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

4 Principle

Phosphorus and potassium contained in inorganic fertilizers that is soluble in a heated, dilute ammonium citrate and disodium-EDTA solvent is determined. The neutral ammonium citrate is the primary solvent for phosphate, while the EDTA is mainly included to chelate calcium, magnesium and other soluble metallic cations to minimize their interaction with phosphate and eliminate the water-wash step (see Reference [7]). Also, since this solvent is a relatively weak solution, it extracts primarily the highly soluble potassium compounds and has been used for fertilizer potassium determination since 1993. It compares very favourably with other soluble potash solvents such as ammonium oxalate. The user of this document is not obligated to determine both phosphorus and potassium; however, one of the main advantages of this approach is to measure both simultaneously in a single extract in a more productive, cost-effective way.

The resulting extract solution is tested by ICP-OES to quantify the levels of phosphorus and potassium, which can be converted to their equivalent P_2O_5 and K_2O concentrations. The extract solution is

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nebulized to produce a fine aerosol, which is introduced to a plasma or high energy source to ionize or excite electrons to higher energy orbitals. As these electrons return to ground state, they emit characteristic ultraviolet or visible wavelengths that are unique to each element. The intensity of the wavelength emission is proportionate to the concentration, which can be quantified by various detector types including a photomultiplier tube (PT), a charge injection device (CID) or a charge coupled device (CCD).

5 Reagents

5.1 Ammonium citrate, dibasic, $(NH_4)_2HC_6H_5O_7$, with a molecular weight (MW) of 226,19 and a purity greater than 98 % or 980,0 g/kg.

5.2 EDTA, disodium salt, dihydrate, $C_{10}H_{14}N_2Na_2O_8{\cdot}2H_2O$, with a MW of 372,24 and a purity greater than 98 % or 980,0 g/kg.

5.3 Ammonium hydroxide, NH_4OH, with a MW of 35,05 and a purity equal to 28,0 % to 30,0 % as NH_3 or ρ = 0,91 g/ml.

5.4 Nitric acid, HNO_3, 67 % to 70 %, with a ρ = 1,40 g/ml.

5.5 Potassium dihydrogen phosphate, KH_2PO_4, with a MW of 136,09, with a purity greater than 99 % or 990,0 g/kg[1)].

5.6 Potassium chloride, KCl, with a MW of 74,55 and a purity greater than 99 % or 990 g/kg.

5.7 Potassium nitrate, KNO_3, with a MW of 101,10 with a purity greater than 99 % or 990,0 g/kg[2)].

5.8 Octyl phenol ethoxylate[3)].

5.9 Beryllium stock solution, of 10 000 µg/ml.

5.10 Scandium stock solution, of 10 000 µg/ml.

5.11 Cesium chloride, CsCl, MW of 168,36, with a purity greater than 99,99 % or 999,99 g/kg.

5.12 Lithium nitrate, $LiNO_3$, MW of 68,95, with a purity greater than 99 % or 990,0 g/kg.

5.13 Citrate-EDTA extraction solution (0,11 M ammonium citrate and 0,033 M disodium-EDTA).

Weigh and transfer 25 g disodium-EDTA (5.2) and 50 g dibasic ammonium citrate (5.1) to a 2 l volumetric flask containing approximately 500 ml of deionized (or equivalent) water. Adjust the pH to near neutral by adding 30 ml of ammonium hydroxide and water (1:1, volume fraction) solution in a fume hood. Adjust the final pH to 7,00 (±0,02) using a pH electrode and meter while adding the ammonium hydroxide and water (1:1, volume fraction) solution drop-by-drop and stirring. After obtaining a stable pH of 7,00 ±

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3) Triton X-100 is the trade name of a product supplied by Sigma-Aldrich. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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0,02, bring the flask to volume with deionized water (or equivalent) and mix. Larger volumes of this solution can be prepared; however, it is susceptible to microbial degradation resulting in a shelf life of no more than three weeks, if stored in a dark location.

5.14 Octyl phenol ethoxylate, 0,5 % or 5 ml/l.

Add 1 ml of octyl phenol ethoxylate (5.8) to a 200 ml volumetric flask and dilute to volume with deionized water.

5.15 Internal standard/ionization buffer (10 µg/ml Sc in 0,018 M CsCl and 4 % nitric acid).

Add 1,00 ml of 10 000 µg/ml scandium stock standard, 3 g cesium chloride, 40 ml nitric acid, and 1 ml of 0,5 % octyl phenol ethoxylate (5.14) to a 1 l volumetric flask containing approximately 500 ml of deionized (or equivalent) water. Bring flask to volume with deionized (or equivalent) water and mix. If beryllium is used as an internal standard, add 4,00 ml of 10 000 µg/ml beryllium stock standard to obtain a concentration of 40 µg/ml beryllium.

Alternatively, lithium from $LiNO_3$ can be substituted for cesium as an ionization buffer at 7 g/l $LiNO_3$.

5.16 Nitric acid rinse solution, 1 %.

Add 10 ml of nitric acid (5.4) to approximately 500 ml of deionized or equivalent water contained in a 1 l volumetric flask and bring to volume.

5.17 2 500 µg/ml phosphorus from phosphate (PO_4).

Add 2,746 1 g of potassium dihydrogen phosphate (5.5) to a 250 ml volumetric flask, then bring to volume with deionized (or equivalent) water. This standard also contains 3 156,5 µg/ml K; also, this standard can take several hours to solubilize and stabilize. This standard should be prepared fresh each time. Alternatively, a commercial custom standard from a phosphate source prepared in a water matrix and preserved with a biocide is acceptable.

The weight added should be adjusted for the purity of the reagent used.

NOTE A commercial stock standard preserved in acid is not acceptable as the acid changes the matrix of the pH neutral ammonium citrate EDTA solution and produces erroneous results.

5.18 7 500 µg/ml potassium from potassium chloride.

Add 1,430 0 g of potassium chloride (5.6) to a 100 ml volumetric flask, then bring to volume with deionized (or equivalent) water. Alternatively, 1,939 4 g of potassium nitrate (5.7) can be used rather than potassium chloride. This standard should be prepared fresh each time. Alternatively, a commercial custom standard from a potassium source prepared in a water matrix and preserved with a biocide is acceptable.

The weight added should be adjusted for the purity of the reagent used.

NOTE A commercial stock standard preserved in acid is not acceptable as the acid changes the matrix of the pH neutral ammonium citrate EDTA solution and produces erroneous results.

6 Apparatus

6.1 Analytical balance, capable of weighing to 0,1 mg.

6.2 pH meter, with a readability to 0,01.

6.3 pH combination electrode.

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