International Standard 6598

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Fertilizers — Determination of phosphorus content — Quinoline phosphomolybdate gravimetric method

Engrais — Dosage du phosphore — Méthode gravimétrique au phosphomolybdate de quinoléine

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Fertilizers — Determination of phosphorus content — Quinoline phosphomolybdate gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method using quinoline phosphomolybdate for the determination of phosphorus (expressed as diphosphorus pentaoxide) in a solution prepared from natural mineral phosphates or fertilizers.

2 References

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

ISO 7497, *Fertilizers — Extraction of phosphates soluble in mineral acids.*

3 Principle

Precipitation, after hydrolysis if necessary, of orthophosphate ions in the form of quinoline phosphomolybdate, in an acid medium and in the presence of acetone, at approximately 75 °C. Filtration, washing, drying and weighing of the precipitate obtained.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Acetone, pure.

4.2 Sodium molybdate dihydrate.

4.3 Quinoline, pure, distilled.

4.4 Citric acid monohydrate.

4.5 Nitric acid, ϱ_{20} = 1,38 g/ml, 63 % (m/m) solution.

NOTE — If acid of another concentration is used, adjust the volume added accordingly.

4.6 Potassium dihydrogenorthophosphate, previously dried at 105 °C.

4.7 Precipitation reagent, prepared as follows:

4.7.1 Solution A

Dissolve 70 g of the sodium molybdate dihydrate (4.2) in 100 ml of water in a 400 ml beaker.

4.7.2 Solution B

Dissolve 60 g of the citric acid monohydrate (4.4) in 100 ml of water in a 1 000 ml beaker. Add 85 ml of the nitric acid solution (4.5).

4.7.3 Solution C

Add solution A to solution B and mix.

4.7.4 Solution D

Mix 35 ml of the nitric acid solution (4.5) and 100 ml water in a 400 ml beaker and add 5 ml of the quinoline (4.3).

4.7.5 Solution E

Add solution D to solution C and mix. Leave overnight. Filter through a sintered glass filter, of porosity P 10 or P 16 (pore size index from 4 to 16 µm) and, if necessary, filter the first part of the filtrate again until a clear filtrate is obtained.

Do not wash the residue with water. Add 280 ml of the pure acetone (4.1) to the filtrate and dilute to 1 000 ml with water.

The solution may be kept for 1 month in a stoppered bottle protected from sunlight and heat.

5 Apparatus

Usual laboratory equipment and in particular

5.1 Conical flask, wide necked, of capacity 500 ml.

5.2 Sintered glass filter crucible, of porosity P 10 or P 16 (pore size index from 4 to 16 µm).

5.3 Oven, capable of being maintained at 250 ± 5 °C.

5.4 Flameless heating apparatus.

5.5 Desiccator, containing silica gel.

6 Procedure

6.1 Aliquot portion

From the solution of fertilizer obtained by extraction in accordance with ISO 5316 or ISO 7497, take an aliquot portion containing preferably 10 to 20 mg of diphosphorus pentaoxide and not more than 20 ml of citrate solution.

Transfer to the conical flask (5.1). Add 25 ml of the nitric acid solution (4.5). Dilute to 100 ml with water.

6.2 Determination

6.2.1 Hydrolysis

If non-orthophosphates are present in the solution carry out a hydrolysis as follows.

Heat the contents of the conical flask gently until they begin to boil and boil until hydrolysis is complete (generally 1 h). Ensure that losses by splashing and excessive evaporation which would reduce the original volume by more than half are avoided, for example by using a reflux condenser system. When the hydrolysis is complete, make up to the original volume with water.

NOTE — If phosphorus is present in the form of orthophosphate only, the hydrolysis is not necessary.

6.2.2 Precipitation

Operating under a fume hood, add, without stirring, 100 ml of the precipitation reagent (4.7.5, solution E) from a measuring cylinder to the contents of the flask.

Cover with a watch-glass and heat the flask immediately using the apparatus (5.4) so that within 10 min boiling just starts (75 to 80 °C) and maintain for about 30 s. Then remove the flask from the heating apparatus and allow to cool for at least 30 min. Swirl 3 or 4 times during cooling. Allow to settle.

6.2.3 Weighing of the crucible

Heat the filter crucible (5.2) in the oven (5.3) maintained at 250 ± 5 °C, to constant mass. Weigh the crucible to the nearest 0,000 1 g after cooling in a desiccator (5.5) containing silica gel in good condition.

6.2.4 Filtration and washing

Decant the supernatant liquid through the filter crucible (5.2), using suction. Wash the precipitate in the conical flask with 30 ml of water. Decant and filter the solution. Repeat this procedure five times. Quantitatively transfer the remainder of the precipitate into the crucible. Wash four times, adding the washing water only when filtration is practically complete. Continue to apply suction until all excess liquid has been extracted.

6.2.5 Drying and weighing

Wipe the outside of the crucible with a filter paper. Heat the crucible in the oven (5.3), maintained at 250 ± 5 °C, to constant mass (15 to 30 min). Cool the crucible in a desiccator (5.5) containing silica gel in good condition, transfer to a balance and then weigh immediately to the nearest 0,000 1 g.

6.3 Check of reagents

For each series of determinations, carry out a check of the reagents using only the reagents and solvents in the proportions used for the extraction (citrate solution, etc.).

6.4 Check test

It is recommended that a determination on an aliquot portion, containing 10 mg of diphosphorus pentaoxide, of potassium dihydrogenorthophosphate (4.6) solution be carried out to check the validity of the method.

7 Expression of results

The diphosphorus pentaoxide content, expressed as a percentage by mass, is equal to

$$\frac{141,95}{4\,425,84} \times m_1 \times \frac{V_1}{V_0} \times \frac{100}{m_0} = 3,207 \times \frac{V_1}{V_0} \times \frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the precipitate;

V_0 is the volume, in millilitres, of the aliquot portion taken from the extraction solution;

V_1 is the volume, in millilitres, of the extraction solution;

141,95 is the relative molecular mass of diphosphorus pentaoxide;

4 425,84 is twice the relative molecular mass of quinoline phosphomolybdate.

8 Precision

8.1 Repeatability

The absolute value of the difference between two individual results (mass of quinoline phosphomolybdate about 350 mg), using equal volumes of the same solution of extraction, under

the same conditions (same operator, same apparatus, same laboratory and short interval of time) shall be less than 2 mg.

8.2 Reproducibility

The absolute value of the difference between two individual results (mass of quinoline phosphomolybdate about 350 mg), using equal volumes of the same solutions of extraction, under different conditions (different operators, different apparatus, different laboratories and/or different times) shall be less than 6 mg.

9 Test report

The test report shall include the following information:

a) the reference of the method used, i.e. ISO 6598;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operations not specified in this International Standard, or in the International Standards to which reference is made, or regarded as optional, together with any circumstances likely to have affected the results.

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