INTERNATIONAL STANDARD ISO 4313

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Washing powders — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

Poudres à laver — Dosage de l'oxyde de phosphore(V) total — Méthode gravimétrique au phosphomolybdate de quinoléine

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FOREWORD

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Washing powders — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the total phosphorus(V) oxide content of commercial washing powders.

2 REFERENCE

ISO 607, *Surface active agents — Detergents — Methods of sample division.*[1]

3 PRINCIPLE

Hydrolysis of polyphosphates by nitric acid. Precipitation of phosphates in the form of quinoline phosphomolybdate in acetone solution. Drying and weighing of the precipitate.

4 REAGENTS

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

4.1 Nitric acid, ρ_{20} approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution.

4.2 Citromolybdic reagent.

4.2.1 Dissolve 70 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in 150 ml of water in a 400 ml beaker.

4.2.2 Dissolve 60 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 150 ml of water in a 1 000 ml beaker and add 85 ml of the nitric acid solution (4.1).

4.2.3 Pour solution (4.2.1) into solution (4.2.2), while stirring.

4.2.4 Place 100 ml of water in a 400 ml beaker and add 35 ml of the nitric acid solution (4.1), followed by 5 ml of pure quinoline.

4.2.5 Pour solution (4.2.4) into solution (4.2.3), while stirring, and allow to stand overnight. Without rinsing with water[2], filter all the liquid through the filter crucible (5.6) into the 1 000 ml one-mark volumetric flask (5.2).

4.2.6 Add to solution (4.2.5) 280 ml of acetone, dilute to the mark with water and mix.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Polyethylene beakers, of capacity 400, 600, 1 000 and 2 000 ml.

A polyethylene beaker is preferable to a glass beaker, as the active solution has to be prevented from coming into contact with glass if determination of the silica content is also to be carried out.

5.2 One-mark volumetric flask, of capacity 1 000 ml, complying with the requirements of ISO 1042.

5.3 One-mark volumetric flask, of capacity 100 ml, complying with the requirements of ISO 1042.

5.4 One-mark pipettes, of capacity 5, 10, 20, 25 and 50 ml, checked periodically and complying with the requirements of Class A of ISO/R 648.

5.5 Filter flask, of capacity 500 ml.

5.6 Filter crucible, with sintered glass disks having a porosity of P16 (pore size index 10 to 16 μm), complying with the requirements of ISO 4793[3]. Before use, dry this crucible for 1 h in the oven (5.7), controlled at 260 ± 20 °C, and cool in a desiccator.

5.7 Oven, capable of being controlled at 260 ± 20 °C.

5.8 Mechanical stirrer.

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1) In preparation. (Revision of ISO/R 607.)

2) If necessary, the first portions should be passed through the filter crucible again until a clear filtrate is obtained.

3) In preparation.

6 SAMPLING

The laboratory sample of washing powder shall be prepared and stored according to the instructions given in ISO 607.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,01 g, about 10 g of the laboratory sample.

7.2 Determination

Transfer the test portion (7.1) to the 2 000 ml beaker (5.1). Fill the one-mark volumetric flask (5.2) to the mark with water at 35 to 40 °C and add to the test portion, allowing a few seconds for drainage. Stir vigorously with the stirrer (5.8) for about 3 min so as to dissolve the test portion, apart from possible small amounts of insoluble silicate, etc., (solution L_1).

Take 25,0 ml of the test solution (L_1) by means of the 25 ml pipette (5.4), transfer to the 100 ml one-mark volumetric flask (5.3) and dilute to the mark with water (solution L_2).

Using one of the pipettes (5.4), take a volume of solution L_2, containing between 5 and 20 mg of phosphorus(V) oxide (for example 20 ml for a washing powder containing about 20 % (*m*/*m*) of phosphorus(V) oxide).

Transfer this solution to 600 ml tall-form beaker. Add 15 ml of the nitric acid solution (4.1) and dilute to about 100 ml. Cover the beaker with a watch-glass, bring the contents gently to the boil and keep boiling gently for 30 min.

If necessary, filter the solution to remove any silica that may have been precipitated and wash the filter with a volume of water corresponding approximately to the amount evaporated during the hydrolysis.

Place the beaker containing the warm solution in a fume cupboard and add 50 ml of the citromolybdic reagent (4.2), without stirring. Cover with a watch-glass and heat immediately with a suitable apparatus (not a flame) so that a temperature of about 75 °C (incipient boiling) is reached in 10 to 15 min. Maintain this temperature for not more than 30 s.

Remove the beaker from the heater and allow to cool to ambient temperature, stirring it three or four times while it is cooling. Allow the precipitate that has formed to settle.

Fit the filter crucible (5.6), which has been previously weighed, on the flask (5.5) with a vacuum connection.

Transfer the decanted solution to the crucible, leaving as much of the precipitate as possible in the beaker. Wash the precipitate six times by decanting it, using 30 ml of water each time. Transfer the precipitate quantitatively to the crucible with the aid of a wash-bottle. In order to do this, wash the beaker four times with water, adding each portion of the washing water to the crucible only after it has been sucked dry. Finally, pack the precipitate thoroughly in the filter crucible (5.6), place it in the oven (5.7), controlled at 260 ± 20 °C, and heat for 1 h. Remove the crucible from the oven, cool in a desiccator and weigh.

Repeat the processes of heating, cooling and weighing until two successive weighings do not differ by more than 0,001 g.

NOTE — It is advisable to leave the filter crucible to cool in the desiccator for the same length of time prior to weighing the precipitate as the crucible was left before weighing.

7.3 Blank test

In parallel with the determination, carry out a blank test following the same procedure and using the same quantities of all the reagents but omitting the test portion. The mass of the precipitate should not be greater than 1,5 mg. If it is greater, renew the reagents.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

The total phosphorus(V) oxide content of the washing powder, expressed as a percentage by mass, is given by the formula

$$\frac{(m_1 - m_2) \times 0{,}032\,07 \times 1\,000 \times 100 \times 100}{V \times 25 \times m_0}$$

$$= \frac{(m_1 - m_2) \times 12\,828}{V \times m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1)

m_1 is the mass, in grams, of the quinoline phosphomolybdate precipitate obtained in (7.2);

m_2 is the mass, in grams, of the precipitate obtained in the blank test (7.3);

V is the volume, in millilitres, of solution L_2 used for the determination;

0,032 07 is the mass, in grams, of phosphorus(V) oxide corresponding to 1 g of quinoline phosphomolybdate $[(C_9H_7N)_3H_3(PO_4.12MoO_3)]$.

8.2 Repeatability

The maximum difference found between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst using the same apparatus, shall not exceed 0,5 % for phosphorus(V) oxide contents between 18 and 30 % (*m*/*m*).

8.3 Reproducibility

The difference between the results obtained with the same sample in two different laboratories shall not exceed 1,1 % for phosphorus(V) oxide contents between 18 and 30 % (*m*/*m*).

9 TEST REPORT

The test report shall include the following particulars :

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

b) the reference of the method used;

c) the results and the method of expression used;

d) the test conditions;

e) any operations not included in this International Standard, or regarded as optional, as well as any incidents likely to have affected the results.

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