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Washing powders — Determination of active oxygen content — Titrimetric method

Poudres à laver — Dosage de l'oxygène actif — Méthode titrimétrique

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FOREWORD

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International Standard ISO 4321 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in August 1975.

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It has been approved by the member bodies of the following countries :

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Washing powders — Determination of active oxygen content — Titrimetric method

0 INTRODUCTION

Because of the specificity of washing powders and the relative stability of peroxyhydrates, the dissolution of washing powders is likely to be incomplete and may result in a loss of oxygen; these disadvantages have been avoided by adoption of a particular method of dissolution appropriate to the determination of active oxygen content.

1 SCOPE

This International Standard specifies a method for the determination of the active oxygen content of commercial washing powders.

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2 FIELD OF APPLICATION

This method may be used for determining peroxyhydrates, for example sodium perborate; it should not be used for washing powders which contain, in addition to peroxyhydrates, products which react with acid permanganates under the analytical conditions.

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The method may be used in the presence of (ethylenedinitrilo)tetraacetic acid (EDTA) or other chelating agents of the same type, provided that the concentration of these products does not exceed 1 % (*m*/*m*).

3 REFERENCE

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

4 PRINCIPLE

Co-reduction, with liberation of oxygen, of the peroxyhydrate and potassium permanganate in an acid solution.

NOTES

1 The relatively long induction period that may occur with certain washing powders may be avoided by adding manganese sulphate.

2 Bismuth nitrate complexes with the EDTA or with any other amine acetate-based chelating agent, thus removing all possible interference.

3 If aluminium sulphate is added, a preferential reaction with the condensed phosphates may be obtained, and the formation of a complex with the manganese ions, which may occur in certain cases and which may lead to an end-point that is not sharp, may be avoided.

5 REAGENTS

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

5.1 Aluminium sulphate, octadecahydrate $[Al_2(SO_4)_3.18H_2O]$.

5.2 Sulphuric acid, solution containing bismuth and manganese.

Dissolve 2 g of bismuth nitrate pentahydrate $[Bi(NO_3)_3.5H_2O]$ and 4 g of manganese sulphate monohydrate ($MnSO_4.H_2O$) [or an equivalent quantity of tetra- or pentahydrate ($MnSO_4.4H_2O$ or $MnSO_4.5H_2O$)] in 1 000 ml of a 5 N sulphuric acid solution.

5.3 (If necessary) **Sulphuric acid,** solution containing aluminium, bismuth and manganese.

Dissolve 50 g of the aluminium sulphate (5.1), 5 g of bismuth nitrate pentahydrate and 5 g of manganese sulphate monohydrate in 1 000 ml of a 5 N sulphuric acid solution.

5.4 Potassium permanganate ($KMnO_4$), freshly standardized, approximately 0,1 N standard volumetric solution.

6 APPARATUS

Ordinary laboratory apparatus and

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

6.2 Conical flask, of capacity 500 ml, complying with the requirements of ISO 1773.

6.3 Mechanical stirrer.

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

7 SAMPLING

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

8 PROCEDURE

8.1 Test portion

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

8.2 Determination

NOTES

1 The determination shall be carried out as quickly as possible after dissolution of the test portion.

2 With regard to the prescribed procedure for dissolution of the test portion, the usual rules of use of volumetric glassware are not respected here; in fact, a compromise has been adopted which allows the test portion to be dissolved in an appropriate manner, depending on the nature of the determination.

Transfer the test portion (8.1) to a 2 000 ml beaker. Fill the one-mark volumetric flask (6.1) 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 (6.3) for 3 min to dissolve the test portion, apart from possible small amounts of insoluble silicate, etc. (solution L_1).

During the operation of dissolution, place 50 ml of the sulphuric acid solution (5.2) in the conical flask (6.2) and add the potassium permanganate solution (5.4), drop by drop, with constant swirling until a permanent pale pink coloration appears.

Using a pipette, take 100,0 ml of solution L_1 and transfer it to the conical flask.

Titrate with the potassium permanganate solution (5.4) until a pale pink colour, persisting for at least 15 s, appears.

If the end-point is not sharp, repeat the determination in the presence of about 1 g of the aluminium sulphate (5.1) or using, for example, 20 ml of the sulphuric acid solution (5.3).

9 EXPRESSION OF RESULTS

9.1 Method of calculation

The active oxygen content of the washing powder is given, as a percentage by mass, by the formula

$$\frac{V \times T \times 8{,}0}{m}$$

where

V is the volume, in millilitres, of the standard volumetric potassium permanganate solution (5.4) used for the determination;

T is the exact normality of the standard volumetric potassium permanganate solution (5.4) used;

m is the mass, in grams, of the test portion (8.1).

9.2 Repeatability

The maximum difference between the results of two determinations carried out in rapid succession on the same product by the same analyst, using the same apparatus, should not exceed 1,3 % of the mean value found for an active oxygen content of about 2 % (*m/m*).

9.3 Reproducibility

The maximum difference between results obtained on the same sample, in two different laboratories, should not exceed 5 % of the mean value found for an active oxygen content of about 2 % (*m/m*).

10 TEST REPORT

The test report shall include the following particulars :

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

b) the reference of the method used (reference to this International Standard);

c) the results and the method of expression used;

d) the test conditions;

e) any operational details 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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