INTERNATIONAL STANDARD 761

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Acetic anhydride and butan-1-ol for industrial use — Determination of bromine number

Anhydride acétique et butanol-1 à usage industriel — Détermination de l'indice de brome

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 761-1968 and found it technically suitable for transformation. International Standard ISO 761 therefore replaces ISO Recommendation R 761-1968, to which it is technically identical.

ISO Recommendation R 761 had been approved by the member bodies of the following countries :

Australia	Germany	Poland
Austria	Hungary	Portugal
Belgium	India	Romania
Chile	Israel	Spain
Colombia	Italy	United Kingdom
Czechoslovakia	Japan	U.S.S.R.
Egypt, Arab Rep. of	Korea, Rep. of	Yugoslavia
France	Netherlands	

No member body had expressed disapproval of the Recommendation.

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Netherlands

Printed in Switzerland

Acetic anhydride and butan-1-ol for industrial use — Determination of bromine number

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the bromine number of acetic anhydride and of butan-1-ol for industrial use.

2 DEFINITION

bromine number : The number of grams of bromine consumed by 100 g of the sample under the conditions of test.

3 PRINCIPLE

Treatment of a test portion, in acid solution, with excess standard volumetric potassium bromide-potassium bromate solution, addition of potassium iodide solution and titration of the liberated iodine with a standard volumetric solution of sodium thiosulphate.

4 REAGENTS

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

4.1 Carbon tetrachloride.

4.2 Acetic acid, glacial.

4.3 Hydrochloric acid, ρ approximately 1,18 g/ml, about 36 % (*m/m*) solution.

4.4 Potassium iodide, 150 g/l solution.

4.5 Sodium thiosulphate, 0,10 N standard volumetric solution.

4.6 Potassium bromide-potassium bromate, approximately 0,1 N standard volumetric solution, accurately standardized.

Dissolve 10,2 g of potassium bromide and 2,8 g of potassium bromate in water and dilute to 1 000 ml. To determine the strength of this solution accurately, take 25,0 ml, add 5 ml of the potassium iodide solution (4.4) and 1 ml of the hydrochloric acid solution (4.3) and titrate with the standard volumetric sodium thiosulphate solution (4.5), adding 1 ml of the starch solution (4.7) towards the end of the titration.

NOTE — 1 ml of 0,10 N sodium thiosulphate solution corresponds to 0,007 99 g of Br_2.

4.7 Starch, 5 g/l solution, freshly prepared.

5 APPARATUS

Ordinary laboratory apparatus, and

5.1 Three iodine flasks, of capacity 500 ml, glass stoppered.

6 PROCEDURE

6.1 Blank test

Carry out a blank test at the same time as the determination, following the same procedure, but omitting the test portion.

6.2 Determination

6.2.1 Weigh accurately 3 to 5 g of the test sample, or pipette an equivalent quantity into a 50 ml one-mark volumetric flask containing 25 ml of the carbon tetrachloride (4.1) as a solvent. Dilute to the mark with the carbon tetrachloride and mix.

6.2.2 Immediately pipette 10 ml of this solution into one of the iodine flasks (5.1) containing 50 ml of the glacial acetic acid (4.2). Add 1 ml of the hydrochloric acid solution (4.3). Shield the flask and contents from direct sunlight and keep it at a temperature of 20 ± 5 °C. With constant swirling of the contents of the flask, titrate with the standard volumetric potassium bromide-potassium bromate solution (4.6) from a burette at the rate of 1 to 2 drops per second until the contents of the flask have assumed a yellow colour that persists for at least 5 s.

The yellow colour should match that obtained in a second iodine flask (5.1) by adding 2,5 ml of the standard volumetric potassium bromide-potassium bromate solution to 50 ml of the glacial acetic acid, 10 ml of the carbon tetrachloride and 1 ml of the hydrochloric acid solution.

6.2.3 Add from the burette an additional 5,0 ml of the standard volumetric potassium bromide-potassium bromate solution as quickly as possible, stopper the flask, and immediately continue swirling for 40 ± 5 s.

Then add 5 ml of the potassium iodide solution (4.4) by placing it in the cup of the flask and lifting the stopper slightly, thus preventing any possible loss of bromine vapour. Replace the stopper, shake vigorously, add 100 ml of water, shake the solution vigorously for 1 min and titrate it immediately with the standard volumetric sodium thiosulphate solution (4.5) from another burette, adding 1 ml of the starch solution (4.7) towards the end of the titration. This back-titration should normally use 5 to 10 ml of the standard volumetric sodium thiosulphate solution. If the volume used lies much outside this range, repeat the determination with a suitably adjusted mass of sample.

7 EXPRESSION OF RESULTS

The bromine number is given by the formula

$$40 \times \frac{[(V_1 - V_2) \times T] - [(V_3 - V_4) \times 0{,}10]}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric potassium bromide-potassium bromate solution (4.6) used for the determination;

V_2 is the volume, in millilitres, of the standard volumetric potassium bromide-potassium bromate solution (4.6) used for the blank test;

V_3 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (4.5) used for the determination;

V_4 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (4.5) used for the blank test;

T is the exact normality of the potassium bromide-potassium bromate solution (4.6);

m is the mass, in grams, of the test portion.

Report the result to the nearest 0,1.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.