INTERNATIONAL STANDARD 761

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

First edition — 1977-03-01

UDC 661.7 : 543.871.3 **Ref. No. ISO 761-1977 (E)**

Descriptors : chemical compounds, butanols, acetic anhydride, chemical analysis, determination, bromine number.

Price based on 2 pages

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.