

International Standard



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Butane-1,4-diol for industrial use — Determination of degree of unsaturation*Butane-diol-1,4 à usage industriel — Détermination du degré d'insaturation*

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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.

International Standard ISO 6794 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in December 1979.

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

Australia	Germany, F. R.	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Bulgaria	Italy	Switzerland
China	Korea, Rep. of	Thailand
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	Philippines	USSR
France	Poland	

No member body expressed disapproval of the document.

Butane-1,4-diol for industrial use — Determination of degree of unsaturation

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the degree of unsaturation, expressed as iodine value, of butane-1,4-diol for industrial use.

2 Definition

iodine value : The number of grams of iodine absorbed by 100 g of a substance under specified conditions of test.

NOTE — The iodine value is a measure of the degree of unsaturation and, in this determination, gives an indication of the presence of unsaturated impurities.

3 Principle

Treatment of a test portion with a solution of bromine in absolute methanol saturated with sodium bromide (Kaufman solution), and, after addition of potassium iodide solution, titration of the liberated iodine with standard volumetric sodium thiosulphate solution.

4 Reagents

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

4.1 Bromine, approximately 16 g/l solution in absolute methanol saturated with sodium bromide (Kaufman solution).

WARNING — This solution is highly flammable and toxic in contact with skin. Avoid contact with skin and eyes. In cases of contact, rinse immediately with plenty of water.

Dissolve 16 g (5,2 ml) of bromine in 1 000 ml of absolute methanol containing 120 g of sodium bromide (NaBr) which has been previously dried in an oven at 110 ± 2 °C for 2 to 3 h and cooled in a desiccator. The resulting solution is saturated at ambient temperature.

4.2 Sodium thiosulphate, standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1$ mol/l.

Check the concentration of the solution before use.

4.3 Potassium iodide, approximately 100 g/l solution.

4.4 Starch solution.

Triturate 1,0 g of soluble starch with 5 ml of water and, whilst stirring, pour the mixture into 100 ml of boiling water. Boil for a few minutes and cool.

Discard the solution after 2 weeks.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Iodine flask, of capacity 250 ml, fitted with a ground glass stopper.

6 Sampling¹⁾

Place the laboratory sample, representative of the material from the bulk, in a clean, dry, ground glass stoppered bottle of such a size that it is nearly filled by the sample.

If it is necessary to seal the bottle, take care to avoid any risk of contamination of the contents.

7 Procedure

7.1 Test portion

In the flask (5.1) weigh, to the nearest 0,01 g, 20 to 30 g of the laboratory sample (clause 6).

7.2 Blank test

Carry out a blank test at the same time as the determination, using the same procedure and the same quantities of all the reagents [except the sodium thiosulphate solution (4.2)] as used for the determination, but omitting the test portion.

7.3 Determination

Add to the flask containing the test portion (7.1), 50,0 ml of the bromine solution (4.1) and swirl to mix. Stopper the flask and,

1) The sampling of liquid chemical products for industrial use will form the subject of a future International Standard.

after a reaction time of 10 min in the dark, add 10 ml of the potassium iodide solution (4.3) and swirl to mix. Titrate the liberated iodine with the standard volumetric sodium thiosulphate solution (4.2) until the colour of the solution is pale yellow. Add about 2 ml of the starch solution (4.4) and continue the titration until the blue colour is just discharged.

8 Expression of results

The degree of unsaturation, expressed as iodine value, is given by the formula

$$\begin{aligned} & (V_0 - V_1) \times \frac{253,81}{2 \times 10^4} \times \frac{100}{m} \\ & = (V_0 - V_1) \times \frac{1,269}{m} \end{aligned}$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (4.2) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (4.2) used for the determination;

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

$\frac{253,81}{2 \times 10^4}$ is the mass, in grams, of iodine (I_2) corresponding to 1 ml of iodine solution $c(1/2 I_2) = 0,100$ mol/l.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

9 Test report

The test report shall include the following particulars :

- a) identification of the sample;
- b) the reference of the method used;
- c) the results and the method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard, or regarded as optional.