International Standard 755/1

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Butan-1-ol for industrial use — Methods of test — Part 1 : General

Butanol-1 à usage industriel — Méthodes d'essai — Partie 1 : Généralités

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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 755/1 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in February 1980.

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

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

The member body of the following country expressed disapproval of the document on technical grounds :

Poland

International Standards ISO 755/1, ISO 755/2 and ISO 755/3 cancel and replace ISO Recommendation R 755-1968, of which they constitute a technical revision.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Butan-1-ol for industrial use — Methods of test — Part 1 : General

1 Scope and field of application

This part of ISO 755 gives general instructions relating to test methods for butan-1-ol for industrial use.

It also specifies the methods to be used for the determination of density at 20 °C, the determination of distillation yield, the determination of dry residue after evaporation on a water bath, the determination of bromine number, the measurement of colour, the determination of water content and the determination of aldehydes and ketones content.

2 References

ISO 758, *Liquid chemical products for industrial use — Determination of density at* 20 °C.

ISO 759, *Volatile organic liquids for industrial use — Determination of dry residue after evaporation on a water bath — General method.*[1]

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 761, *Acetic anhydride and butan-1-ol for industrial use — Determination of bromine number.*

ISO 918, *Volatile organic liquids for industrial use — Determination of distillation yield — General method.*[2]

ISO 1843/3, *Higher alcohols for industrial use — Methods of test — Part 3 : Determination of carbonyl compounds content — Potentiometric method.*

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale).*

3 Sampling[3]

Store the laboratory sample in a clean, dry, air-tight bottle fitted with a ground glass stopper or a bottle fitted with a screw cap with an air-tight polyethylene seal, 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 contamination of the contents.

NOTE — A sample of not less than 1 000 ml is necessary for performing all the tests specified for the product.

4 Determination of density at 20 °C

Use the method specified in ISO 758.

5 Determination of distillation yield

Use the method specified in ISO 918, subject to the following modifications appropriate for 1-butanol.

5.1 Thermometer, complying with the requirements of ISO 918, sub-clause 5.1.2 and table 1.

Table 1 — Specifications for the thermometer

Thermometer range	Graduations	Maximum error	Maximum error in an interval of 10 °C
°C	°C	°C	°C
98 to 152	0,2	0,4	0,4

5.2 Temperature correction

If the corrected barometric pressure deviates from 1 013 mbar[4], apply a correction to the observed temperature by substracting 0,028 °C for every millibar above, or adding 0,028 °C for every millibar below, 1 013 mbar (see ISO 918, clause 9).

1) At present at the stage of draft. (Revision of ISO/R 759.)

2) At present at the stage of draft. (Revision of ISO/R 918.)

3) Sampling of liquid chemical products will form the subject of a future International Standard.

4) 1 bar = 10^5 Pa

5.3 Distillation

Regulate the rate of heating so that the first drop of distillate shall fall from the end of the condenser after 10 to 15 min (see ISO 918, sub-clause 7.2).

6 Determination of dry residue after evaporation on a water bath

Use the method specified in ISO 759.

7 Determination of bromine number

Use the method specified in ISO 761.

8 Measurement of colour

Use the method specified in ISO 2211.

9 Determination of water content

Use one of the methods specified in ISO 760.

10 Determination of aldehydes and ketones content

Use the method specified in ISO 1843/3, subject to the following modifications.

10.1 Ethanol, carbonyl compounds-free, purified as follows :

Boil under reflux 500 ml of ethanol (see ISO 1843/3, sub-clause 3.1) with 5 g of 2,4-dinitrophenylhydrazine and 5 drops of hydrochloric acid solution, ϱ approximately 1,19 g/ml, for 2 to 3 h. Distil off the ethanol slowly using a Widmer distillation column about 300 mm long and about 25 mm in diameter, or any other suitable column. Reject the first 50 ml of distillate and collect the next 400 ml, rejecting the remainder. If the distillate is found to be coloured, carry out a redistillation.

10.2 Determination (see ISO 1843/3, sub-clause 5.3)

Use a 400 ml beaker for the titration.

10.3 Expression of results

The carbonyl compounds content is given by the formulae shown in table 2.

Table 2 — Carbonyl compounds content

	Potassium hydroxide solution concentration	
	c(KOH) = 0,1 mol/l	c(KOH) = 0,01 mol/l
Method of expression	**Calculation formulae**	
milligrams per kilogram (mg/kg)	$\frac{100\,M\,(V_1 - V_0)}{m}$	$\frac{10\,M\,(V_1 - V_0)}{m}$
percentage by mass [% (m/m)]	$\frac{M\,(V_1 - V_0)}{m \times 10^2}$	$\frac{M\,(V_1 - V_0)}{m \times 10^3}$

In the formulae in table 2 :

V_0 is the volume, in millilitres, of the standard volumetric potassium hydroxide solution (see ISO 1843/3, sub-clause 3.3 or 3.4) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric potassium hydroxide solution (see ISO 1843/3, sub-clause 3.3 or 3.4) used for the determination;

M is the molar mass, in grams per mole, of the carbonyl compound, in terms of which the results are to be expressed;

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

11 Test report

The test report for each determination shall contain the following particulars :

a) an 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 the appropriate part of ISO 755 or in the other International Standards to which reference is made, or regarded as optional.

Annex

ISO publications relating to butan-1-ol for industrial use

ISO 755/1 — General.

ISO 755/2 — Determination of acidity — Titrimetric method.

ISO 755/3 — Sulphuric acid colour test.

ISO 761 — Determination of bromide index.[1)]

1) Also applicable to acetic anhydride for industrial use.