International Standard 6795

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Phthalic and adipic esters for industrial use — Determination of free alcohols content — Spectrometric method

Esters des acides phtalique et adipique à usage industriel — Dosage des alcools libres — Méthode spectrométrique

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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 6795 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in May 1982.

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

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Italy	Switzerland
Czechoslovakia	Korea, Dem. P. Rep. of	Thailand
Egypt, Arab Rep. of	Korea, Rep. of	USSR
France	Poland	
Germany, F. R.	Portugal	

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

China

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Phthalic and adipic esters for industrial use — Determination of free alcohols content — Spectrometric method

1 Scope and field of application

This International Standard specifies a spectrometric method for the determination of the free alcohols content of the phthalic and adipic esters for industrial use, containing the following organic radicals :

dibutyl
dioctyl
dinonyl
didecyl
diundecyl
didodecyl
ditridecyl

The method is applicable to products having free alcohol contents between 0,01 and 5,0 % (m/m).

2 Principle

Reaction of the free alcohol with the vanadium(V) sodium salt of quinolin-8-ol (8-hydroxyquinoline), to form a coloured complex. Extraction of the complex with toluene, followed by treatment with dichloroacetic acid. Spectrometric measurement at a wavelength of about 620 nm.

3 Reagents

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

WARNING — Avoid breathing the vapours or dusts of the reagents used for this determination and prevent any contact with skin and eyes. Take appropriate precautions with the flammable reagents.

3.1 Dichloroacetic acid, solution in glacial acetic acid.

WARNING — Dichloroacetic acid and glacial acetic acid are toxic and cause severe burns.

Mix 10 ml of dichloroacetic acid with 90 ml of glacial acetic acid.

3.2 Toluene, alcohol free.

WARNING — Toluene is toxic and highly flammable; hydrazinium (2+) sulfate is toxic, causes burns and is irritating to skin and eyes.

If an alcohol-free toluene is not available, purify it as follows :

In a 2 litre separating funnel, wash 1 000 ml of toluene with 100 ml of a 50 g/l hydrazinium (2+) sulfate (hydrazine sulfate) solution containing 1 g of potassium chromate; then wash with 100 ml of a 40 g/l sodium hidroxide solution and finally wash four times with water and dry over anhydrous sodium sulfate.

3.3 Sodium hydroxide, 4 g/l solution.

3.4 Standard solution of the alcohol corresponding to the alcohol to be determined.

Weigh, to the nearest 0,001 g, 1 g of the alcohol to be determined (see clause 1), place it in a 100 ml one-mark volumetric flask, dilute to the mark with the toluene (3.2) and mix.

Place 5,0 ml of this solution in a 100 ml one-mark volumetric flask, dilute to the mark with the same toluene and mix.

1 ml of this standard solution corresponds to 0,5 mg of the alcohol to be determined.

3.5 Reagent of vanadium(V) quinolin-8-ol (8-hydroxyquinoline) sodium salt

3.5.1 Preparation of the salt

WARNING — Ammonium vanadate and quinolin-8-ol are toxic.

Dissolve 1,35 g of ammonium vanadate (NH_4VO_3) in 35 ml of a 40 g/l sodium hydroxide solution in a 250 ml beaker. Add 15 ml of water and boil for 5 min to remove all the ammonia (solution A).

Dissolve 5 g of quinolin-8-ol in 35 ml of hot 40 g/l sodium hydroxide solution in a second 250 ml beaker. Add 40 ml of water, bring the solution to boiling and place it on a boiling water bath. Add solution A and stir for at least 10 min.

Then add to the beaker placed on the boiling water bath, drop by drop, a 2,5 % (V/V) acetic acid solution in the toluene (3.2), until the black precipitate formed no longer redissolves.

Filter the hot solution through a medium filter paper washed twice with hydrochloric acid (20 % m/m) and cool it in ice until yellow crystals of the sodium salt are deposited. Filter these off, wash with a few millilitres of water and dry them in vacuo over an efficient dehydrating agent. Grind down to a powder using a mortar and sieve through a 71 µm sieve conforming to the requirements of ISO 565. Use that which passes through the sieve.

3.5.2 Preparation of the reagent

WARNING — Dimethylformamide is toxic, flammable and harmful in contact with skin and eyes.

Weigh 0,2 g of the sodium salt (3.5.1) into a 150 ml conical flask, add 10 ml of dimethylformamide and shake to dissolve. Add 100 ml of the toluene (3.2) and attach the flask to a reflux condenser. Heat to a gentle reflux, and add 100 ml of a 2,5 % (*V*/*V*) acetic acid solution in the same toluene. Allow the solution to reflux for 2 min before cooling in ice water.

This reagent is stable for only 1 day.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Measuring cylinders, provided with ground glass stopper of capacity 25 and 10 ml.

4.2 Spectrometer.

4.3 Water bath, capable of being controlled at 60 ± 1 °C.

5 Sampling

Place the laboratory sample[1] in a clean, dry, dark-coloured glass bottle fitted with a ground glass stopper, of such a size that it is nearly filled by the sample.

If it is necessary to seal this bottle, care shall be taken to avoid the risk of contamination.

6 Procedure

6.1 Test portion and preparation of the test solution

Weigh the mass of laboratory sample indicated in the following table into a 25 ml one-mark volumetric flask and dilute to the mark with the toluene (3.2).

Expected alcohol content	Mass of test portion
% (*m*/*m*)	g
0,01 to 0,05	20,0
0,05 to 0,10	10,0
0,10 to 0,50	5,0
0,50 to 1,0	1,0
1,0 to 2,0	0,50
2,0 to 5,0	0,25

6.2 Determination

Take three 25 ml measuring cylinders (4.1) (A, B, C).

Into cylinders A and B, pipette 2 ml aliquot portions of the test solution (6.1). To A add 1 ml of the standard alcohol solution (3.4).

Make up the volume of all three cylinders to 5 ml with the toluene (3.2). Add 10 ml of the reagent (3.5) to each cylinder, stopper, shake and heat on the water bath (4.3), controlled at 60 ± 1 °C for 20 min.

Allow to cool, and add 10 ml of the sodium hydroxide solution (3.3) to each cylinder; shake for 2 min, or until all the black colour has disappeared.

Allow the phases to separate then pipette 5,0 ml of the upper toluene phase from each 25 ml cylinder into the corresponding 10 ml measuring cylinders (4.1). Add 1 ml of the dichloroacetic acid solution (3.1) to each and mix.

Measure the absorbances of the solutions contained in the measuring cylinders A and B at a wavelength of about 620 nm in cells of 1 cm optical path length, using the spectrometer (4.2) after having adjusted it to zero absorbance against the solution contained in the final measuring cylinder C (compensation solution).

7 Expression of results

The free alcohols content, expressed as a percentage by mass as the corresponding alcohol, is given by the formula

$$\frac{m}{1\,000} \times \frac{A_B}{(A_A - A_B)} \times \frac{25}{2} \times \frac{100}{m_0}$$

$$= 1{,}25 \times \frac{A_B}{(A_A - A_B)} \times \frac{m}{m_0}$$

where

A_A is the absorbance of the solution in cylinder A;

A_B is the absorbance of the solution in cylinder B;

m_0 is the mass, in grams, of the test portion (6.1);

m is the mass, in milligrams, of the alcohol corresponding to the alcohol to be determined contained in the volume of the standard solution (3.4), taken in 6.2.

8 Test report

The test report shall include 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 this International Standard or regarded as optional.

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