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THE DETERMINATION OF THE APPEARANCE, COLOR, DENSITY OF ETHYL ACETATE OBTAINED ON THE BASIS OF EAF

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Abstract:
Methods have been developed for the production of ethyl acetate, with the continuous interaction of acetic acid with ethyl alcohol in the presence of heterogeneous acid catalysts with simultaneous distillation of the resulting ethyl acetate at a temperature of 75-90 ° C. Ethyl acetate in appearance and color, density, mass fraction of the main substance, mass fraction of acids in terms of acetic acid, mass fraction of water corresponded to SS 8981-78.

Keywords: Ethyl acetate, preparation of ethyl acetate from the ether-aldehyde fraction, ethanol, ethanol dehydrogenation catalysts.

Ethyl acetate is widely used as a solvent, due to its low cost and low toxicity, as well as acceptable odor. In particular, as a solvent of cellulose nitrates, acetylcellulose, fats, waxes, for cleaning printed circuit boards, mixed with alcohol - a solvent in the production of artificial leather.

As a solvent, being an active solvent of nitro- and ethyl cellulose, it is widely used in the production of paints and inks for printing machines. It is also included in the composition of solvents of nitroglyptal, perchlorovinyl and epoxy enamels, various lubricating oils, waxes, polyester lacquers, paints, organosilicon lacquers and enamels. For these purposes, up to 30% of the total ethyl acetate produced is consumed.

At the stage of packaging various goods with flexible packaging materials - as a solvent for films and inks when applying inscriptions and images by stencil method. As a reagent and as a reaction medium in the production of pharmaceuticals (metoxazole, hydrocortisone, rifampicin, etc.).

One of the most popular poisons used in entomological stains for killing insects. Insects after killing in its vapors are much softer and more pliable in dissection than after killing in chloroform vapors.

- Ethyl acetate can be used as a solvent for resin oil paints, waxes, fats, cellulose esters. The substance can also be used in the following cases:
- Dissolution of film-forming substances, pigments in the production of lacquers and paints, inks for printing machines.
- Manufacture of multicomponent adhesives.
- Dissolution of ink when applying images and inscriptions by stenciling on packaging materials.
- Creation of a reaction environment in the production of medicines.
- Cleaning and degreasing of surfaces in the electronics industry.
- Extraction of organic components from aqueous solutions (for example, caffeine from coffee).
- Gelatinization of explosives.
- Production of fruit essences, etc.

A number of works are devoted to synthetic methods of obtaining esters, methods of their purification and separation, as well as to the study of their physico-chemical properties [1,2].

We studied the production of ethyl acetate based on acetic acid and the etheraldehyde fraction, and also studied the appearance, color, density [3, 4, 5].

Research methods. The sample of the analyzed ethyl acetate is mixed, 50 cm 3 is poured into a clean dry cylinder made of transparent colorless glass and the appearance is determined in transmitted light.
Chromaticity is determined according to SS 18522-73 by visual method.

The definition is allowed to be compared with the fringe scale prepared according to SS 14871-76. The chromaticity of the analyzed product should not exceed the chromaticity of the comparison solution with a concentration of 2 mg of potassium bicarbonate in 1 dm3 of the solution obtained by diluting the basic solution 500 times.

In case of disagreement in the assessment of the chromaticity of the analyzed product, the determination is carried out according to SS 18522-73.

The density at 20 ° C is determined according to SS 18995.1-73, sec.1. It is allowed to determine the density at the temperature of the analyzed product (20

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± 5 ° C), while the average temperature correction of density (a) by 1 ° C for ethyl acetate is 0.001 g /cm. A glass laboratory thermometer with a division price of 0.1 ° C is used for analysis.

The determination of the mass fraction of the basic substance.

The mass fraction of the basic substance is determined according to SS 21533-76.

The mass fraction of the basic substance is allowed to be determined by the saponification method. All operations are carried out at room temperature.

The 5 cm 3 of alcohol is added to a dry flask and weighed with an error of not more than 0.0002 g, about 1.5 g (1.7 cm3) of the analyzed ethyl acetate is added with a pipette and again weighed with the same error.

Add 2-3 drops of phenolphthalein solution to the flask and use a burette of 25 cm3 of alkali solution. The flask is closed with a stopper, the contents of the flask are carefully but thoroughly re-soaked by gentle rotation until a homogeneous solution is obtained. After stirring, the flask with the contents is left at rest for 10 minutes. The excess alkali is titrated with a sulfuric acid solution.

At the same time, a control experiment is carried out with the same quantities of reagents, but without the analyzed product.

The mass fraction of the main substance, in terms of ethyl acetate (X), in percent, is calculated by the formula:

$$X = \frac{(У_1 - У)м^1 \cdot 100}{м},$$

where Y - the volume of a solution of sulfuric acid with a concentration of exactly 1 mol / dm3, consumed for titration in the main experiment, cm3.

U1 - the volume of a solution of sulfuric acid with a concentration of exactly 1 mol / dm3, consumed for titration in the control experiment, cm3.

M 1 is the mass of ethyl acetate (0.0881), corresponding to 1 cm3 of a sulfuric acid solution with a concentration of exactly 1 mol dm3, g.

m - sample of the analyzed product, g.

The arithmetic mean of two parallel determinations is taken as the result of the analysis, the allowable discrepancies between which at a confidence level of P = 0.95 should not exceed 0.5%.

The admissible discrepancies between the arithmetic mean values of parallel determinations during interlaboratory control should not exceed 0.5%.

In case of disagreement in the assessment of the mass fraction of the main substance, the analysis is carried out in accordance with GOST 21533-76.

Determination of the mass fraction of acids in terms of acetic acid. Pour 25 cm3 of alcohol and 25 cm3 of ethyl acetate into a flask and quickly titrate with an alkali solution in the presence of phenolphthalein until a slightly pink color of the solution appears, which does not disappear within 5 s.

The mass fraction of acids in terms of acetic acid (X1) in percent is calculated by the formula:

$$X_1 = \frac{У \cdot 0{,}0006 \cdot 100}{25 \cdot p^1}$$

where Y - the volume of an alkali solution of concentration, exactly 0.01 mol / dm3, consumed for titration, cm3.

0.0006 - mass of alkali solution with concentration, exactly 0.01 mol / dm3;

25 - the volume of the analyzed product taken for analysis, cm3;

p1 is the density of the analyzed product at the temperature of the analysis; g / cm3

The arithmetic mean of two parallel determinations is taken as the result of the analysis, the allowable discrepancies between which at a confidence level of P = 0.95 should not exceed 0.0006%.

The admissible discrepancies between the arithmetic mean values of parallel determinations during interlaboratory control should not exceed 0.001%.

Determination of the mass fraction of the non-volatile residue. In a cup, weighed with an error of not more than 0.0002 g and brought to constant weight at (110 ± 2) ° C, pour 50 cm3 of ethyl acetate in two portions, the contents of the cup are evaporated under an infrared lamp, placing it at a distance of 20 cm from a radiating surface or in a water bath. The cup with the remainder is placed in an oven and kept at (110 ± 2) °C for 30 minutes, cooled with the same error.

The mass fraction of the acids of the non-volatile residue (X2) in percent is calculated by the formula:

$$X_2 = \frac{м \cdot 100}{У \cdot p^1}$$

where m - the mass of the residue after drying, g;

Y - the volume of the analyzed product taken for analysis, cm3;

p1 - density of the analyzed product at the temperature of analysis, g / cm3

The arithmetic mean of two parallel determinations is taken as the result of the analysis, the allowable discrepancies between which at a confidence level of P = 0.95 should not exceed.

0.0002% - for the mass fraction of non-volatile residue up to 0.001% inclusive.

0.007 - for the mass fraction of non-volatile residue over 0.001 to 0.01%.

The admissible discrepancies between the arithmetic

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mean values of parallel determinations during interlaboratory control should not exceed 0.001%.

Table 1 The technical characteristics parameters of ethyl acetate according to SS 8981-78.

Index	Grade A		Grade B
	Top grade	First grade	
Appearance	Transparent liquid without mechanical impurities		
The density of ethyl acetate, g/cm^3	0,898–0,900	0,897–0,900	0,890–0,900
Chromaticity, Hazen units	5	10	10
The mass fraction of acids in terms of acetic acid,%	0,004	0,008	0,01
The mass fraction of the main substance,%	от 99	от 98	91 ± 1
The mass fraction of non-volatile residue,%	0,001	0,003	0,007
The mass fraction of water,%	0,1	0,2	1
The mass fraction of aldehydes in terms of acetic acid,%	0,05	Is not marked	

The determination of the mass fraction of aldehydes in terms of acetaldehyde.

25 ml of water and 5 ml of hydroxyl amine hydrochloric acid solution, 5 ml of the analyzed product and 2-3 drops of methyl orange are poured into the flask. The flask is tightly closed with a stopper, the contents are thoroughly mixed and left until an orange-pink color appears.

The mass fraction of aldehydes in terms of acetaldehyde (X3) in percent is calculated by the formula:

$$X_3 = \frac{У \cdot 0{,}00044 \cdot 100}{5 \cdot p^1}$$

where У - the volume of an alkali solution with a concentration of exactly 0.01 mol / dm3, consumed for titration, cm3.

0.00044 is the mass of acetaldehyde corresponding to 1 ml of an alkali solution with a concentration of exactly 0.01 mol / dm3 / g;

5 - the volume of the analyzed product taken for analysis, cm3;

p1 - density of the analyzed product at the temperature of analysis, g / cm3

The arithmetic mean of two parallel determinations is taken as the result of the analysis, the allowable discrepancies between which at a confidence level of P = 0.95 should not exceed 0.005%.

Conclusions. Methods for the production of ethyl acetate based on the ether-aldehyde fraction with a high yield have been developed. Compliant with the standards of GOST 8981-78 grades A and B (table 1).

The chromaticity of ethyl acetate was studied according to SS 18522-73, the density at 20 ° C was determined according to SS 18995.1-73.

The mass fraction of the main substance was determined according to SS 21533-76, the mass fraction of the non-volatile residue, the mass fraction of aldehydes in terms of acetaldehyde.

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