26.1.31 - Distilled Liquors / Spirits

AOAC Official Method 972.10 Alcohols (Higher) and Ethyl Acetate in Distilled Liquors

Alternative Gas Chromatographic Method
First Action 1972
Final Action 1975

A. Apparatus

Gas chromatograph.—Equipped with flame ionization detector. (*1*) *Column*.—2% glycerol and 2% 1,2,6-hexanetriol. Pack 3 m (10 ft) 3 mm (in.) od tube. Condition overnight in 80°C column oven with He flow rate of 10-25 mL/min and detector end of column disconnected.

(*2*) *Approximate parameters*.—Column, injector, and detector temperatures (°C)—80, 100, and 125, respectively; gas flows (mL/min)—He carrier and H 25, air 250-400; attenuation 64 .

Optimum operating conditions vary with column and instrument and must be determined by using standard solutions. Adjust parameters for maximum peak sharpness and optimum separation. Analysis is complete in ca 11 min.

B. Reagents

(Use absolute alcohol throughout when alcohol is specified.)

(**a**) *n-Propyl alcohol.*

(**b**) *Isobutyl alcohol*.—(Fisher Scientific Co., Certified Reagent No. A-379.)

(**c**) *Amyl alcohol*.—(Gallard-Schlesinger Industries, Inc., 584 Mineola Ave, Carle Place, NY 11514-1731, USA, AnalaR No. 10038.) Mixture of active-amyl and isoamyl alcohols, ca 22 and 78%, respectively. Concentration of 2 isomers varies from batch to batch. Determine composition of reagent by **C**. Measure areas of 2

peaks by triangulation (height width at half-height), and obtain concentration of each by dividing area of each peak by sum of both peak areas.

(**d**) *3-Pentanol internal standard solution*.—40.76 mg/mL (Aldrich Chemical Co., Inc., No. P802-5). Prepare solution containing 10 mL reagent in 200 mL alcohol-H_2O (1 + 1).

(**e**) *Ethyl acetate*.—(Fisher Scientific Co., Certified Reagent No. E-145.)

(**f**) *n-Propyl alcohol, isobutyl alcohol, and amyl alcohol standard solutions*.—Prepare 3 or 4 standard solutions containing varying amounts alcohols as follows: Into tared 100 mL volumetric flasks containing alcohol-H_2O (1 + 1), pipet fusel alcohols and weigh after addition of each component. Proportions of fusel alcohols in each standard solution should vary so that desired concentration range of each is represented in random manner in series of standard solutions. Suggested amounts: 0.25-1.5 mL *n*-propanol, 1.0-2.5 mL isobutyl alcohol, and 2.0-5.0 mL amyl alcohol. Dilute each to volume with alcohol-H_2O (1 + 1).

(**g**) *n-Propyl alcohol, isobutyl alcohol, and amyl alcohol working standard solutions*.—Dilute 10 mL each standard solution and 2.0 mL 3-pentanol internal standard solution to 200 mL with alcohol-H_2O (1 + 1) (1:20 dilution).

(**h**) *Ethyl acetate standard solutions*.—Prepare 3 or 4 standard solutions containing 0-0.5 g/L (0-50 g/100 L) in H_2O or alcohol-H_2O (1 + 1). Use for preparing direct standard curve by plotting peak height (mm) against concentration in g/100 L.

C. Determination

Pipet 10 mL test portion into convenient vessel (e.g., 1 oz French square glass bottle with screw cap), add, by pipet (0.2 mL pipet graduated in 0.01 mL), 0.1 mL 3-pentanol internal standard solution, and mix. Inject 2 µL test portion and working standard solutions. Measure peak height of each component in working standard solution and calculate peak height ratio of each to internal standard. Calculate concentration ratio of each by dividing weight of component by that of internal standard. (Proportion of active-amyl and isoamyl alcohols in mixture must be taken into consideration in calculations of actual weights of each isomer in working standard solutions.)

Plot concentration ratios (horizontal axis) against peak height ratios (vertical axis) for each higher alcohol in all working standards to obtain family of curves. For ethyl acetate, plot peak height directly against concentration.

Similarly, measure peak height of each component on test portion chromatogram and calculate peak height ratios. Read concentration ratios of all alcohols, using proper standard curve. Multiply concentration ratio of each fusel alcohol in test portion by 40.76 to obtain g/100 L. New standard curves need be prepared only when new instruments, parameters, or standards are used.

Reference:
JAOAC **55,** 549(1972).

CAS-71-41-0 (amyl alcohol)

CAS-141-78-6 (ethyl acetate)

CAS-78-83-1 (isobutyl alcohol)

CAS-71-23-8 (*n*-propyl alcohol)