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**26.1.30 - Distilled Liquors / Spirits**

**AOAC Official Method 968.09**  
**Alcohols (Higher) and Ethyl Acetate**  
**in Distilled Liquors**

**Gas Chromatographic Method**  
**First Action 1968**  
**Final Action 1969**

***A. Apparatus***

(a) *Gas chromatograph*.—Equipped with flame ionization detector. (1) *Column*.—23% Carbowax 1500 (w/w) on Chromosorb W (60-80 mesh, acid-washed).

(2) *Approximate parameters*.—Column temperature 70°C (isothermal); detector and inlet temperature 150°C; He carrier flow 150 mL/min.

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. With high level standard, *n*-Propanol should give almost complete baseline separation from ethanol.

(b) *Syringe*.—10 µL, Hamilton Co. No. 701, or equivalent.

***B. Reagents***

(a) *Isobutyl alcohol*.

(b) *Isoamyl alcohol*.

(c) *n-Propyl alcohol*.

(d) *Ethyl acetate*.

(e) *n-Butyl alcohol*.

(f) *n*-Butyl alcohol internal standard solutions.—(1) *High level*.—Dilute 10 mL *n*-butanol to 100 mL with 40% alcohol. (1 mL added to 100 mL test portion or standard is equivalent to ca 81 g *n*-butanol/100 L.) (2) *Low level*.—Dilute 1 mL *n*-butanol to 200 mL with 95% alcohol. (1 mL added to 100 mL test portion or standard is equivalent to ca 4.1 g/100 L.)

(g) *n*-Propyl alcohol, isobutyl alcohol, isoamyl alcohol, and ethyl acetate high level standard solutions.—(1) *Stock solution*.—Accurately weigh 1 mL *n*-propanol, 1 mL isobutyl alcohol, 2 mL isoamyl alcohol, and 1 mL ethyl acetate into 100 mL volumetric flask and dilute to volume with 40% alcohol. (2) *Intermediate solution*.—Dilute 10 mL stock solution to 200 mL with 40% alcohol. (3) *Working solution*.—(Approximately 40.2, 41.1, 81.2, and 45.1 g/100 L *n*-Propanol, isobutyl alcohol, isoamyl alcohol, and ethyl acetate, respectively.) Dilute 5 mL stock solution to 100 mL with 40% alcohol. Add 1 mL high level *n*-butanol internal standard solution, (f)(1), and mix. Prepare fresh weekly.

(h) *n*-Propyl alcohol, isobutyl alcohol, isoamyl alcohol, and ethyl acetate low level working solutions.—(Approximately 2.0, 2.1, 4.1, and 2.3 g/100 L *n*-propanol, isobutyl alcohol, isoamyl alcohol, and ethyl acetate, respectively.) Dilute 5 mL high level intermediate solution, (g)(2), to 100 mL with 95% alcohol. Add 1 mL low level internal standard solution, (f)(2), and mix. Prepare fresh weekly.

Prepare standard solution of ca same concentration as test solution if latter differs grossly from appropriate (high or low level) standard.

### **C. Determination**

Make preliminary injection of 10 µL test portion to determine absence of *n*-butanol. (If present, subtract its amount from total *n*-butanol [original and internal standard] content.) Add 1 mL internal standard solution, B(f) (high or low level, depending on higher alcohol and ethyl acetate concentration), to 100 mL test portion in volumetric flask, and chromatograph 10 µL aliquots of test portion and standard solutions in triplicate.

Measure peak heights of *n*-propanol, isobutyl alcohol, isoamyl alcohol, and ethyl acetate to nearest 0.05 cm and calculate peak height ratio of each to *n*-butanol (internal standard) in test and standard solutions. (For more accurate determination of isoamyl alcohol, use peak areas.)

$X = H \ S/H$ , where  $X$  = concentration of higher alcohol or ethyl acetate in test portion (g/100 L);  $H$  = peak height (or area for isoamyl alcohol) ratio of higher alcohol or ethyl acetate to *n*-butanol in test portion;  $H$  = peak height (or area for

isoamyl alcohol) ratio of higher alcohol or ethyl acetate to *n*-butanol in standard; *S* = concentration of higher alcohol or ethyl acetate in standard (g/100 L).

Sum of isoamyl and isobutyl alcohol concentrates is ca equivalent to fusel oil concentration as determined in [963.10](#) (*see* 26.1.29).

**Reference:**

*JAOAC* **51**, 915(1968).

CAS-141-78-6 (ethyl acetate)

CAS-78-83-1 (isobutyl alcohol)

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

CAS-71-36-3 (*n*-butyl alcohol)