AOAC Official Method 972.11
Methanol in Distilled Liquors

Gas Chromatographic Method
First Action 1972
Final Action 1973

A. *Apparatus*

See **968.09**A (*see* 26.1.30).

B. *Reagents*

(**a**) *Alcohol*.—Methanol-free.

(**b**) *Methanol stock solution*.—Dilute 10 mL methanol, 99.9 mol % (Fisher Scientific Co., A-936, or equivalent) to 100 mL with 40% alcohol.

(**c**) *n-Butyl alcohol internal standard stock solution*.—Dilute 10 mL *n*-butanol, 99.9 mol % (Fisher Scientific Co., A-384, or equivalent) to 100 mL with 40% alcohol.

(**d**) *Methanol standard solution*.—0.050% methanol plus 0.030% *n*-butanol internal standard. Fill 100 mL volumetric flask to ca 99 mL with 40% alcohol and add, by syringe, 500 µL mixture stock solution, (**b**), and 300 µL *n*-butanol stock solution, (**c**). Mix and dilute to volume with 40% alcohol. Mix again.

C. *Determination*

Inject 10 µL mixture standard solution. Adjust operating parameters and attenuation to obtain measurable peak height (ca   full scale deflection). Determine retention time of methanol and *n*-butanol (ca 3 and 12 min, respectively). Inject 10 µL test portion to estimate methanol, using attenuation if necessary, and to check for absence of *n*-butanol. On basis of presence or absence of *n*-butanol in test portion, determine methanol content from standard curve prepared according to (**a**) or (**b**):

(**a**) *n-Butyl alcohol absent*.—On basis of estimate of methanol, prepare series of standards (4 or 5) in which range of concentration includes methanol concentration in test portion. Add internal standard to both test portion and standard solutions at concentration similar to that of methanol in test portion. Calculate peak height ratios of methanol:*n*-butanol, using average of duplicate injections, and plot ratios against methanol concentration.

(**b**) *n-Butyl alcohol present*.—Prepare series of methanol standards as in (**a**), but do *not* add *n*-butanol to test portion or standards. Plot actual peak height of methanol against concentration.

Reference:
JAOAC **55,** 564(1972).

CAS-67-56-1 (methanol)