

8.1.02

AOAC Official Method 975.06
Denaturants (Volatile) in Alcoholic Products
Gas Chromatographic Method
First Action 1975

A. Apparatus and Reagents

(a) *Gas chromatograph and integrator.*—With flame ionization detector (Agilent Technologies, Division of Hewlett Packard, 2850 Centerville Rd, Wilmington, DE 19808, USA; www.chem.agilent.com; 6890 series or equivalent). Column 1.2 m (4 ft) 2 mm id glass packed with 100–200 mesh Chromosorb 102; column temperature 160°C, detector and inlet 200°C; He flow rate 50 mL/min; relative retention times: ethyl alcohol 1.00 (100 s), *n*-propyl alcohol 2.06, and tetrahydrofuran 3.04. Integrator: Hewlett-Packard 3396 or equivalent. Microsyringe: Hamilton Model 7001 or equivalent.

(b) *Standard solutions.*—6% (v/v). Dilute 6.00 mL of each denaturant of interest to 100 mL with anhydrous alcohol in separate volumetric flasks. Approximate slopes and retention times relative to *n*-propyl alcohol are given in Table 975.06.

B. Determination

Pipet 25 mL of each expected denaturant standard solution into separate flasks and add 1.00 mL *n*-propyl alcohol as internal standard. Cap immediately with rubber stoppers, shake 3 min, and let stand 10 min at room temperature. Inject 0.3 L aliquots from 1 L microsyringe. Determine peak areas and calculate slope for each compound as:

$$S_x = \frac{PA_x / PA_i}{6.00}$$

Table 975.06. Approximate slopes and retention times (RT) relative to *n*-propyl alcohol for denaturants

Compound	Slope	RT
Acetone CAS-67-64-1	0.207	0.694
Benzene CAS-71-43-2	0.646	2.309
<i>n</i> -Butyl alcohol CAS-71-36-3	0.269	2.283
<i>sec</i> -Butyl alcohol CAS-78-92-2	0.246	1.621
Chloroform CAS-67-66-3	0.058	1.543
Ethyl acetate CAS-141-78-6	0.192	1.640
Ethylene glycol monoethyl ether CAS-629-14-1	0.187	3.868
Ethylene glycol monomethyl ether CAS-109-86-4	0.151	2.071
Isopropanol CAS-67-63-0	0.210	0.727
Methanol CAS-67-56-1	0.130	0.266
Methyl isobutyl ketone CAS-108-10-1	0.275	5.436
Toluene CAS-108-88-3	0.454	5.302

where PA_x and PA_i = peak areas of compound X in standard solution and of *n*-propyl alcohol internal standard, respectively, and 6.00 = % compound X in standard solution. Slopes and retention times should approximate those listed in Table 975.06.

$$\% \text{ Compound X in test sample} = (PA/PA_i) = (1/S_x)$$

where PA = peak area of compound X in test sample.

Reference: *JAOAC* 57, 148(1974).