

8.1.05

AOAC Official Method 971.03 Methanol in Hazardous Substances

Gas Chromatographic Method

First Action 1971

Final Action 1973

(Applicable in presence of acetone, butyl acetate, ethyl alcohol, isopropanol, hexane, methyl ethyl ketone, methylene chloride, 2-methoxyethanol, paraffin, toluene, and H₂O, including many paint removers, fuels, liquid sanders, antifreezes, and paint products.)

A. Apparatus and Reagents

(a) *Gas chromatograph*.—With flame ionization detector, temperature programmable oven, and data system. Column: 1.8 m 4 mm id packed with 120–150 mesh Porapak R (Waters Associates, Inc., PO Box 246, Milford, MA 01757, USA); condition column for 2 h at 235°C. Conditions: temperatures—oven profile: 160°C for 8 min, 160 to 235°C at 5°C/min, 235°C for 10 min (or 2 min greater than dioxane retention time), injector 200°C, detector 210°C; nitrogen carrier flow ca 25 mL/min; set electrometer so that 8 L standard solution response is at least half of full scale peak. Adjust initial column temperature (and if necessary carrier flow) so that methanol retention time is 5–7 min.

(b) *Methanol standard solution*.—0.4% (v/v). Dilute 4.00 mL methanol to 100 mL with dioxane; dilute 10.0 mL of this solution to 100 mL with dioxane. Rinse pipet (TC pipet) into flask with dioxane before diluting to volume with dioxane. Prepare fresh daily. (*Caution*: Dioxane is highly toxic and a possible carcinogen. Use extreme caution when handling to avoid exposure.)

B. Preparation of Test Material

(a) *For asphalt-base compounds and viscous adhesives*.—Refrigerate unopened test sample container 3 h (longer for larger containers) at 1–10°C, open container, and mix well; close container and refrigerate 30 min more. Transfer 1.5–3 g

test portion to 250 mL, wide-mouth glass-stoppered Erlenmeyer flask (tared with stopper in place). Let test portion reach room temperature in stoppered Erlenmeyer and weigh. Refrigerate 30 min and quickly add 100.0 mL dioxane. Stopper and shake mechanically 1 h. Refrigerate 30 min and filter through rapid paper (S&S sharkskin, or equivalent). Filter as quickly as possible, covering funnel with watch glass and placing funnel against neck of narrow-mouth glass-stoppered receiver. Dilute further with dioxane if necessary for determination.

(b) *For less viscous products*.—Prepare solution containing ca 0.4% (v/v) methanol, diluting with dioxane. Avoid excessive shaking of semiviscous products and do not fill pipet above mark. Wash "to contain" (TC) pipet with dioxane and add washings to solution.

If approximate methanol concentration in test material is unknown, prepare a 2% solution. Perform additional dilutions as needed.

C. Determination

Separately inject portions of standard and test solutions using 10 L syringe, noting volumes. (Presence of other solvents in test material may result in changes in retention times.)

D. Calculation

Average peak areas for test portion (A) and for standard (A_s).

$$\text{Percent methanol (w/v) in test solution} \\ = F \left(\frac{A}{A_s} \right) \left(\frac{V_s}{V} \right) C_s \cdot 0.79$$

where F = dilution factor, C_s = % (v/v) standard solution, V and V_s = volume of test and standard solution injected, respectively, and 0.79 = density of methanol.

References: *JAOAC* **54**, 558(1971); **55**, 242(1972).

CAS-67-56-1 (methanol)