

### 3.7.08

#### AOAC Official Method 979.01 Nicotine on Cambridge Filter Pads

##### Gas Chromatographic Method

First Action 1979

Final Action 1984

#### A. Apparatus and Reagents

(a) *Gas chromatograph*.—With flame ionization detector, heated injection port, and thermostated column oven. Following conditions have been found satisfactory: column, 1.8 m (6)  $\frac{1}{8}$  in. (3.175 mm) stainless steel; packing, 2% KOH and 10% Carbowax 20M (based on final packing weight) on 45–60 mesh calcined diatomaceous earth (such as Chromosorb W, or equivalent), resieved before use to mesh range to remove fines and lumps; temperatures (°C): column 165, detector and injection port 200–250; carrier gas flow, ca 40 mL/min. Adjust H<sub>2</sub> and air flows for maximum sensitivity and stability. Under these conditions, column should have height equivalent to theoretical plate (HETP) <1 mm and resolution of >2, calculated with nicotine and anethole.

(b) *Measuring system*.—Measure peak areas with electronic integrator or other system with resolution of 1 count/mV-s.

(c) *Mechanical shaker*.—Extracting 99% nicotine. Burrell wrist action shaker has been found satisfactory.

(d) *Extracting solution*.—2-Propanol containing 1 mg anethole/mL as internal standard for nicotine. If H<sub>2</sub>O is also to be determined, add 20 mg ethyl alcohol/mL 2-propanol as additional internal standard.

(e) *Nicotine standard solutions*.—(1) *Stock solution*.—Weigh 2.500 g nicotine, 960.07C (see 3.7.06), or equivalent amount of nicotine salt. Transfer quantitatively into 100 mL volumetric flask,

and dilute to volume with extracting solution. (2) *Working standard solutions*.—Pipet 1, 2, 3, 4, and 5 mL stock solution into five 100 mL volumetric flasks, and dilute to volume with extracting solution (0.25, 0.50, 0.75, 1.00, and 1.25 mg nicotine/mL). [Caution: See precaution in 960.07C (see 3.7.06).]

#### B. Extraction

Place Cambridge filter material containing absorbed nicotine in flask or serum bottle accommodated by shaker used, add 10.00 mL extracting solution, stopper, and shake until 99% of nicotine is extracted (usually ca 15 min).

#### C. Standardization

Prime column with aliquots of 1.25 mg/mL standard solution. Let baseline stabilize, inject 1  $\mu$ L each standard solution in succession, and repeat sequence 3 times. Determine area ratio (nicotine:anethole) for each injection, and calculate slope and intercept of response curve, preferably by method of least squares [See *Definition of Terms and Explanatory Notes* No. (26)]. Intercept 0.05 mg/mL.

#### D. Determination

Prime column with aliquots of extract, B. Let baseline stabilize, and inject 1  $\mu$ L of each test solution. Calculate nicotine concentration in solution ( $C$ , mg/mL) =  $mx + b$ , where  $m$  = slope of standardization curve,  $b$  = intercept, and  $x$  = area ratio of nicotine to anethole.

$$\text{Nicotine yield/cigarette} = \frac{C \ 10.00}{\text{No. cigarettes / pad}}$$

Reference: *JAOAC* 62, 229(1979).

CAS-55-11-5 (nicotine)