Designation: D 5075 – 01 (Reapproved 2007)

Standard Test Method for Nicotine and 3-Ethenylpyridine in Indoor Air[1]

This standard is issued under the fixed designation D 5075; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the sampling/analysis of nicotine and 3-ethenylpyridine (3-EP) in indoor air. This test method is based upon the collection of nicotine and 3-EP by adsorption on a sorbent resin, extraction of nicotine and 3-EP from the sorbent resin, and determination by gas chromatography (GC) with nitrogen selective detection. (**1**)[2]

1.2 The active samplers consist of an XAD-4 sorbent tube attached to a sampling pump. This test method is applicable to personal or area sampling.

1.3 This test method is limited in sample duration by the capacity of the XAD-4 tube for nicotine (about 300 µg). This test method has been evaluated up to 24-h sample duration; however, samples are typically acquired for *at least* 1 h (sometimes *only* 1 h). (**2**)

1.4 For this test method, limits of detection (LOD) and quantitation (LOQ) for nicotine at a sampling rate of 1.5 L/min are, respectively, 0.11 µg/m^3 and 0.37 µg/m^3 for 1-h sample duration and 0.01 µg/m^3 and 0.05 µg/m^3 for 8-h sample duration. The LOD and LOQ for 3-EP at a sampling rate of 1.5 L/min are, respectively, 0.06 µg/m^3 and 0.19 µg/m^3 for 1-h sample duration and 0.01 µg/m^3 and 0.02 µg/m^3 for 8-h sample duration (**2**). Both LOD and LOQ can be reduced by increasing the sensitivity of the thermionic specific detector.

1.5 The values stated in SI units are to be regarded as standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary information is given in 13.6.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere

D 3631 Test Methods for Measuring Surface Atmospheric Pressure

D 5337 Practice for Flow Rate Calibration of Personal Sampling Pumps

E 260 Practice for Packed Column Gas Chromatography

E 355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1356 and Practice E 355.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *environmental tobacco smoke (ETS)*—an aged, dilute composite of exhaled tobacco smoke and smoke from tobacco products.

3.2.2 *nitrogen-phosphorus detector (NPD)*—a highly sensitive device selective for detection of nitrogen- and phosphorus-containing organic compounds.

3.2.3 *XAD-4 resin*—macroreticular polystyrene-divinylbenzene copolymer beads.

4. Summary of Test Method

4.1 A known volume of air is drawn through a sorbent sampling tube containing XAD-4 resin to adsorb the nicotine and 3-EP present.

4.2 The XAD-4 sorbent tube contents are transferred to a 2-mL autosampler vial, and the nicotine and 3-EP are desorbed with ethyl acetate containing 0.01 % triethylamine and a known quantity of quinoline, the internal standard.

4.3 An aliquot of the desorbed sample is injected into a gas chromatograph equipped with a thermionic-specific (nitrogen-phosphorus) detector.

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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[2] The boldface numbers in parentheses refer to a list of references at the end of the text.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4.4 The areas of the resulting nicotine and 3-EP peaks are each divided by the area of the internal standard peak and compared with area ratios obtained from the injection of standards.

5. Significance and Use

5.1 In order to estimate ETS concentrations, there needs to be a marker or tracer for ETS that is unique or highly specific to tobacco smoke, in sufficient concentrations in air to be measured easily at realistic smoking rates, and in constant proportion to the other components of ETS for a variety of tobacco blends and environmental conditions. Nicotine and 3-ethenylpyridine have been used as tracers of the vapor phase of ETS. Nicotine is the major alkaloid of tobacco and a major constituent of ETS. The determination of nicotine concentration has often been used to estimate the concentration of ETS; however, due to its unpredictable decay kinetics, nicotine may not be an ideal tracer. Because nicotine readily adsorbs to building materials and room furnishings and is depleted from ETS at a rate faster than most other components, some have suggested that nicotine concentrations underestimate ETS concentrations. Although this is true in many environments during the generation of smoke, the converse is true in environments with a recent past history of smoking. The adsorbed nicotine slowly desorbs over time, resulting in an overestimation of ETS concentrations. Thus, measured concentrations of nicotine precisely assess only airborne nicotine and indicate only that smoking has taken place; they do not necessarily indicate the presence, and certainly not the concentrations, of other ETS constituents. 3-Ethenylpyridine, on the other hand, has been shown to track exactly the vapor phase of ETS as measured by CO and FID response (**3**). It is for these reasons that 3-ethenylpyridine may be a better tracer of ETS (**1**,**4**,**5**). The ETS at high concentrations is known to be annoying and irritating to individuals, and concerns over potential health effects have also been expressed. There is a definite need to have reliable methods for the estimation of ETS levels in order to evaluate its effect. The NIOSH has previously set a threshold limit value (TLV) for nicotine in the workplace of 0.5 mg/m^3.

5.2 Studies show that more than 90 % of nicotine in indoor air is found in the vapor phase (**6**,**7**). The described test method collects vapor-phase nicotine quantitatively. Early studies on freshly generated ETS indicated that some but not all of the particulate phase was trapped on the XAD-4 resin (**7**). A more recent investigation of the trapping of particulate materials by sorbent beds suggests that the trapping of the particles from indoor air may be nearly quantitative (**8**). 3-Ethenylpyridine is found exclusively in the vapor phase.

5.3 Nicotine concentrations typically range from ND (not detected) to 70 µg/m^3 in various indoor environments with values usually at the lower end of this range (**9**). Because such low concentrations of nicotine are often encountered, sophisticated analytical procedures and equipment are required for quantifying nicotine in indoor air. Other methods for the determination of nicotine in indoor air have also been reported (**6**,**10**,**11**,**12**). 3-Ethenylpyridine concentrations typically are about one third the concentrations of nicotine in real-world environments (**13**).

6. Interferences

6.1 Use of packed GC columns may result in readings lower than expected because nicotine can adsorb onto undeactivated glass, metal, and solid support particles. Fused silica capillary columns and the modified extraction solvent prescribed here can circumvent this problem.

6.2 Quinoline (internal standard) is present in ETS at a concentration approximately 1 % of that for nicotine and is collected by the XAD-4 resin. If >10 µg nicotine is collected on the resin, there will be sufficient quinoline present to cause a detectable bias in results (approximately 1 %). (For example, this quantity of nicotine would be collected if a nicotine concentration of 167 µg/m^3 was sampled at 1 L/min for 1 h.) In these cases, one of the following alternative procedures should be followed:

6.2.1 Quantitatively dilute the sample with the same modified solvent containing internal standard (described in 11.2) used to extract the original sample; that is, decrease the amount of quinoline (and also nicotine) present in the sample while keeping the quinoline concentration in the solvent constant. To prevent significant interference, the nicotine concentration in the most concentrated sample should be less than or equal to the quinoline concentration in the solvent.

6.2.2 Use an alternate internal standard [N′-ethylnornicotine is recommended (**14**)].

7. Apparatus

7.1 *Sample Collection*:

7.1.1 *XAD-4 Sorbent Tube*—Glass tube with both ends flame-sealed, approximately 7 cm long with 6-mm outside diameter and 4-mm inside diameter, containing one section of 120 mg of 20/40 mesh XAD-4 resin. A glass wool plug is located at the front end (inlet) and back end of the tube. The glass wool plug at the inlet end of the tube is held in place with a metal lockspring.

7.1.2 *Tube Holder,* with clip attachment for attaching tube to clothing or objects.

7.1.3 *Tube Breaker,* to break sealed ends from sample tubes.

7.1.4 *NIOSH-approved Plastic Caps*, for capping tubes after sampling.

7.1.5 *Barometer and Thermometer*, for taking pressure and temperature readings at the sampling site (optional).

7.1.6 *Bubble Flowmeter,* for sample pump calibration.

7.1.7 *Personal Sampling Pump,* portable constant-flow sampling pump calibrated for the flow rate desired (up to 1.5 L/min).

7.2 *Analytical System*:

7.2.1 *Gas Chromatograph*, with a nitrogen-phosphorus (thermionic) detector and autosampler.

7.2.2 *GC Column*—A 30-m by 0.32-mm inside diameter fused silica capillary column, coated with a 1.0-µm film of 5 % phenyl methylpolysiloxane (DB-5).

7.2.3 *Chromatography Data Acquisition System,* for measuring peak areas electronically.

7.2.4 *Sample Containers*, borosilicate glass autosampler vials, 2-mL capacity, with PTFE-lined septum closures.

7.2.5 *Dispensing Pipets*, 1.25-mL.

7.2.6 *Triangular File*, for scoring and breaking open sample tubes.

7.2.7 *Forceps*, for assisting transfer of sorbent tube contents from tube to autosampler vial.

7.2.8 *Glass Wool Removal Tool,* for assisting transfer of sorbent tube contents from tube to autosampler vial.

7.2.9 *Wrist-action Shaking Device*, for solvent extraction.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Ethyl Acetate*, chromatographic quality.

8.3 *Quinoline (internal standard)*, 99+ %.

8.4 *Triethylamine*, 99+ %.

8.5 *Nicotine*, 99+ %.

8.6 *4-Ethenylpyridine (4-EP)*, 95 %, commercially available isomer of 3-ethenylpyridine.

8.7 *Helium Cylinders*, for carrier or detector makeup gas, or both, 99.995 % grade.

8.8 *Hydrogen Cylinders*, for detector gas, 99.995 % grade.

8.9 *Air*, for detector gas (<0.1 ppm hydrocarbon).

9. Sampling

9.1 *General*—For planning sampling programs, refer to Practice D 1357.

9.2 *Procedure*:

9.2.1 Prepare XAD-4 sampling tubes immediately before sampling. Break both ends of the sealed sorbent tube using a tube breaker tool. The opening should measure at least 2 mm in diameter.

9.2.2 Connect the sorbent tube to the personal sampling pump with tubing. Position the sorbent tube so that the air being sampled will pass first through the front section of resin and then through the backup section. The inlet end of the tube is exposed directly to the atmosphere, and the outlet end is inserted in the tubing; or the tube itself is put into a safety casing in the personal sampling setup and attached accordingly. Adjust the potentiometer on the sampling pump until the desired flow rate (≤1.5 L/min) is obtained. With the bubble flowmeter connected to the inlet end of the sorbent tube, measure and record the rate of airflow through the sorbent tube in litres per minute. Refer to Practice D 5337 for standard practice in calibrating personal sampling pumps.

9.2.3 After the XAD-4 sorbent tube is correctly inserted and positioned, turn on the power switch for the pump to begin sampling. Record the start time.

NOTE 1—Most pumps have microprocessing capabilities for preset sampling periods.

9.2.4 Record the barometric pressure and ambient temperature (optional).

9.2.5 Turn off the pump at the end of the desired sampling period, and record the elapsed time in minutes.

9.2.6 Measure and record the flow rate after sampling so that an average of initial and final flow rates can be used in subsequent calculations.

9.2.7 Remove the sorbent tube from the sampling system and place plastic caps over both ends of the tube.

9.2.8 Treat a minimum of two sorbent tubes in the same manner as the sample tubes (break, measure flows, cap, and transport). Label and process these tubes as flow blanks.

9.2.9 Transport capped sorbent tubes to the laboratory for analysis.

NOTE 2—If the samples are not prepared and analyzed immediately, they should be stored at 0°C or less. All sorbent tube samples should be analyzed within eight weeks after sample collection. It has been established that samples are stable for at least eight weeks at −10°C.

10. Analysis

10.1 *System Description*:

10.1.1 Analysis is performed using a GC fitted with a nitrogen-phosphorus detector and an autosampler equipped for split/splitless injection.

10.1.2 The GC column is as listed in 7.2.2.

10.1.3 The GC conditions are as listed in Table 1.

10.1.4 The autosampler uses default settings for the injection sequence, and 1 or 2 µL of sample is injected with a 30-s splitless period.

10.1.5 Peak areas are measured electronically with a chromatography data acquisition system.

10.2 *Systems Performance Criteria*:

10.2.1 Approximate retention times for 3-EP, 4-EP, quinoline, and nicotine are listed in Table 1.

10.2.2 Desorption efficiency should be determined for each new lot of sorbent tubes. Failure to determine the desorption efficiency and adjust results may impair the accuracy of the test.

10.2.3 Breakthrough (>5 % of tube contents found in backup resin section) can occur after collecting approximately 300 µg nicotine in a single XAD-4 tube. A shorter sampling time is necessary if sample concentration and duration of sampling suggest a breakthrough occurrence.

TABLE 1 Summary of Gas Chromatograph Conditions

Temperatures	
Injector	225°C
Oven	
Initial temperature	50°C
Hold time	1 min
Program Step 1	
Rate	10°C/min
Final temperature	215°C
Hold time	0 min
Program Step 2	
Rate	20°C/min
Final temperature	295°C
Hold time	1 min
Detector	300°C
Gas flows	
He, carrier	4 mL/min (15 psig)
H_2, detector	3 mL/min
Air, detector	75 mL/min
He, makeup	15 mL/min
Retention times	
3-EP, 4-EP	8.5 min
Quinoline	13.5 min
Nicotine	15 min