Designation: D5075 – 01 (Reapproved 2022)

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

This standard is issued under the fixed designation D5075; 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 macroreticular polystyrene-divinylbenzene copolymer (for example, XAD-4) sorbent tube attached to a sampling pump. Macroreticular polystyrene-divinylbenzene copolymer is referred to "sorbent resin" throughout this method. 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 sorbent 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 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary information is given in 13.6.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3631 Test Methods for Measuring Surface Atmospheric Pressure

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

E260 Practice for Packed Column Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1356 and Practice E355.

3.2 *Definitions of Terms Specific to This Standard:*

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

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

## 4. Summary of Test Method

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

[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.

4.2 The 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.

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 recommended exposure limit (REL) 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 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 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 *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 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.