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.

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 D1357.

9.2 *Procedure:*

9.2.1 Prepare 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 D5337 for standard practice in calibrating personal sampling pumps.

9.2.3 After the 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.

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

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 tube. A shorter sampling time is necessary if sample concentration and duration of sampling suggest a breakthrough occurrence.

11. Procedure

11.1 *Sorbent Tube Analysis*—The analytical procedure for nicotine and 3-EP is performed by extracting the sorbent resin with modified ethyl acetate solvent followed by GC/NPD analysis. Ethyl acetate extracts nicotine and 3-EP from the sorbent resin, but the solvent is modified with 0.01 % *v/v* triethylamine to prevent any adsorption of nicotine on the glass walls of the vials (**14**). The solvent also contains the internal standard quinoline, at a concentration of approximately 8 µg/mL. Solvent henceforth will refer to this modified ethyl acetate solvent.

11.2 *Preparation of Modified Ethyl Acetate Solvent:*

11.2.1 To a previously unopened 4-L bottle of ethyl acetate, add 0.5 mL triethylamine and 30 µL quinoline. Shake vigorously to mix.

11.2.1.1 To a separate freshly opened 4-L bottle of ethyl acetate, add 0.5 mL triethylamine; shake vigorously to mix. Use modified solvent containing no internal standard only when specifically called for in the procedure.

11.2.2 Store the modified ethyl acetate solvent containing quinoline at 4 °C or less when not in use. Allow the solvent to reach room temperature before using it to prepare standard solutions or samples.

11.2.3 Prepare fresh modified solvent as needed. Deterioration of the modified solvent has not been observed, and no definitive time interval has been established for its replacement; however, storage and use for more than 12 months is not recommended.

NOTE 3—In order to keep the amount of internal standard constant for both standards and samples, the same batch of modified solvent that is used to prepare standard solutions must be used to extract samples. Therefore, whenever a new batch of modified solvent is prepared, a new batch of standard solutions must also be prepared. Otherwise, if standards and samples contain different amounts of internal standard, the exact amounts in both solutions must be known precisely, and the regression and equations in 11.6.1 and 12.1 must be modified to reflect the different internal standard concentrations.

11.3 *Preparation of Standard Solutions:*

11.3.1 Clean all volumetric flasks and screw-cap jars used for the preparation and storage of standard solutions with detergent, thoroughly rinse with tap water followed by distilled water followed by ethyl acetate containing only 0.01 % triethylamine with no quinoline, and allow to air dry.

11.3.2 Prepare a primary standard of nicotine containing 400 µg/mL by weighing 100 mg of nicotine directly into a 250-mL volumetric flask, diluting to volume with solvent, and shaking to mix. Prepare a primary standard of 4-EP containing 500 µg/mL by weighing 100 mg of 4-EP into a 200-mL volumetric flask, diluting to volume with solvent, and shaking to mix. Prepare a secondary standard containing 4.8 µg/mL nicotine and 2.0 µg/mL 4-EP by transferring 3.0 mL of the primary nicotine standard and 1.0 mL of the primary 4-EP standard to a 250-mL volumetric flask, diluting to volume with solvent, and shaking to mix. Use the secondary standard as one of five calibration standards, and prepare the remaining four calibration standards from the secondary standard by transferring 30.0, 15.0, 6.0, and 2.0 mL of the secondary standard to each of four 100-mL volumetric flasks, diluting each to volume with solvent, and shaking each standard to mix. This provides a calibration range with the following concentrations of nicotine: 6.0, 1.80, 0.90, 0.36, and 0.12 µg/1.25 mL. The corresponding range for 4-EP is: 2.5, 0.75, 0.375, 0.15, and 0.05 µg/1.25 mL. These ranges typically cover the expected ranges of nicotine and 3-EP concentrations in the samples.

11.3.2.1 For the determination of desorption efficiency, prepare primary spiking standards of nicotine and 4-EP as described in 11.3.2, except dilute them in the ethyl acetate that contains only 0.01 % triethylamine and no quinoline. Then prepare a secondary spiking standard containing 9.6 µg/mL nicotine and 4.0 µg/mL 4-EP by transferring 6.0 mL and 2.0 mL of the primary nicotine and 4-EP spiking standards, respectively, to a 250-mL volumetric flask, also diluting with ethyl acetate containing only 0.01 % triethylamine and no quinoline, and shaking to mix. Use this secondary spiking standard in 11.7.2 for desorption efficiency determination.

11.3.3 Store all standards in borosilicate glass screw-cap jars at −10 °C or less when not in use. Allow standards to reach room temperature and transfer approximately 1 mL of each of the five calibration standards to two 2-mL autosampler vials each day for instrument calibration. Cap and tightly seal the vials.

11.3.4 Prepare fresh secondary and calibration standards as needed. Prepare fresh primary standards from neat nicotine and 4-EP once every 6 months.

11.4 *Extraction/Desorption of Sorbent Resin:*

11.4.1 In preparation for analysis, the analyst thoroughly washes his or her hands with soap and water immediately prior to handling the samples and refrains from smoking or otherwise contacting a known nicotine-containing environment until all samples and standards have been prepared and loaded in the autosampler tray.

11.4.2 Extraction/desorption of the sorbent resin requires transferring the contents of each sorbent tube to an autosampler vial for extraction. Prepare and analyze two previously unopened sorbent tubes as laboratory blanks. If sorbent tube samples have been stored frozen, allow them to equilibrate to room temperature before beginning the extraction procedure. Remove the plastic caps from the ends of each sorbent tube. To facilitate the transfer of the sorbent tube contents, widen the front and back openings of the tube by scoring the glass with a file and breaking. Use forceps and a glass wool removal tool to help transfer the entire contents of the tube (resin, glass wool plugs, and metal lockspring) to the autosampler vial.

NOTE 4—If the resin beads cling to the glass walls of the tube, push

them out using the glass wool. If this does not work, flush them out of the tube with a stream of air.

11.4.2.1 Label each vial. Add exactly 1.25 mL of solvent to each sample vial. Cap and tightly seal the vials and place them in a holding tray. After all samples have been prepared, transfer the tray to a wrist-action shaking device and extract under agitation for 30 min.

11.5 *Loading the Autosampler:*

11.5.1 Load one set of the five calibration standards at the beginning of the autosampler queue. Next, load all samples, flow blanks, and laboratory blanks. Load the second set of five calibration standards at the end of the autosampler queue.

NOTE 5—In the event that more than 40 sample vials are loaded after the first five standards, additional sets of standards should be loaded within the tray so that no more than 40 samples are analyzed between standards. Place the same number of samples before and after the middle set of calibration standards.

11.5.1.1 Load the autosampler with wash and waste vials. The wash vials should contain ethyl acetate with 0.01 % triethylamine and no quinoline. The operating conditions for the GC are listed in Table 1. Make one trial injection of the first calibration standard in the queue in order to verify correct operation of the GC in terms of peak location and detector sensitivity.

11.5.2 Obtain integrated peak areas and peak area ratios of analyte to quinoline for all standards, samples, and blanks by way of the chromatography data acquisition system. The peak area ratios of the samples and standards are compared, and concentrations of nicotine and 3-EP are calculated using the nicotine and 4-EP calibration curves. Fig. 1 shows a typical chromatogram from an ETS sample.

NOTE 6—Response factors for 3-EP and 4-EP have been determined to be equivalent (**15**), and the two isomers have the same retention time and peak shape under the chromatographic conditions listed.

11.6 *Constructing the Calibration Curve:*

11.6.1 For the internal standard method of quantitation, two calibration curves are constructed: (*1*) a plot of the mean peak area ratio of nicotine to quinoline (*y*-axis) versus the concentration of nicotine (in µg/1.25 mL on the *x*-axis) in the calibration standards, and (*2*) a plot of the mean peak area ratio of 4-EP to quinoline (*y*-axis) versus the concentration of 4-EP (in µg/1.25 mL on the *x*-axis) in the standards. The data from each plot are then fit to a second-order polynomial regression model with $1/x$ weighting.

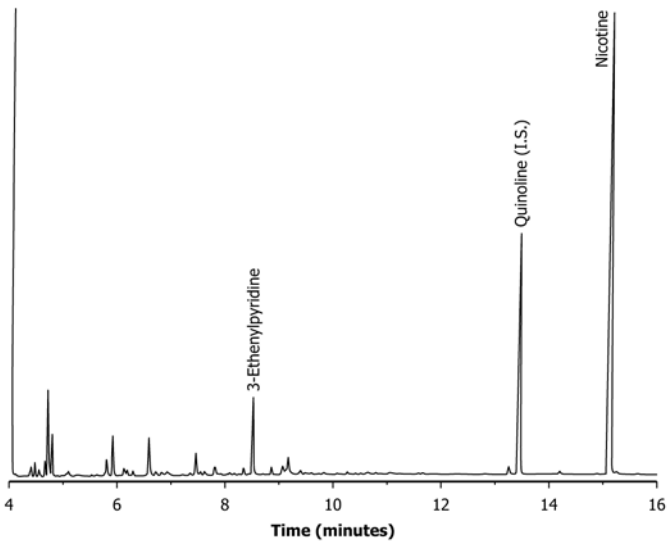

FIG. 1 Chromatogram of an Environmental Tobacco Smoke (ETS) Sample

NOTE 7—Other regression models may be deemed more appropriate and, if so, may be used instead of the second-order regression. If other models are used, the appropriate regression equations must be substituted in the calculations in 12.1.

11.6.2 The correlation coefficients of the fitted lines are expected to be at least 0.990 for this test method. A significantly lower value indicates unusual scatter in the data points defining the calibration curve, and preparation and analysis of additional standards should be carried out.

11.7 *Determination of Desorption Efficiency:*

11.7.1 Determine the decimal fraction of nicotine and 4-EP recovered in the desorption process for every batch of sorbent tubes that are received.

11.7.2 Break open 20 sorbent tubes and transfer the resin of each tube together with the front (inlet) glass wool plug and the metal lockspring to a 2-mL autosampler vial. Spike three sets of five vials by adding the secondary spiking standard prepared in 11.3.2.1 directly to the bed of resin in each vial. Add 10 µL (0.096 µg nicotine; 0.04 µg 4-EP) of the secondary spiking standard to each vial in the first set, 20 µL (0.192 µg nicotine; 0.08 µg 4-EP) to each vial in the second set, and 50 µL (0.48 µg nicotine; 0.20 µg 4-EP) to each vial in the third set. The remaining set of vials will be blanks.

NOTE 8—For determination of desorption efficiency, it is important that the standard solutions prepared in 11.3.2 are not used to spike the vials of resin because these standards already contain quinoline, the internal standard.

11.7.3 Cap, tightly seal, and store all vials under the same storage conditions as the samples. Since the desorption efficiency may be dependent on the length of time the sample tubes are stored, choose the storage time of the vials as the average time required to analyze field samples.

11.7.4 Aliquot ten calibration standards as described in 11.3.3.

11.7.5 If the vials have been stored frozen, allow them to equilibrate to room temperature before beginning the desorption procedure. Uncap each of the 20 vials. Then desorb and analyze as described in 11.4 and 11.5.

11.7.6 The desorption efficiency is defined as the average weight of analyte recovered from the tube divided by the weight of analyte added to the tube:

$$\text{desorption efficiency} = \frac{\text{average weight } (\mu g) \text{ recovered}}{\text{weight } (\mu g) \text{ added}} \quad (1)$$

11.7.7 The desorption efficiency may be dependent on the amount of analyte collected on the sorbent resin. If so, construct a plot of desorption efficiency versus weight of analyte found experimentally (not the amount added).

11.7.8 In most cases, the desorption efficiency is 1.00 over the calibration ranges suggested in 11.3.2 (**2,14**).