

**Designation:** E1413 – 13 E1413 – 19

# Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration onto an Adsorbent Tube<sup>1</sup>

This standard is issued under the fixed designation E1413; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 This practice describes the procedure for separation of small quantities of ignitable liquid residues from fire debris samples using the method of dynamic headspace concentration. dynamic headspace concentration onto an adsorbent tube, with subsequent solvent elution or thermal desorption.
- 1.2 Dynamic headspace concentration uses adsorption and subsequent solvent elution or thermal desorption.onto an adsorbent tube takes place from a closed, rigid sample container (typically a metal can), using a source of dry inert gas or a vacuum system.
- 1.3 Both positive and negative <u>applied</u> pressure systems for <del>adsorption are described, as well as a thermal desorption system.dynamic headspace concentration onto an adsorbent tube are illustrated and described.</del>
- 1.4 While this This practice is suitable for successfully extracting ignitable liquid residues over the entire range of concentration, the headspace concentration methods are best used when a high level of sensitivity is required due to a very low concentration preparing extracts from fire debris samples containing a range of volumes (µL to mL) of ignitable liquid residues in the sample: residues, with sufficient recovery for subsequent qualitative analysis (1).<sup>2</sup>
- 1.5 Alternate separation and concentration procedures Alternative headspace concentration methods are listed in Section 2. If archival of the extract is of importance, then this practice's thermal desorption procedure, SPME (Practice (see Practices E2154), and headspace (Practice E1388), E1412 sample, E3189 separation techniques are not recommended unless a portion of the extract can be split and retained. In order to have an archivable extract, then this practice's sample collection on charcoal, solvent extraction (Practice , and E1386E2154), or passive headspace concentration (Practice ).E1412) is recommended.
  - 1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.7 This practice offers a set of instructions for performing one or more specific operations. This standard cannot replace knowledge, skill, skills, or abilityabilities acquired through appropriate education, training, and experience (Practice E2917) and should is to be used in conjunction with sound professional judgment by individuals with such discipline-specific knowledge, skills, and abilities.
- 1.8 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 safety, health, and health environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.9 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:<sup>3</sup>

E1386 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction E1388 Practice for Static Headspace Sampling of Vapors from Fire Debris Samples

<sup>&</sup>lt;sup>1</sup> This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics. Current edition approved Aug. 15, 2013 June 1, 2019. Published September 2013 June 2019. Originally approved in 1991. Last previous edition approved in 2007 2013 as E1413 – 07.E1413 – 13. DOI: 10.1520/E1413-13.10.1520/E1413-19.

<sup>&</sup>lt;sup>2</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

<sup>&</sup>lt;sup>3</sup> 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.



- E1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal
- E1459 Guide for Physical Evidence Labeling and Related Documentation
- E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory
- E1618 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry
- E1732 Terminology Relating to Forensic Science
- E2154 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)
- E2451 Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples
- E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs
- E3189 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Static Headspace Concentration onto an Adsorbent Tube

# 3. Terminology

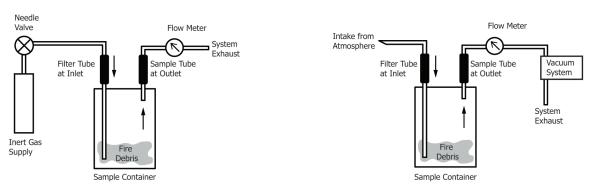
- 3.1 Definitions—For definitions of terms used in this practice, refer to Terminology E1732.
- 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 dynamic headspace concentration, n—an extraction technique in which a portion of the headspace vapors is exchanged from the sample container and concentrated onto an adsorbent medium through applied positive or negative pressure.
- 3.2.2 *static headspace concentration, n*—an extraction technique in which a portion of the headspace vapors is removed from the sample container and concentrated onto an adsorbent medium.

## 4. Summary of Practice

- 4.1 The sample, preferably in its original container, is heated, forcing volatile compounds to vaporize. The headspace in the sample is then drawn or pushed through a tube containing an adsorption media (typically activated charcoal or Tenax) which adsorbs the vaporized compounds. Headspace vapors from the closed sample container are collected and concentrated onto an adsorbent tube by means of headspace exchange performed through the use of a system based on either positive or negative applied pressure. The adsorbent tube is subsequently eluted with solvent or desorbed thermally prior to instrumental analysis (typically by gas chromatography-mass spectrometry (GC-MS)).
- 4.2 Other solid adsorbents The technique of dynamic headspace concentration onto an adsorbent tube is illustrated in Fig. 1 and collection systems can be used as long as the. The sample container, and the adsorbent tube when necessary, can be heated during sampling, as outlined in Section 9 method has been validated with a wide variety of ignitable liquids with a range of both flash points and polarity.

# 5. Significance and Use

- 5.1 This practice is useful for preparing extracts from fire debris for <u>later\_subsequent\_qualitative\_analysis</u> by gas <del>chromatography-mass spectrometry (GC-MS),</del>chromatography mass spectrometry, see Test Method E1618.
- 5.2 This is a very sensitive separation procedure, capable of isolating quantities smaller than The sensitivity of this practice is such that a sample consisting of a laboratory tissue onto which as little as 0.1 µL of ignitable liquid residue from a sample has been deposited, in an otherwise empty sample container, will result in an extract that is sufficient for identification and classification using Test Method E1618 (1).



Left: Positive applied pressure apparatus. Right: Negative applied pressure apparatus.

FIG. 1 Illustration of Dynamic Headspace Concentration onto an Adsorbent Tube

- 5.2.1 <u>Actual recovery Recovery from fire debris samples</u> will vary, depending on <u>several factors</u> including <u>adsorption debris</u> temperature, <u>container size</u>, <u>and adsorbent temperature</u>, <u>container size</u>, <u>adsorptive material</u>, <u>headspace volume</u>, sampling time and flow rate, and adsorptive competition from the sample <u>matrix</u>.matrix (2).
- 5.3 This is a potentially destructive technique. Portions of the sample subjected to this procedure may not be suitable for re-sampling. Therefore, a portion of the sample extract should The principal concepts of dynamic headspace concentration are similar to those of static headspace concentration (Practice E3189be saved for potential future analysis. Consider using passive headspace concentration as described in Practice). The dynamic headspace concentration technique can be more sensitive than the static headspace concentration technique. However, sample containers E1412. subjected to dynamic headspace concentration could be unsuitable for re-sampling.
- 5.3.1 Dynamic headspace concentration alters the original composition of the test sample because a portion of the original headspace from the sample container is removed and exchanged with dry inert gas or air. A portion of the concentrated headspace sample should be preserved for potential future analysis, if possible and if required, in accordance with Practice E2451.
- 5.4 Common solid adsorbent/desorption procedure combinations in use are activated carbon/solvent elution, and Tenax<sup>4</sup> TA/thermal desorption.
- 5.5 Solid adsorbent/desorption procedure combinations not specifically described in this standard can be used as long as the practice has been validated as outlined in Section 11.

#### 6. Apparatus

- 6.1 Positive Pressure Applied Pressure-Based Sampling System—A system capable of increasing the pressure inside the sample container by applying dry inert gas at the sample container inlet, thereby pushing the headspace vapors toward and through the outlet.
- 6.1.1 Sample Pressurization Device—A system capable of delivering pressurized dry nitrogen (or other inert gas) inert gas at up to 40 psi (276 kPa) to an orifice to be inserted into the bottom of the sample container approximately 276 kPa (40 psi) to the inlet.
- 6.1.1.1 A needle valve capable of fine control of the flow rate of the dry nitrogen, inert gas, at up to 1500 cc/min.approximately 400 mL/min.
- 5.1.1.2 A flow meter capable of measuring the flow of dry nitrogen through the end of the adsorption tube at the rate of 0 to 1500 ce/min.
- 5.1.1.3 Container Closure—A device suitable for sealing the container and directing the effluent nitrogen (or other inert gas) and vapors to the adsorption tube.
- 6.1.2 A filtering tube, consisting of an adsorbent tube as described in 7.5, attached to the sample container inlet, for the purpose of filtering incoming dry inert gas.
- 6.1.3 A *flow meter* capable of measuring the flow of the dry inert gas through the end of the adsorption tube at the rate of approximately 40 to 400 mL/min.
- 6.1.4 Design and use the sampling system in a manner that prevents the loss of volatile compounds from the container and contamination of the sample from the apparatus itself or the laboratory environment.
  - 6.2 Negative Pressure Apparatus: Applied Pressure-Based Sampling System—
- 5.2.1 Inlet and Outlet System—A tube containing approximately 1 cm of charcoal is fitted into the lid of the original container with a suitable penetrable seal. This serves as a filter for incoming room air. A vacuum is pulled on an adsorption tube also fitted into the lid of the original container with a suitable penetrable seal. A system capable of creating partial vacuum inside the sample container by connecting a vacuum system to the outlet, thereby pulling the headspace vapors toward and through the outlet.
- 6.2.1 A *vacuum system* capable of pulling between <del>200 and 1500 cc/min on the sample collection tube.</del> approximately 40 and 400 mL/min at the outlet.
- 6.2.2 A filtering tube, consisting of an adsorbent tube as described in 7.5, attached to the sample container inlet, for the purpose of filtering incoming air.
- 6.2.3 A *flow meter* capable of measuring the flow of <del>air through the end of the charcoal tube filtered air and headspace vapors through the outlet at the rate of <del>200 to 1500 ce/min.approximately 40 to 400 mL/min.</del></del>
- 6.2.4 Design and use the sampling system in a manner that prevents the loss of volatile compounds from the container and contamination of the sample from the apparatus itself or the laboratory environment.
- 6.3 Heating System—An oven large enough to accommodate the sample container, or a heating mantel designed to fit the sample container, capable of maintaining the required temperature range uniformly throughout.
- 6.4 Temperature Measuring Device—A thermometer or thermocouple capable of measuring the temperature of the heating system in the required range of operation, to within approximately 5°C.
  - 6.5 Thermal Desorption Apparatus: System—

<sup>&</sup>lt;sup>4</sup> Tenax is a trademark of Teijin Carbon America, Inc., Rockwood, TN.

- 5.3.1 Sampling System—An air-tight syringe is connected to the back-end of a Tenax tube. The front end of the Tenax tube is fitted into the heated container. A system capable of desorbing trapped volatile compounds from an adsorbent tube by means of elevated temperature, refocusing them on a cold-trap, and subsequently introducing them to a capillary GC column by flash heating. The desorption apparatus is directly coupled to a GC-MS.
- 5.3.2 In order to prevent leakage and contamination, the system should be designed to minimize loss of vapors from the container. For example by means of a septum mounted on top of the lid of the container.
- 5.3.3 Thermal Desorption Device—A system capable of desorbing the volatiles by means of elevated temperature and trapping the volatiles in a cold-trap. This apparatus is directly coupled to a GC-MS.
  - 6.6 Adsorption Tubes: Puncturing Device—
- 5.4.1 Charcoal Tubes—Suitable charcoal filter and sample adsorption tubes may be made by inserting a small (approximately 1 cm) plug of glass wool or cotton in the bottom of a Pasteur pipette (approximately 5 mm diameter), then adding 2.5 to 5 cm of activated charcoal, and finally, holding the charcoal in place with an additional plug of glass wool or cotton.
- 5.4.1.1 Alternatively, charcoal tubes are available from commercial sources. A device, such as a nail, that is capable of puncturing small holes in the lid of the sample container. The size of the holes is such that the adsorbent tubes can be introduced.
- 5.4.2 Tenax Tubes—Suitable Tenax tubes are commercially available. Note that solvent extraction of these tubes results in complications in the desorbing phase because certain solvents cause the dissolution of the Tenax. Tenax should be employed when thermal desorption is to be performed.
  - 5.5 Heating System—A heating mantel designed to fit the evidence container or an oven or a hot plate.
  - 5.5.1 An oven may be set up with any number of stations to allow for multiple sample preparation.
- 5.6 Temperature Measuring Device—A thermometer or thermocouple capable of measuring temperatures in the range of 40 to 150°C.

#### 7. Reagents and Materials

- 7.1 Purity of Reagents—Reagent Use reagent grade or better 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 through evaluation of appropriate blank(s) to permit its use without lessening the accuracy of the determination.
  - 7.2 Solid Adsorbent: Filter—
- **Document Preview**
- 6.2.1 Activated Charcoal (coconut).
- 6.2.1.1 Charcoal may be activated and cleaned by heating in a 400°C oven for approximately 4 hours and cooling in a desiceator.

  ASTM F1413-19
- 6.2.1.2 Test each new or reconditioned batch of charcoal for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media according to Test Method E1618. Permeable material such as silanized glass wool, wire mesh or cotton held in place with a frit.
  - 6.2.2 Tenax:
  - 6.2.2.1 Tenax may be cleaned and conditioned according to the manufacturer's instructions.
- 6.2.2.2 Test each new or reconditioned batch of Tenax for residual hydrocarbons by analyzing a concentrated extract of blank adsorption media according to Test Method E1618.
  - 7.3 Solid Adsorbent—Activated carbon, Tenax TA or equivalent.
- 7.4 Sampling Tubes—Glass Pasteur pipettes or equivalent glass tubes for solvent elution, and stainless steel or glass tubes for thermal desorption.
- 7.5 Glass Wool, Adsorbent Tubes—or cotton, free of extractable hydrocarbons. Sampling tubes packed with a solid adsorbent.
- 7.5.1 Pre-packed adsorbent tubes are commercially available. Empty sampling tubes that can be packed by the purchaser are also available.
- 7.5.1.1 Activated carbon tubes and equivalent for solvent elution can be made by inserting a filter into the bottom of a sampling tube (approximately 5-mm diameter), then adding 2.5 cm to 5 cm of activated carbon or equivalent, which is held in place with a second filter.
- 7.5.1.2 Tenax TA tubes and equivalent for thermal desorption can be made by inserting a filter into one end of a sampling tube, adding approximately 80 mg of pre-conditioned Tenax TA or equivalent, and then packing tightly with a second filter.
  - Note 1—Tenax TA or equivalent is conditioned by heating, in accordance with instrument manufacturer or supplier instructions.

<sup>&</sup>lt;sup>5</sup> Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. PharmaceuticalPharmacopeial Convention, Inc. (USPC), Rockville, MD.



- 7.6 Extraction solvent (for example, Screw cap earbonor disulfide, pentane, ethylcrimp-top glass vials ether). with polytetrafluoroethylene (PTFE) lined seals.
  - 6.4.1 Read and follow the safety precautions described in the Safety Data Sheets (SDS) of the extraction solvent that is used.
  - 6.4.2 Test each new lot number of the extraction solvent by analyzing a concentrated portion according to Test Method E1618.
  - 7.7 Tape, rubber sleeve stopper, or equivalent.
- 7.8 <u>Elution Solvent—Pasteur pipettes and glass vials freeSuitable elution solvents include carbon disulfide, of nextractable hydrocarbons.</u>-pentane, dichloromethane and diethyl ether.

### 8. Sample Preparation

- 8.1 Observe the appropriate procedures for handling and documentation of all submitted samples (Guide (see Guide E1459 and Practice E1492).
  - 7.1.1 Examine the fire debris sample in order to determine that it is consistent with its description.
- 7.1.1.1 Resolve any discrepancies between the submitting agent's description of the evidence and the analyst's observation with the submitting agent prior to the completion of the report.
- 7.2 This is a potentially destructive technique. Portions of the sample subjected to this procedure may not be suitable for re-sampling. Therefore, a portion of the sample extract should be saved for potential future analysis. Consider using passive headspace concentration as described in Practice E1412 which is essentially nondestructive.
- 8.2 The system should be designed so that the majority of the samples can be extracted from the container in which they are delivered to the laboratory. Prepare the fire debris sample container for sampling by dynamic headspace concentration.
- 8.2.1 Alternatively, the sample or a portion of the sample can be placed in an appropriate, clean sampling container which is designed to be flushed by positive or negative pressure. The sampling system is designed so that the headspace sample can be extracted from the container in which it was received at the laboratory.
- 8.2.1.1 Alternatively, the fire debris sample, or a portion of it, can be transferred to a clean, rigid sample container suitable for dynamic headspace sampling, such as a metal can with a friction-seal lid.
- 8.2.1.2 Verify the cleanliness of the transfer container prior to sample transfer. Cleanliness is determined by means of analysis of a dynamic headspace concentration sample obtained from the empty transfer container using the same conditions as will be used for the questioned sample.
  - 8.2.1.3 Allow the transfer container with sample to equilibrate for at least one hour before sampling
- 8.2.2 Create two holes in the lid of the rigid sample container using a puncturing device, such that the adsorbent tubes can be introduced.
  - 8.2.2.1 Seal the holes with tape, rubber sleeve stopper, or equivalent.
  - Note 2—Cans that are designed for fire debris samples, with a hole pre-fitted with a rubber sleeve stopper, are commercially available.

# 9. Adsorption Procedure ai/catalog/standards/sist/ea5417ca-c410-4e93-a2e1-4a7e88e741b8/astm-e1413-19

- 9.1 A laboratory typically has one method of adsorption and clution that is employed. The most common combinations in use are charcoal adsorption followed by solvent clution, and Tenax adsorption followed by thermal desorption. Both adsorbents can be collected using uses either a positive applied pressure-based system or a negative pressure system. Other solid adsorbents and collection systems applied pressure-based system. Either type of system can be used as long as the method has been validated with a wide variety of ignitable with the adsorbent tubes described in 7.5 liquids with a range of both flash points and polarity.
- 8.1.1 The volume of air sampled is generally less when thermal desorption is employed as the entire collected sample is commonly injected into the GC-MS system for analysis.
- 8.1.2 With thermal desorption there is typically no material that can be archived once GC-MS analysis has been performed. If archival of a portion of the sample extract is needed, then a different adsorbent and elution combination should be chosen either following this practice, or following one of the other sample collection methods for fire debris analysis (Practices E1386 or E1412). Some instrument manufacturers now have the facility to collect the unused sample from the split; if your instrument has such capability, then there will be material available for archival when thermal desorption is used.
  - 9.2 Positive Pressure: Applied Pressure-Based System:
- 9.2.1 Place the inlet inert gas filtering adsorption tube and the outlet sample adsorption tube through the holes in the lid of the sample container by penetrating the tape, rubber sleeve stoppers, or equivalent covering the holes.
  - 9.2.1.1 Use a new inlet inert gas filtering adsorption tube for each sample container.
- 9.2.2 Place the sample container in the heating system and connect to the inert gas source (commonly nitrogen) which will introduce the gas into the bottom of the container, then connect the exhaust line to the adsorbing tube. Connect the sample pressurization device to the inlet inert gas filtering adsorption tube. Then connect the exhaust tube to the outlet sample adsorption tube. Direct the system exhaust to a lab fume hood or equivalent.
- 8.2.1.1 Direct connection of the adsorption tube to the sample container is possible, unless an oven is used. Placement of the adsorption tube outside the oven requires the use of an intervening line which must be carefully cleaned between uses to remove any adsorbed or condensed residues.

- 9.2.3 Pressurization—Deliver the dry inert gas to the sample through a ¼ in. (3.2 mm) line on the inlet side of the container at a pressure of 40 psig (276 kPa) upstream of the needle valve controlling the flow rate. Connect a container using the sample pressurization device ¼ in. (6.35 mm) line on the downstream side of the container to the charcoal tube. Attach the flow meter to the outlet side of the charcoal tube, and use the needle valve to and adjust the flow to the rate determined to be optimum for this system.rate.
- 9.2.3.1 Determine the optimum system conditions (flow rate, temperature and time) by placing known quantities of gasoline, kerosene, mineral spirits, and diesel fuel onto exemplar substrate material such as squares of carpetingA suitable flow rate is determined using the method verification as outlined in Section 11 or filter paper. Determine the percent recovery by gas chromatographic analysis. The conditions resulting in the highest percent recovery is the optimum. Typical flow rates are within the range of approximately 40 and 400 mL/min.
- 8.2.2.2 Remove the flow meter once the flow is adjusted. Check the flow at least once after the sample has reached the final temperature.
  - 9.2.4 Heat the sample container, when necessary, during the adsorption procedure.
- 9.2.4.1 Heating is not recommended if there are other evidentiary considerations such as preservation of DNA or latent fingerprints.
- 9.2.4.2 Place the sample container into the heating system at a suitable heating temperature, based upon parameters determined during the method verification as outlined in Section 11.
- Note 3—Sampling at room temperature is suitable for the extraction of the more volatile compounds, while heating increases the concentration of the less volatile compounds in the headspace.
- 9.2.4.3 Heat the container of fire debris at the selected temperature in order to release any ignitable liquid residues as a vapor into the headspace.
- 9.2.4.4 Heating the adsorbent tube while extracting wet samples can prevent condensation from interfering with the adsorption process (3).
- 9.2.5 Continue the adsorption procedure for the period of time determined during the method verification as outlined in Section 11.
- 9.2.5.1 Adsorption times can vary from minutes to hours, depending on the selected heating temperature, the size of the container, the concentration of ignitable liquid residues present in the sample, the type and amount of sample material in the container, and the selected adsorbent type and desorption procedure (solvent elution or thermal desorption).
- 9.2.5.2 An indication of the ignitable liquid residue concentration in a questioned sample can be obtained from a preanalysis using an alternative method such as static headspace sampling (Practice E1388) at room temperature, or elevated temperature, and subsequent analysis with gas chromatography-flame ionization detection (GC-FID) or GC-MS.
- 9.2.6 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample. Turn on the heating system and increase the temperature to no more than 150°C. At this temperature, most petroleum distillates can be volatilized in less than one hour, and there will be a minimum of pyrolysis of common polymers. Upon completion of the adsorption procedure, turn off the flow of dry inert gas, remove the inlet inert gas filtering adsorption tube and the outlet sample adsorption tube, and then seal the openings in the container lid with tape, rubber sleeve stoppers, or equivalent.
- 9.2.6.1 Allow 20 min for the sample to reach temperature, and an additional one hour flushing the sample out. At this point, turn off the gas. The charcoal If an adsorption tube is now ready for elution to be thermally desorbed, recap it immediately.
- 9.2.6.2 The adsorption time may be reduced if it can be shown that equal or superior recovery rates are achieved in the reduced time period. If an adsorption tube is to be desorbed by solvent elution and it was heated, allow the adsorption tube to cool to room temperature prior to performing the desorption procedure.
  - 9.3 Negative Pressure: Applied Pressure-Based System:
- 9.3.1 Place the inlet air filtering adsorption tube and the outlet adsorption tubes sample adsorption tube through the holes in the lid of the evidence container.sample container by penetrating the tape, rubber sleeve stoppers, or equivalent covering the holes.
  - 9.3.1.1 Use a new inlet air filtering adsorption tube for each sample container.
- 8.3.2 Place the temperature measuring device in an appropriate location for the system used in order to monitor the temperature of the sample.
- 9.3.2 Apply a vacuum to the <u>outlet</u> sample adsorption tube. The flow through the tube should be between 200 and 1500 cc/min. tube using the vacuum system, and direct the system exhaust to a lab fume hood or equivalent. Adjust the applied vacuum to achieve the selected flow rate.
- 9.3.2.1 A suitable flow rate is determined during the method verification as outlined in Section 11. Typical flow rates are within the range of approximately 40 and 400 mL/min.
  - 9.3.3 Heat the sample container, when necessary, during the adsorption procedure.
- 9.3.3.1 Heating is not recommended if there are other evidentiary considerations such as preservation of DNA or latent fingerprints.
- 9.3.3.2 Place the sample container into the heating system at a suitable heating temperature, based upon parameters determined during the method verification as outlined in Section 11.