ACTIVATED CHARCOAL STRIPS

Instruction Manual
& Technical Guide
For Forensic Laboratories

ARROWHEAD FORENSICS

INTRODUCTION

In general, forensic chemists may use one of three techniques to extract flammable and or combustible liquids from fire debris. These are:

1. Manual Treated HeadspacePurge and Trap
2. Passive Headspace

Manual Treated Headspace

This technique involves heating a sealed sample container in order to drive flammable and or combustible organic vapors from the fire debris into the air space within the container. After a suitable time period, a gas tight syringe is used to extract a given volume of the resulting air/vapor mixture. This mixture is then injected directly into a gas chromatograph for analysis.

While this technique is rapid and unobtrusive, it has several drawbacks. First, the technique is not very efficient because the extraction volume is usually limited to five milliliters or less. As such, the total volume of flammable or combustible organic vapors withdrawn may be too low to detect. In addition, significant condensation of the heavier molecular weight compounds can occur in the barrel of the syringe thus skewing the resulting chromatogram to favor the lighter weight compounds.

Purge and Trap

Many of the drawbacks associated with the manual heated headspace technique are overcome with the purge and trap technique. The purge and trap process involves placing the fire debris into an unused metal can which is sealed with a specially outfitted lid. The lid has three fittings, two of which contain glass tubes packed with charcoal and one of which contains a thermometer. Each charcoal tube is open at both ends so that air can be drawn through the charcoal. One of the charcoal tubes is typically connected to a vacuum source, and while heating, air is drawn into the sample container through the other charcoal tube.. The air movement carries any existing flammable or combustible organic vapors into the charcoal tube connected to the vacuum source where they are trapped.

The purge and trap technique is generally superior to the manual heated headspace technique because all or most of the flammable and or combustible organic vapors are removed from the fire debris and concentrated onto the charcoal. While this technique is very efficient, it too suffers from a number of disadvantages. The primary disadvantage is that the technique is very slow and occupies a great deal of the chemist's time.

Another disadvantage results from the aggressive nature of the extraction. Because most samples contain water, the water vapor often acts to strip the VOC vapors from the charcoal. Furthermore, organic vapors associated with thermal degradation of the fire debris are also carried onto the charcoal as efficiently as the flammable and or combustible organic vapors, which can make data interpretation more difficult.

Passive Headspace

This extraction technique is performed by placing one or more charcoal strips (ACS) into a fire debris container, sealing, and heating for a given time period. During this time, flammable and combustible organic vapors will be released from the fire debris and enter the air space within the sample container. Due to molecular diffusion, the vapors will move through the air space where they will eventually contact the charcoal and be trapped.

Introduced in 1991, this technique has been adopted by over 250 forensic labs worldwide. This popularity is due primarily to the fact that the technique is very rapid and cost effective. Sample preparation is quick and requires minimal sample manipulation. Additionally, the number of samples that can be processed by a single chemist is limited only by the number of sample containers that can be placed into available oven space.

The passive diffusion technique exhibits equivalent if not superior extraction efficiency when compared to Purge and Trap or Manual Heated Headspace techniques. Because the technique is "passive", vapor loss mechanisms associated with the other techniques are completely precluded. In addition, the effects of humidity on collection efficiency are insignificant.

PRINCIPLE OF OPERATION

Activated charcoal (ACS) exhibits a very strong affinity for most vapors. This affinity is due primarily to the extremely high surface area associated with the charcoal. When a vapor molecule collides with the surface of the charcoal, it will be aggressively retained. As time passes, the vapor concentration in the container will drop, thereby upsetting the liquid/vapor equilibrium. To compensate, an equivalent number of molecules will evaporate from the liquid to make up for those adsorbed onto the charcoal which will continue until essentially all of the liquid is transferred to the charcoal surface. Eventually a new equilibrium will be established between vapor phase molecules and adsorbed phase molecules wherein the equilibrium will favor the adsorbed phase.

If the temperature of the sample container is increased, the vapor concentration will be increased thereby increasing the number of vapor molecule collisions with the charcoal. Accordingly, the transfer time will be reduced in direct proportion to the increase in collision frequency. In actual practice the temperature cannot be increased without limit because excessive pressures may develop within the container. Furthermore, high temperatures

encourage release of the by-products of pyrolytic decomposition which can act to mask flammable or combustible liquid components.

In summary, the key to an efficient extraction is to select a temperature which:

1. Provides maximum transfer of all VOCs to the charcoal,
2. Minimizes transfer of pyrolysis decomposition products from sample.
3. Accomplishes items 1 and 2 in a reasonable time period. Research has demonstrated that a temperature of 60 degrees centigrade and an extraction time of 16 hours are optimal for most samples.

LABORATORYAPPLICATION

1. Remove the cover from the fire debris sample container.

2. Place an ACS strip into the sample container.

   NOTE 1: DO NOT TOUCH THE ACS. USING CLEAN TWEEZERS, SIMPLY "DROP" THE ACS INTO THE FIRE DEBRIS SAMPLE CONTAINER.

3. Seal the container tightly.

4. Place the sample container into the oven for at least 16 hours at 60 degrees centigrade.

   NOTE 2: SOME LABS HAVE REPORTED THAT THEY USE HIGHER TEMPERTURES FOR EXTRACTION. THE HIGHER TEMPERATURE ALLOWS FOR A SHORTER EXTRACTION PERIOD. EXTREME CAUTION MUST BE USED WHEN USING HIGHER TEMPERATURES BECAUSE SIGNIFICANT VAPOR PRESSURE CAN DEVELOP WITHIN THE SEALED SAMPLE CONTAINER.

5. After 16 hours, turn the oven off and **allow the oven to cool to room temperature before removing the sample containers.**

6. Using clean tweezers, remove the ACS from the sample container.

   NOTE 3: PROCESS ONLY ONE SAMPLE AT A TIME. IN MOST CASES THE ACS WILL STILL BE ACTIVE AFTER IT IS REMOVED FROM THE SAMPLE CONTAINER AND, AS SUCH, THE CHARCOAL MUST BE PROMPTLY ANALYIZED OR SEALED IN A GLASS VIAL.

7. Place the charcoal strip into a suitable glass vial and add 0.5 milliliters of carbon disulfide. Agitate for 15 minutes to complete extraction.

   NOTE 4: MANY CHEMISTS USE LOWER EXTRACTION VOLUMES

OF CARBON DISULFIDE TO INCREASE SENSITIVITY DURING ANALYSIS. PLEASE NOTE, HOWEVER, THAT EXTRACTION VOLUMES LESS THAN 0.5 MILLILITERS MAY DECREASE SENSITIVITY SINCE EXTRACTION EFFICIENCY DROPS AS THE CARBON DISULFIDE VOLUME TO CHARCOAL SURFACE AREA RATIO DECREASES. WE HAVE FOUND THAT WHEN EXTRACTING THE FULL ACS STRIP (20mm) A VOLUME OF 0.5 MILLILITERS OF CARBON DISULFIDE PROVIDES AT LEAST 95% EXTRACTON EFFICIENCY FOR MOST AROMATIC AND ALIPHATIC COMPOUNDS.

8. It may be desirable to archive one or more pieces of the ACS for future confirmatory analysis. If this is required, the following steps are recommended:

    a. Before adding CS2, place the archive piece into a glass vial that can be sealed with a screw top cap. It is recommended that caps which employ a Teflon coated neoprene gasket be used rather than paper gaskets which may contain adhesive compounds.

    b. Place the sealed, labeled vial into a freezer (at least -4 degrees centigrade) for long term storage.

    c. Do not use 0.5 milliliters of carbon disulfide to extract the remaining ACS. Instead use a volume proportional to the amount of ACS remaining. For example, if the ACS was cut in half, use a 0.25 milliliter extraction volume to preserve analytical sensitivity.

NOTE 5: BE SURE THAT THE SYRINGE PROJECTS DEEPLY ENOUGH INTO THE GLASS VIAL TO EXTRACT SUFFICIENT ANALYTE.

9. Proceed with your normal analytical technique.

NOTE 6: WITH HUNDREDS OF LABS USING ACS, THERE ARE MANY TECHNIQUES FOR PLACING THE STRIPS IN THE SAMPLE CONTAINER. WE HAVE HEARD OF MANY INNOVATIVE METHODS, ALL OF WHICH SEEM TO WORK SATISFACTORILY. MANY FORENSIC SCIENTISTS JUST DROP AN ACS INTO THE CONTAINER. OTHERS USE TOOTHPICKS, FISH HOOKS, OR PAPER CLIPS AND TEFLON DENTAL FLOSS TO SUSPEND THE STRIP INTO THE HEADSPACE. WE DO NOT RECOMMEND ANY ONE TECHNIQUE BUT DO ADVISE YOU TO USE A METHOD THAT MINIMIZES THE AMOUNT OF TIME THAT THE SAMPLE CONTAINER IS UNSEALED AND THE ACS IS EXPOSED AFTER OPENING ITS CONTAINER. IT

IS EQUALLY IMPORTANT TO INSURE THAT THE ACS SUPPLY CONTAINER IS TIGHTLY CLOSED IMMEDIATELY AFTER REMOVING THE STRIP

QUALITY ASSURANCE

Meeting customer expectations with superior performance is the basis of our quality assurance standards.

1. **Charcoal Capacity.** The charcoal capacity is a critical parameter for ensuring effective extractions under most situations. It is a quality goal that the charcoal strip has enough capacity to hold a minimum of 20 ul of SAM.

    a. The charcoal capacity is tested by placing an ACS into a sealed can along with 100 ul of SAM. The can is placed into the oven for 16 hours at 60 degrees centigrade. The strip is removed from the device and is desorbed in 1 milliliter of carbon disulfide. The TICA is compared to a calibration curve prepared from SAM standards to determine the charcoal capacity.

    While the design capacity for ACS is 20 ul of SAM, the average capacity of a representative number of tests cannot be less than 25 ul for acceptance.

2. **Charcoal cleanliness.** The charcoal must be free of organic contaminants in order to ensure unambiguous analytical results. It is a quality goal that each ACS is a contaminant free charcoal strip. In this context, an ACS is determined to be contaminant free when no peaks above the peak height of a .001 uL/mL sample of Toluene are observed above the chromatogram baseline which is produced from a Flame Ionization Detector at maximum sensitivity and, under conditions of minimal split ratio.

    a. The charcoal is activated and cleaned in a single proprietary step. After each activation/cleaning process a random selection of charcoal strips are analyzed to determine cleanliness as defined above. If it is determined that any pieces are contaminated the activation process is repeated until cleanliness is achieved.

3. **Vendor archive.** As a final QA effort, a representative number of ACS are retained from each lot. These ACS can be retrieved when requested for additional QA testing if required.

4. **Documentation.** All documentation generated while performing these QA tests are retained for a period of five years at the manufacturing facility. These documents can be ordered for your own records at any time within this five year period for a nominal processing charge.

5. **Storage.** ACS should be stored in a clean, dry place and at temperatures which do not exceed 33 degrees Celsius.