Polycyclic Aromatic Hydrocarbons in Cigarette Smoke by LC/MS

REFERENCE: Wingen. L.M.; J.C. Low; B.J. Finlayson-Pitts. Chromatography, Absorption, and Fluorescence: A New Instrumental Analysis Experiment on the Measurement of Polycyclic Aromatic Hydrocarbons in Cigarette Smoke. J. Chem. Ed 1998, 75, 1599-1603.

Purpose: Identify and quantify polycyclic aromatic hydrocarbons (PAHs) present in the smoke of unfiltered cigarettes.

Materials

Hirsch funnel with fritted disk	Flexible tubing
Ring stand with clamps	4 mL glass vial
2 mL sample vial with glass insert.	commercial, unfiltered cigarettes
Whatman 0.2 μm syringe filter	50μL glass syringe
Matches	1:1 solution of methylene chloride/methanol, both HPLC grade

Procedure

1. Attach one end of the flexible tubing to the Hirsh funnel, and the other to the vacuum line. The tube-funnel connection needs to be secured with a tubing clamp. This experiment will be done in the fume hood.
2. Attach the Hirsh funnel to the ring stand, cone side down.
3. Light the cigarette, and squeeze at the other end to simulate smoking. Be careful to position the cigarette so that as much of the smoke as possible enters the funnel. Continue until approximately 80% of the cigarette has been consumed. Repeat with a second cigarette.
4. Remove the funnel from the smoking apparatus. Add 1 mL of the 1:1 methylene chloride/methanol solution to the funnel, cone side up. Swirl the solvent wetting as much of the wall as possible with out spilling it.
5. Wet the Whatman syringe filter by tranfering ~1 mL of solvent (not your sample) through the sample.
6. Transfer the smoke particle containing solvent to the Whatman 0.2 μm syringe filter into a small glass vial. Filter.
7. Place the filtered solution in the fume hood in an un-capped vial to allow the solvent to evaporate.
8. During the next lab period, add 50μL of the solvent to the remaining residue. Wet the walls as much as possible.
9. Transfer to the glass insert placed inside of the 2 mL sample vial.

Analysis

Your cigarette sample will be analyzed using LC/MS and compared to an Ultrascientific PM-810 standard that contains 16 known PAHs. The samples will be analyzed using a total ion count in the mass-to-charge range 100.0 – 400.0. The instructor will show you several different traces of these data to assist you in identifying any PAHs present in the cigarette smoke. A calibration curve generated from the standard will be given to you to allow you to quantify the PAHs in your sample.