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Analysis of Atmospheric PAHs in the Austrian Pine

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Introduction

Polycyclic aromatic hydrocarbons (PAH) are a common pollutant created by natural (forest fires) or anthropogenic sources, such as incomplete combustion in engines (vehicular traffic) or incinerators (1). Many PAHs are known or suspected carcinogens and also have mutagenic properties and endocrine disrupting effects (2).

Although most atmospheric PAHs are measured using high-volume sample collection on filters, a convenient way of monitoring these pollutants is through plant matrices, such as pine needles that act as passive samplers. The needles are covered with a lipid-rich cuticle that absorbs the pollutants (1). Evergreens are especially useful because they continue to accumulate pollutants during the winter (3).

In order to use plants as monitors, a method to extract the PAHs must be used. Ultrasonication is a method that leaches the PAHs from the needles and into the solvent. The sonication agitates the pine needles and produces increased temperatures, which further abet the efficiency of the solvents' extraction. This rapid method can be applied to batches of samples. The procedure is followed by a cleanup step (4).

Experimental

Sample Collection

- Samples of third year pine needles were collected with scissors and placed in an amber bottle.
- Samples were kept in the dark due to PAH photosensitivity.
- Samples were allowed to air dry prior to extraction.

Extraction

- Needles were chopped into 2 mm size bits with a coffee grinder.
- Amber bottles were filled with 5.00 g of chopped needles and spiked with 1000 ng of deuterated standard.
- 30.0 mL of solvent was added to sample. Sample was sonicated three times after being left to soak overnight.
- All extracts were combined in a round bottom flask and rotovapped down to 2 mL.

Clean up

- Cartridges were prepared with 2.5 g of Florisil and 0.5 g of Na₂SO₄ and conditioned with 30 mL of solvent.
- The extract was loaded onto the column and eluted with solvent.
- The filtrate was then blown down and stored in vials.

Analysis

- Samples and standards were spiked with a 1 ppm solution before analysis
- Analysis was ran under SIM.

References

- (1) Nicola, Flavia De; Chemosphere 61 (2005) 432-440
- (2) Ratola, Nuno; Journal of Chromatography A, 1114 (2006) 198-204
- (3) Prajapati, Santosh Kumar; Environmental Pollution 151 (2008) 543-548
- (4) McGowin, A.E., "Polycyclic aromatic hydrocarbons" in Chromatographic Analysis of the Environment. CRC Press, UK, 2005
- (5) Srogi, K. "Monitoring of Environmental Exposure to Polycyclic Aromatic Hydrocarbons: A Review". Environ Chem Lett (2007) 5:169-195

Table 1 Analytes, Internal Standard, Surrogates

| Peak # | Compound Name | CAS no. | Molecular wt. | Retention Time |
|--------|-------------------------------------|--------------|---------------|----------------|
| 1 | Naphthalene ^a | [91-20-3] | 128 | 7.629 |
| 2 | Naphthalene-d8 ^a | [1146-65-2] | 136 | 7.589 |
| 3 | Acenaphthene ^a | [83-32-9] | 152 | 11.49 |
| 4 | Acenaphthene-d10 ^a | [15067-26-2] | 164 | 11.613 |
| 5 | Acenaphthene ^b | [208-96-8] | 153 | 11.683 |
| 6 | Fluorene ^a | [86-73-7] | 166 | 12.892 |
| 7 | Phenanthrene-d10 ^a | [1517-22-2] | 188 | 15.246 |
| 8 | Phenanthrene ^a | [85-01-8] | 178 | 15.303 |
| 9 | Anthracene ^a | [120-12-7] | 178 | 15.421 |
| 10 | Fluoranthene ^a | [206-44-0] | 202 | 18.551 |
| 11 | Pyrene ^a | [129-00-0] | 202 | 19.165 |
| 12 | p-terphenyl ^a | [92-94-4] | 230 | 19.808 |
| 13 | Benzo[a]anthracene ^a | [56-55-3] | 228 | 23.008 |
| 14 | Chrysene-d12 ^a | [1719-03-5] | 240 | 23.067 |
| 15 | Chrysene ^a | [218-01-9] | 228 | 23.152 |
| 16 | Benzo[b]fluoranthene ^a | [205-99-2] | 252 | 27.125 |
| 17 | Benzo[a]pyrene ^a | [50-32-8] | 252 | 27.220 |
| 18 | Benzo[k]fluoranthene ^a | [207-08-9] | 252 | 28.359 |
| 19 | Perylene-d12 ^a | [1520-96-3] | 264 | 28.811 |
| 20 | Indeno[1,2,3-cd]pyrene ^a | [193-39-5] | 276 | 32.697 |
| 21 | Dibenzo[a,h]anthracene ^a | [53-70-1] | 278 | 32.821 |
| 22 | Benzo[ghi]perylene ^a | [191-24-2] | 276 | 33.619 |

Legend
^a Deuterated Standard
^b Internal Standard
^c PAH

Pictures

Figure 1



Figure 2



Figure 3



GC Settings

Table 2 GC/MS Column Settings

| | |
|----------------------|---------|
| Column Length: | 30.00 m |
| Column: | 0.320 |
| Diameter: | mm |
| Gas: | He |
| Vacuum Compensation: | On |

Table 3 Oven Programming

| | |
|----------------------|----------|
| Initial Temperature: | 80 C |
| Initial Time: | 1.00 min |

| Level | Rate (C/minute) | Final Temperature (C) | Final Time (min) |
|-------|-----------------|-----------------------|------------------|
| 1 | 10 | 190 | 0 |
| 2(A) | 8 | 250 | 0 |
| 3(B) | 4 | 320 | 0 |

Next Run Time: 37 minutes

Table 4 Inlet Purge Settings

| Inlet Purge | Init Value | On Time | Off Time | Splitless Injection |
|-------------|------------|---------|----------|---------------------|
| A | Off | 0.50 | 5.00 | Yes |
| B | Off | 0.50 | 0.00 | No |

Figure 4

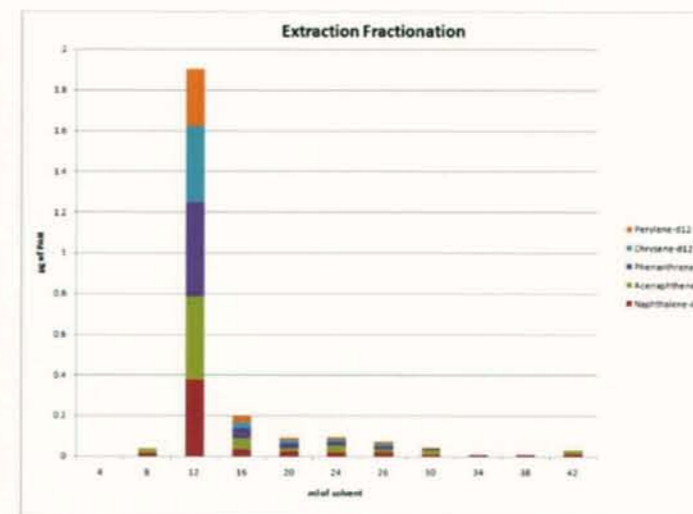


Table 5 μg of PAH in Each Fraction

| ml of Solvent | 4 | 8 | 12 | 16 | 20 | 24 | 28 | 32 | 36 | 40 | |
|--------------------------------|---|----------|----------|----------|----------|----------|----------|----------|----------|----------|---------|
| Naphthalene-d8 μg | 0 | 0.013442 | 0.373655 | 0.833393 | 0.021435 | 0.020162 | 0.019681 | 0.008383 | 0.006911 | 0.008273 | 0.01246 |
| Acenaphthene-d10 μg | 0 | 0.025515 | 0.409987 | 0.052328 | 0.017662 | 0.032771 | 0.012698 | 0.020458 | 0 | 0 | 0.01601 |
| Phenanthrene-d10 μg | 0 | 0 | 0.464331 | 0.049994 | 0.02385 | 0.018839 | 0.018158 | 0.008221 | 0 | 0 | 0 |
| Chrysene-d12 μg | 0 | 0 | 0.373745 | 0.031792 | 0.015289 | 0.011771 | 0.011417 | 0.002279 | 0 | 0 | 0 |
| Perylene-d12 μg | 0 | 0 | 0.278218 | 0.029684 | 0.012999 | 0.010501 | 0.010968 | 0.002804 | 0 | 0 | 0 |

Data and Results

Figure 5

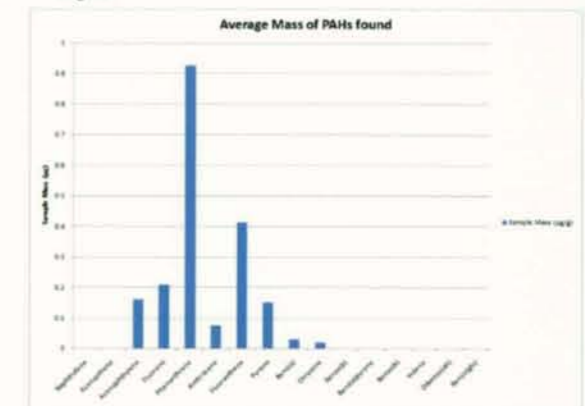


Table 6 Concentration of PAHs in Pine Needles

| Analyte | Sample A ($\mu\text{g/g}$) | Sample B ($\mu\text{g/g}$) | Average ($\mu\text{g/g}$) |
|----------------|------------------------------|------------------------------|-----------------------------|
| Naphthalene | 4.12042E-05 | 0 | 2.06021E-05 |
| Acenaphthene | 0 | 0 | 0 |
| Acenaphthylene | 0.143652209 | 0.176965365 | 0.160308787 |
| Fluorene | 0.190416537 | 0.226809904 | 0.20861322 |
| Phenanthrene | 0.878491698 | 0.975596 | 0.927043849 |
| Anthracene | 0.073089272 | 0.076943617 | 0.075016445 |
| Fluoranthene | 0.379905236 | 0.445438893 | 0.412672065 |
| Pyrene | 0.144134669 | 0.156893657 | 0.150514163 |
| Benzo(a) | 0.027250721 | 0.032378731 | 0.029814726 |
| Chrysene | 0.012496953 | 0.025256556 | 0.018876754 |
| Benzo(b) | 0 | 0 | 0 |
| Benzo(a)pyrene | 0 | 0 | 0 |
| Benzo(k) | 0 | 0 | 0 |
| Indeno | 0 | 0 | 0 |
| Dibenzo(ah) | 0 | 0 | 0 |
| Benzo(ghi) | 0 | 0 | 0 |
| Total | | | 1.982880612 |

Conclusion

Phenanthrene was exceptionally high, 927 ng/g, in the tree located at a busy urban intersection between two major roads and along a railroad track. The total PAH concentration (Σ_{PAHs}) was 1982 ng/g. This is comparable to pine needles concentrations in typical urban areas that range from 800-1600 ng/g(5). A high level of phenanthrene indicates the source is vehicle exhaust.

Improvements to the method will include increasing recovery and analyzing a larger sample.