NETCHEM

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ERASMUS+ PROJECT

NETCHEM

ICT Networking for Overcoming Technical and Social Barriers in Instrumental Analytical Chemistry Education

UNIVERSITY OF BELGRADE

NETCHEM Remote Access Lecture Guide

Distribution and sources of polycyclic aromatic hydrocarbons (PAHs) in sedimentary environment

Background

- Polycyclic aromatic hydrocarbons (PAHs) have been listed as priority compounds of environmental concern due to their carcinogenicity, mutagenicity, acute toxicity, and persistence.

- A total of 16 PAHs are listed by United States Environmental Protection Agency (US EPA) as priority pollutants.

- Polycyclic aromatic hydrocarbons originate from many natural and anthropogenic sources.

- PAHs have two anthropogenic sources: petrogenic and pyrogenic.

- Petrogenic source PAHs come from petroleum and petroleum products while pyrogenic source PAHs originate from incomplete combustion of fossil fuels and wood.

- However, because PAHs are generated by multiple activities, source identification and apportionment become necessary for future environmental management strategy.

❖ This identification has been based on specific molecular or isomeric ratios of individual PAH species, which are characteristic of particular sources.

Requirements

❖ Basic knowledge about
- ✓ PAHs,
- ✓ Gas chromatography –mass spectrometry analysis (GC-MS),
- ✓ Data Analysis

PAH analysis

❖ One of the difficulties associated with determination of PAHs in environmental samples is the complexity of PAH mixture in these samples. Even after extensive and rigorous clean-up, the PAH fraction may contain hundreds of compounds.

❖ Analytical methods that offer combinations of good chromatographic resolving power and detector selectivity are usually required to quantify selected compounds in such mixtures.

❖ There is essentially a three-step procedure for the analysis and determination of PAHs in environmental samples:
- ✓ extraction and isolation of PAHs from the sample matrix;
- ✓ (2) clean-up of the PAH mixtures from impurities and fractionation of PAH into subgroups; and
- ✓ (3) identification and quantitative determination of the individual components in each of these subgroups using GC-MS.

Sediment sampling

Freeze drying

Sohxlet extraction

Fractionation on column with SiO_2 and

Evaporation of eluate

GC-MS

Conditions of GC/MS

Injector	splitless
Injection volume	1 µl
Injector temp.	300°C
Velocity	1,0 $см^3$/min
Column	HP-5MS (30m, 0.25mm 0.25 µm)
Oven program	
First temp.	70°C, 1 min
Heating rate	10°C·min^{-1}
Isotherm	120°C, 1 min
Heating rate	7°C·min^{-1}
Isotherm	270°C, 10 min
MSD parameters	
Ionization	E+, 70 eV
method of data acquisition	SIM

❖ Data Analysis in order to identify individual PAHs (Figure 1 and corresponding mass spectra) and calculate diagnostic parameter for source identification (Table 1).

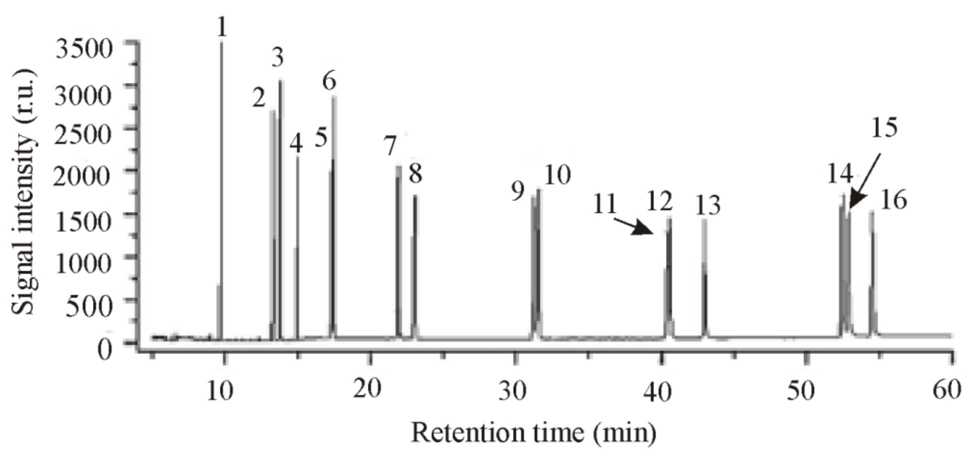
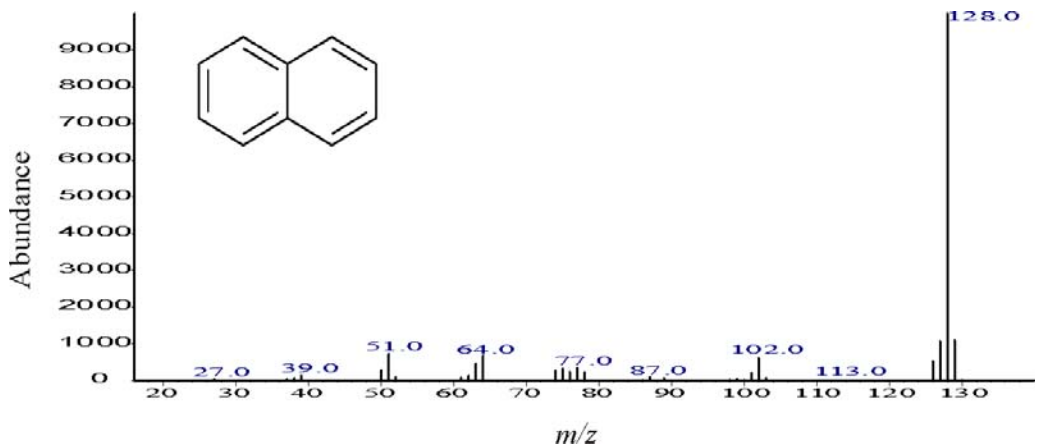


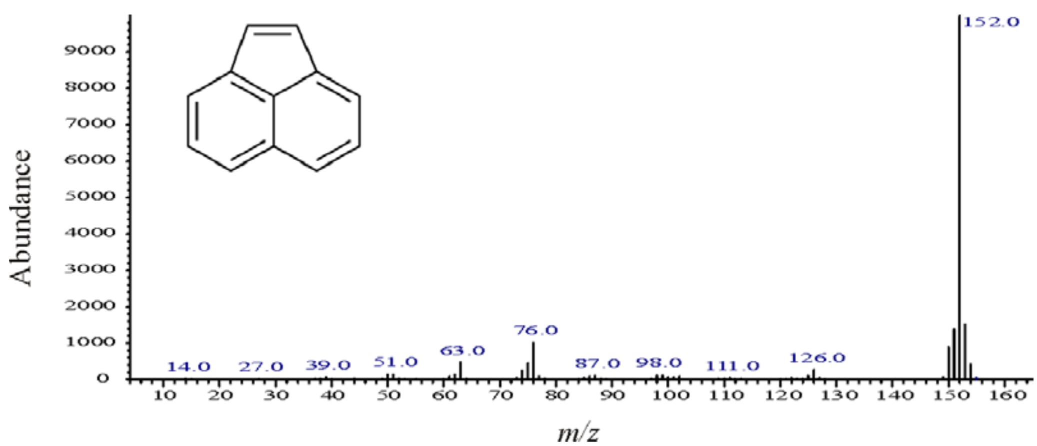
Figure 1. GC-MS chromatogram of a standard mixture of the sixteen EPA-PAHs.

❖ Mass spectra individual 16 PAHs from figure 1 are given below (source: library NIST05a).

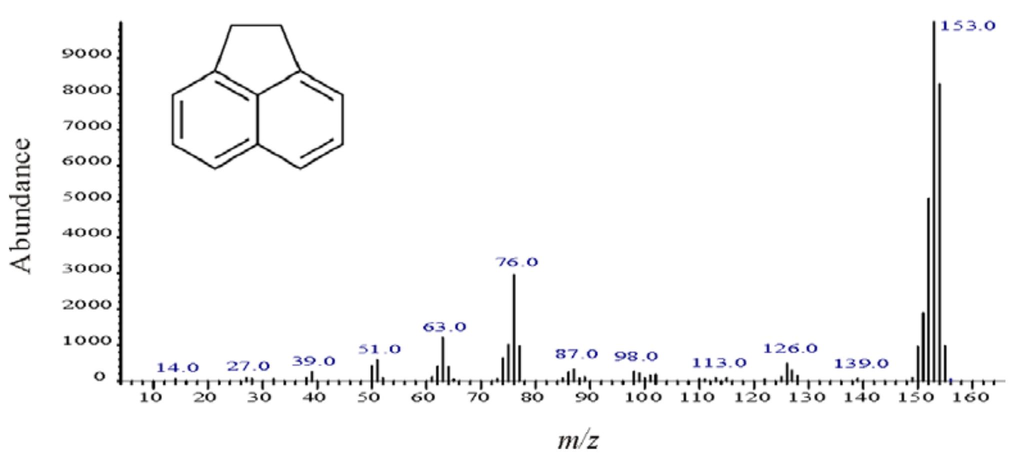
1. **Naphthalene (N)**



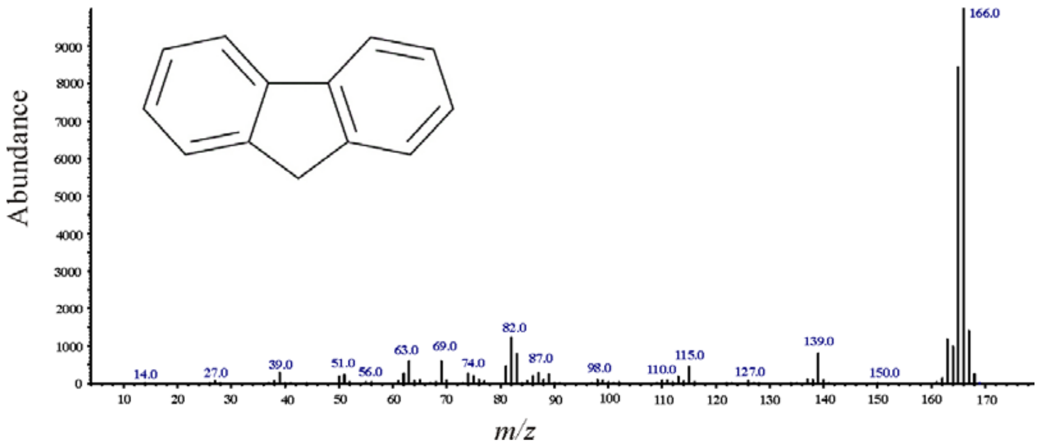
2. **Acenaphthylene (AY)**



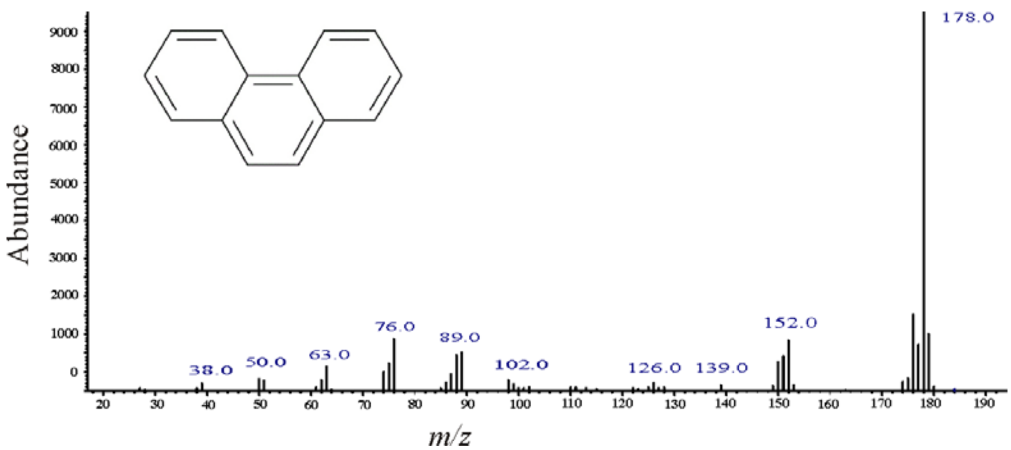
3. **Acenaphthene (AE)**



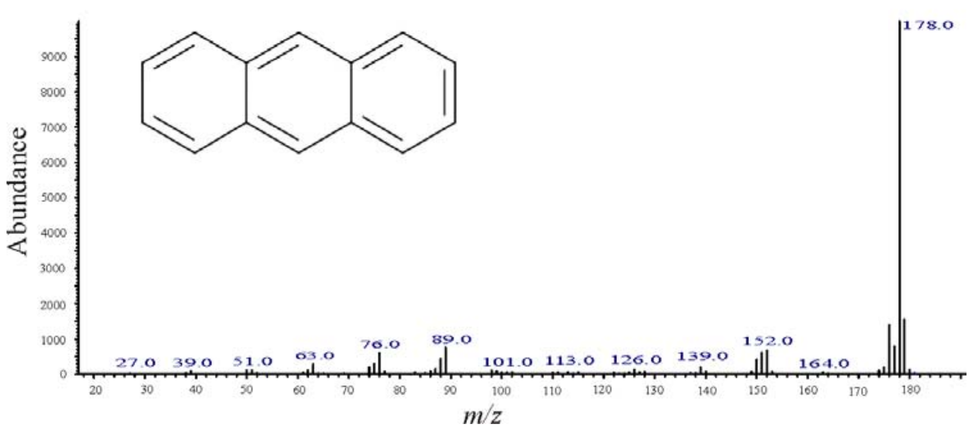
4. **Fluorene (F)**



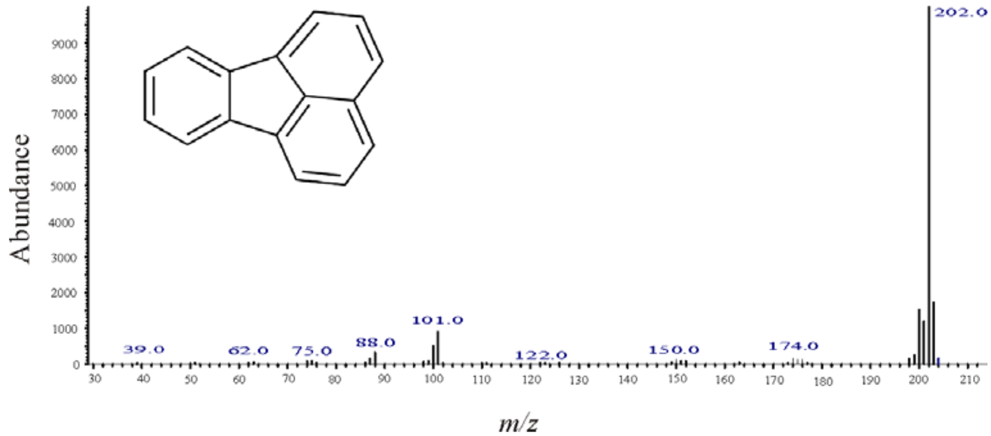
5. **Phenanthrene (P)**



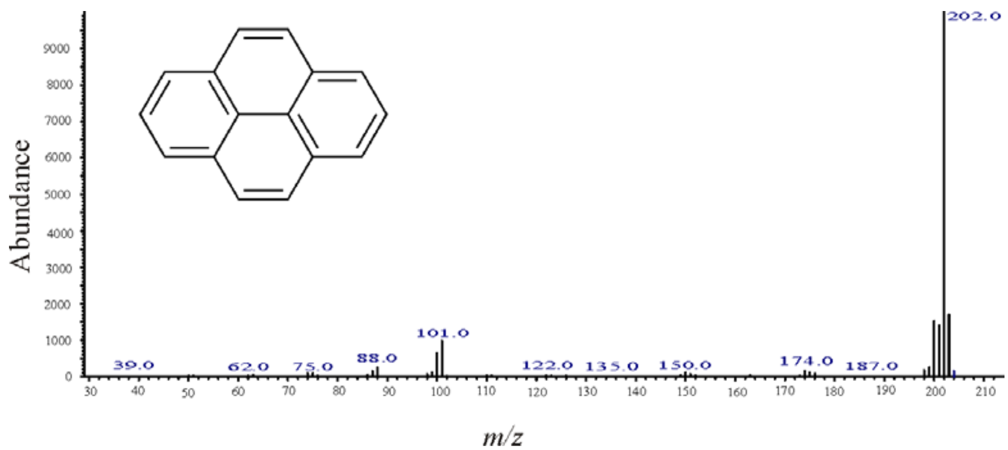
6. **Anthracene (A)**



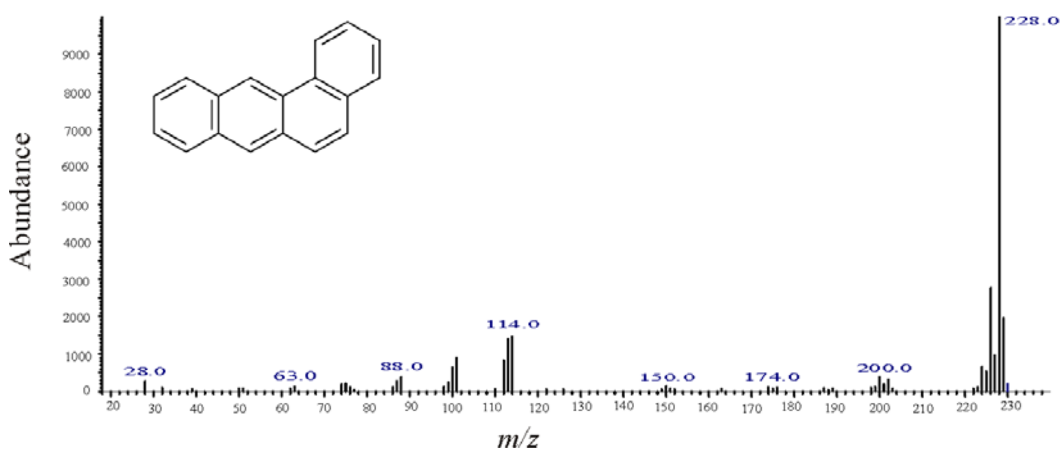
7. **Fluoranthene (FL)**



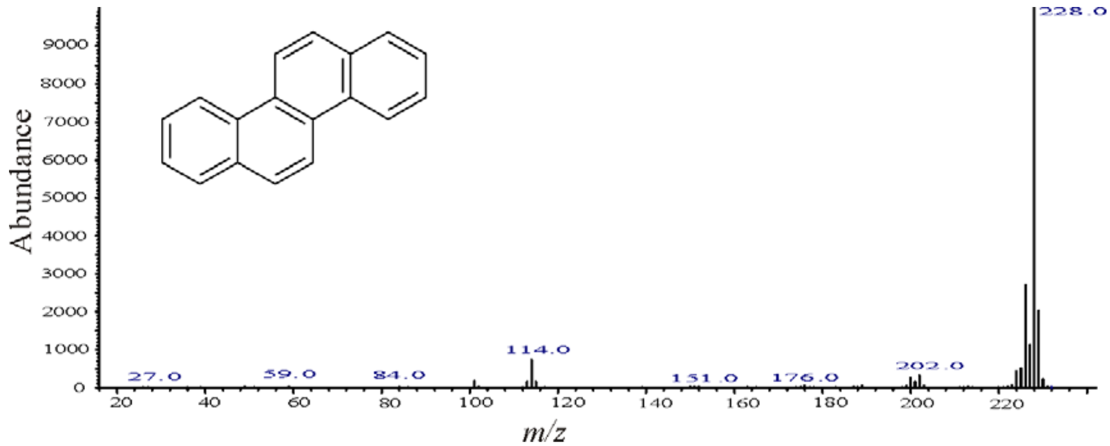
8. **Pyrene (PY)**



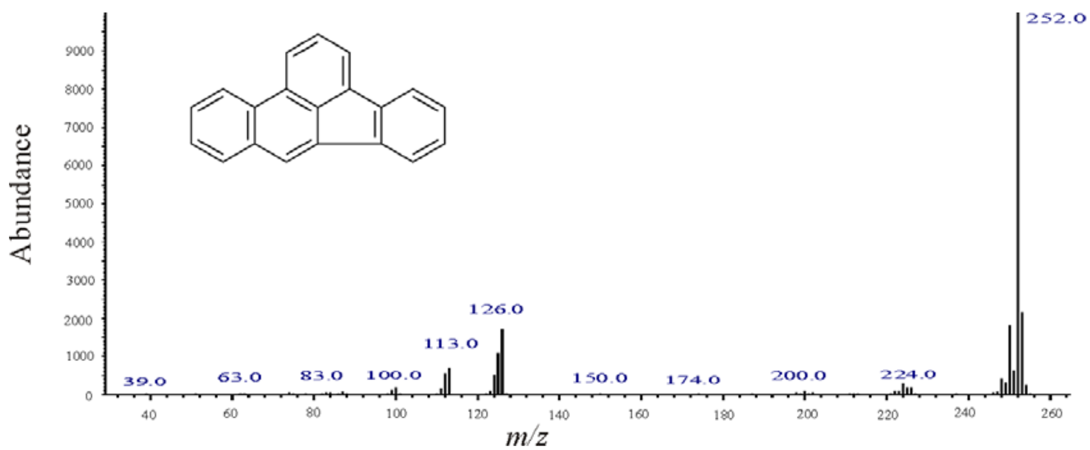
9. **Benz(a)anthracene (BaA)**



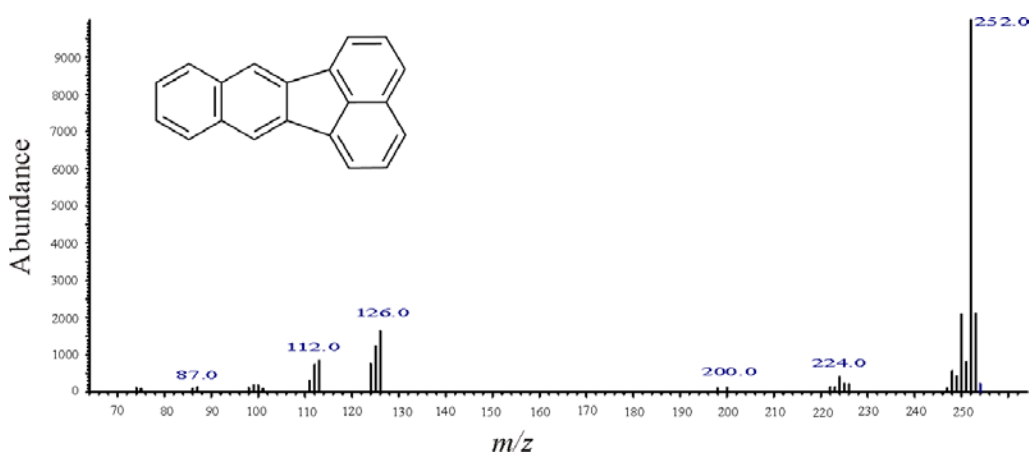
10. **Chrysene (C)**



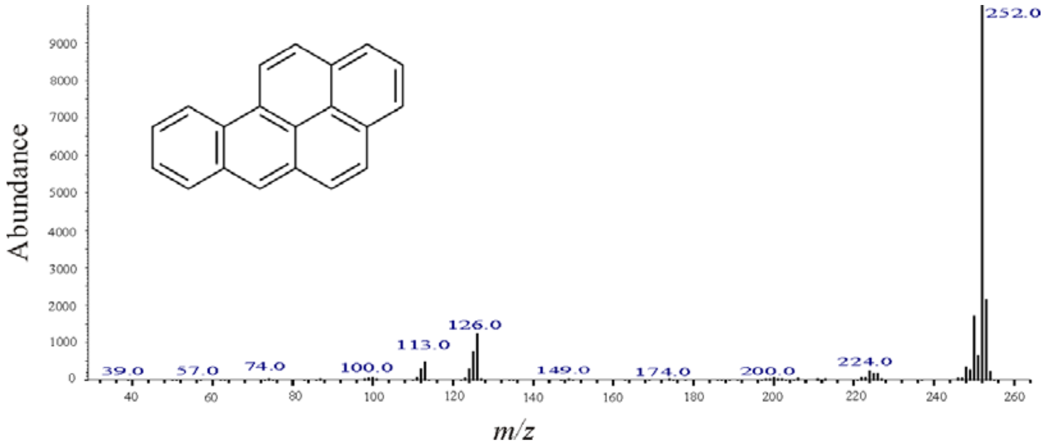
11. **Benzo(b)fluoranthene (BbF)**



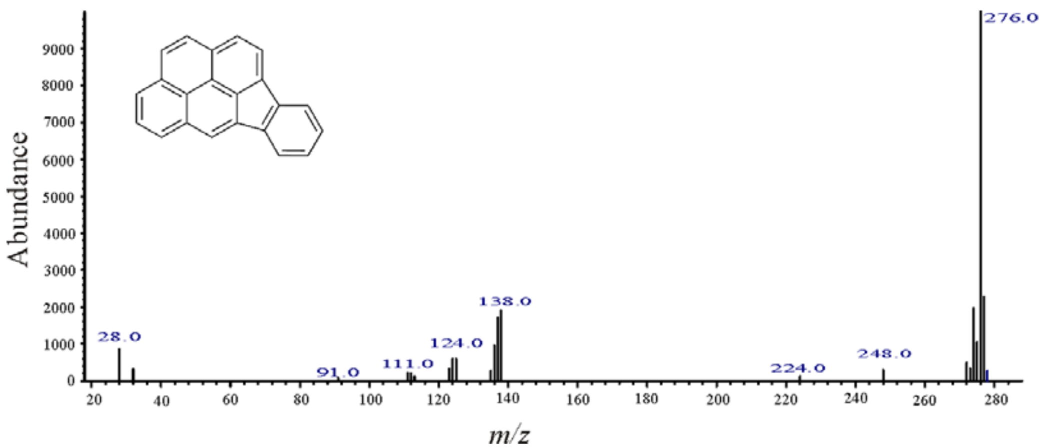
12. **Benzo(k)fluoranthene (BkF)**



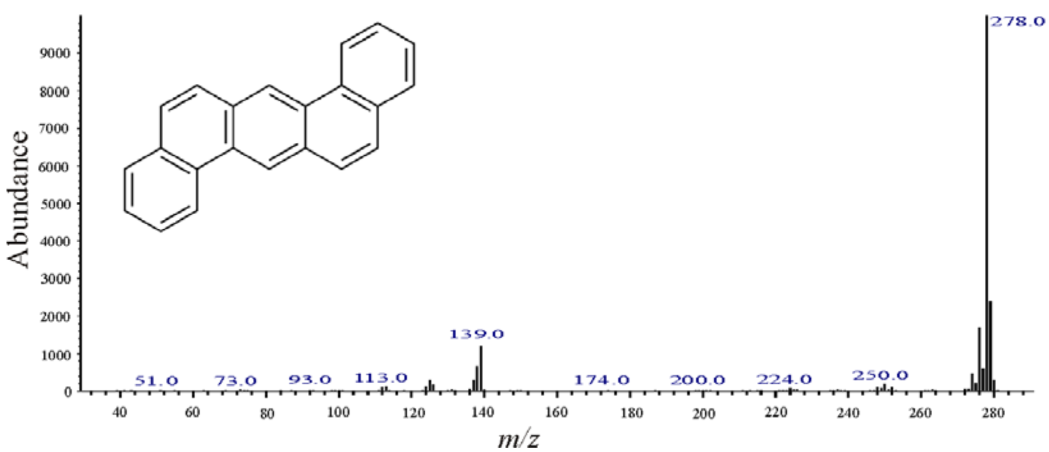
13. Benzo(a)pyrene (BaP)



14. Indeno(1,2,3-cd)pyrene (IP)



15. Dibenz(a,h)anthracene (DA)



16. **Benzo(g,h,i)perylene (BeP)**

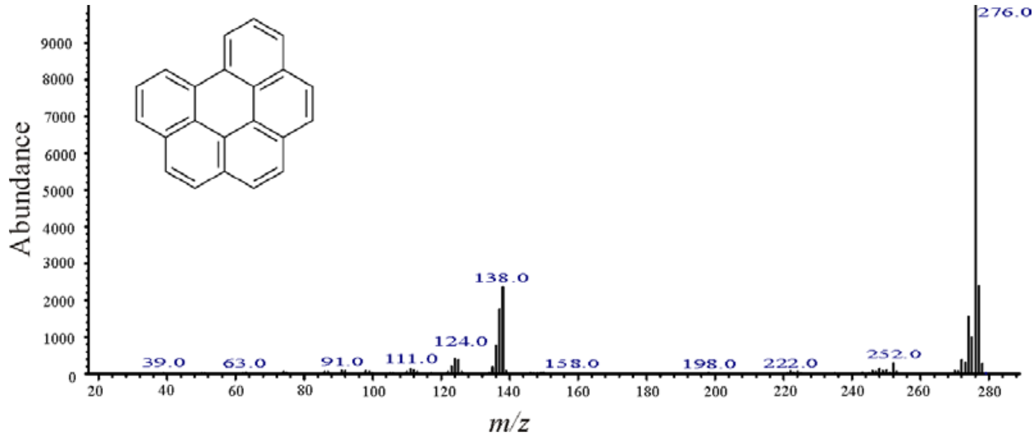


Table 1. Characteristic diagnostic ratios values for particular pollution emission sources

Diagnostic ratio	Petrogenic	Fuel combustion	Coal, grass, wood burning
A/(P+A)	< 0.1	> 0.1	-
FL/(FL+PY)	< 0.4	0.4-0.5	>0.5
BaA/(BaA+C)	< 0.2	> 0.35	0.2-0.35
IP/(IP+BeP)	< 0.2	0.2-0.5	>0.5

Remark

❖ PhD course Fuels does not include laboratory exercise, and does not require record of attendance. Students are provided with materials for exam preparation, and regular consultations with the professor.

Taking into account the above, this document provides a procedure for Remote access in order to familiarize the students with this possibility and explanations how this approach can help them in future work.

Remote Access Connection Instructions

❖ What makes these labs different and unique from other classroom experiments is that we have incorporated a section in each activity to remotely characterize your samples from your classroom.

❖ Request a remote lab session specifying information such as: the day, the time, and the instrument you are interested in using by visiting our web site:
http://netchem.ac.rs/remote-access

❖ You will see the list of partners with the instruments provided to chose from.

❖ You will be contacted by a Remote Access staff member to set up a test run to ensure you are set up properly and have the required infrastructure.

❖ Send samples or verify the in-house sample you would like us to prepare and load for characterization.

❖ Send your samples to the Remote Access center that you chose on your request.

❖ There are two communications soft-ware packages, that will allow us to communicate instructions and answer questions during the session.

❖ - TeamViewer: You can obtain a free download at:

- ✓ https://www.teamviewer.com/en/index.aspx
- ✓ Skype

❖ You will need:

- ✓ Computer with administrator access to install plug-ins and software
- ✓ Laptop
- ✓ An internet connection
- ✓ Speakers
- ✓ Microphone

- ✓ Projector connected to the same computer
- ✓ Web browser (Firefox preferred)

❖ During the test run you can refer to this guide to perform the following steps, but it's very important that you only proceed with these steps during your scheduled times. You may interfere with other remote sessions and potentially damage equipment if you log in at other times.

- ✓ Open and logon to your Zoom/Team-viewer account. You will be given the access code to enter at the time of your test and then again during the remote session.
  - ➢ If you are using the Zoom software, Remote Access staff will give you the access code.
  - ➢ If you are using the Team-viewer software, Remote Access staff will give you the ID & password.
- ✓ You should soon see the Remote Access desktop and at this point you can interact with the icons on the screen as if it were your desktop.
- ✓ Switch to full screen mode by selecting the maximize screen option in the top right corner of the screen.
- ✓ Upon completion of the session, move your mouse to the top right corner of the screen, and click on the X to disconnect the remote session. It will ask if you want to end the remote session. Click Yes.

Author, Editor and Referee References

This remote access lecture was created thanks to work done primarily at University of Belgrade.

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Refereeing of this material was done by: ______________________

Editing into NETCHEM Format and onto NETCHEM platform was completed by: _______________

References and Supplemental Material

The NETCHEM platform was established at the University of Nis in 2016-2019 through the Erasmus Programme.

Please contact a NETCHEM representatives at your institution or visit our website for an expanded contact list.

The work included had been led by the NETCHEM staff at your institution.