Data Article

Data of polycyclic aromatic hydrocarbons concentration in the Siberian Arctic seas sediments

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A B S T R A C T

Polycyclic Aromatic Hydrocarbons (PAHs) are one of the most dangerous persistent organic pollutants in the Arctic. They have different sources and pathways of entering into the environment. Because of their lipophilic properties, PAHs can easily accumulate in marine sediments. This work gives a new data about concentration of PAHs in Siberian arctic seas. Sixteen priority PAHs as well as 1- and 2-methyl-naphthalenes were analyzed by gas chromatography – tandem mass spectrometry in the twenty-four sediment samples taken from Kara, Laptev and East Siberian Seas in October 2020. The obtained sum concentrations ranged from 31 to 223 ng g⁻¹ with the greatest contribution of phenanthrene, benzo[b]fluoranthene, benzo[k]fluoranthene, as well as naphthalene and its methyl derivatives while the greatest PAH levels were observed in Laptev Sea. The toxic equivalent in benzo[a]pyrene units was from 2.2–18.2 ng g⁻¹. Total organic carbon (TOC) and black carbon (BC) content in arctic were in the ranges of 0.18–1.98 % and 0.03–0.40 %, re-
Specifications Table

| Subject                      | Pollution                                                       |
|------------------------------|-----------------------------------------------------------------|
| Specific subject area        | Pollution of marine sediments in the Siberian Arctic seas with highly toxic polycyclic aromatic hydrocarbons |
| Type of data                 | Table                                                           |
| How the data were acquired   | PAH concentrations were measured using pressurized liquid extraction (PLE) on an ASE 350 PLE system (Dionex, Sunnyvale, USA). Clean up was carried out in Pasteur pipette filled with activated copper and silica. Concentration was carried out in the presence of nitrogen using Reacti-Therm evaporator (Thermo scientific, Waltham, USA). The instrument used is Agilent 7890A gas chromatography system equipped with an Agilent 7000 MS/MS triple quadrupole mass spectrometry detector (Agilent, Santa Clara, USA) with triple quadrupole mass analyzer and autosampler. Mass Hunter software (Agilent, Santa Clara, USA) was used for data collection and processing. |
| Data format                  | Raw and analyzed data                                           |
| Description of data collection | Sediments samples were collected from the Kara, the Laptev, and the East Siberian seas. Sediment samples were collected using a van Veen grab. The top 0–2 cm layers of the grabbed sediments were carefully collected with a stainless-steel spatula which was washed with acetonitrile in advance. All samples were placed into solvent-rinsed air-tight glass containers, which were stored at −10°C until further analysis. Samples were collected from 24 stations roughly 12.5m to 308.5m water depth. The number of replicate samples - 1. The total organic carbon (TOC) and black carbon (BC) contents were determined using an EA3000 CHNS-analyzer (EuroVector, Pavia, Italy). |
| Data source location         | Institution: Core Facility Center “Arktika”, Lomonosov Northern (Arctic) Federal University |
|                             | City: Arkhangelsk                                               |
|                             | Country: Russia                                                 |
| Data accessibility           | Repository name: Mendeley Data                                  |
|                             | Data of polycyclic aromatic hydrocarbons concentration in the Siberian Arctic seas sediments |
|                             | Data identification number: 10.17632/h36jzyp7v8.1              |
|                             | Direct URL to data: https://data.mendeley.com/datasets/h36jzyp7v8/1 |
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|                             | https://10.1016/j.marpolbul.2022.113741                        |

Value of the Data

- These data are useful because they provide information about up to date situation of PAH concentrations in the Siberian arctic seas in hard-to-reach areas. In addition, they give information about source of PAHs.
- These data will be most helpful for researchers and ecologists interested in long-term monitoring of persistent organic pollution in the Arctic and for compartment with other regions.
• The data provide insights, which can be used to assess the risks associated with the presence of PAH in arctic marine sediments.
• The data provide detailed information on concentration levels of 18 PAH in 24 arctic marine sediment samples.

1. Data Description

The data set covers the results of study of 24 sediments samples from three arctic seas (the Kara, the Laptev and the East Siberian Seas). Marine sediment samples were collected using Van Veen grab sampler (Fig. 1)

Locations of sampling points are presented in Data file 1. We present data file in this article. Data file 1 (Excel spreadsheet) presents information about the average concentrations of PAH in 24 sediment samples, information about sampling points, limits of detection and quantification, recovery, total organic carbon and black carbon. PAHs concentration was determined by GC-MS/MS in MRM transition mode.

All concentrations are presented with standard deviation value calculated on the basis of two replicate analyses of each sediment sample. The main parameters of GC-MS/MS analysis are presented in Table 1.

![Fig. 1. Sampling usage a Van Veen grab.](image)

| Analyte               | Ion transition, m/z | Dwell | Collision energy, eV | Ion transition, m/z | Dwell | Collision energy, eV |
|-----------------------|---------------------|-------|----------------------|---------------------|-------|----------------------|
| Naphthalene           | 128.1               | 102.1 | 45                   | 128.1               | 78.1  | 45                   |
| 2-Methylnaphthalene   | 142.1               | 141.1 | 15                   | 141.1               | 115.1 | 15                   |
| 1-Methylnaphthalene   | 142                 | 141   | 15                   | 142                 | 115.1 | 15                   |
| Naphthalene-d8        | 136                 | 134   | 45                   | 136                 | 158   | 22                   |
| Acenaphthene          | 153.1               | 127   | 22                   | 152.1               | 126   | 22                   |
| Acenaphthylene        | 152.1               | 126   | 22                   | 151.1               | 77    | 22                   |
| Acenaphthylene-d8     | 160                 | 158   | 22                   | 158                 | 156   | 22                   |
| Fluorene              | 166                 | 165   | 30                   | 165                 | 164   | 30                   |
| Anthracene            | 178.1               | 152.1 | 25                   | 178.1               | 151.1 | 25                   |
| Phenanthrene          | 178.1               | 152.1 | 25                   | 176.1               | 150.1 | 25                   |

(continued on next page)
Table 1 (continued)

| Analyte                        | Ion transition, m/z | Dwell | Collision energy, eV | Ion transition, m/z | Dwell | Collision energy, eV |
|--------------------------------|---------------------|-------|----------------------|---------------------|-------|----------------------|
| Phenanthrene-d10               | 188.3               | 186.3 | 25                   | 15                  | 188.3 | 160.2               |
| Fluoranthene                   | 202.1               | 152.1 | 10                   | 30                  | 201.1 | 200.1               |
| Fluoranthene-d10               | 212                 | 208   | 10                   | 35                  | 210   | 208                 |
| Pyrene                         | 200.1               | 200   | 10                   | 25                  | 200.1 | 174                 |
| Pyrene-d10                     | 212                 | 208   | 10                   | 35                  | 210   | 208                 |
| Benz[a]anthracene              | 228.1               | 226.1 | 30                   | 30                  | 114   | 101.1               |
| Chrysene                       | 228.1               | 226.1 | 30                   | 30                  | 113.1 | 112.1               |
| Benzo[b]fluoranthene           | 252.1               | 250.1 | 50                   | 35                  | 126   | 113.1               |
| Benzo[k]fluoranthene           | 252                 | 250   | 50                   | 30                  | 126.1 | 125.1               |
| Benzo[a]pyrene                 | 252.1               | 250.1 | 60                   | 35                  | 125   | 124.1               |
| Benzo[a]pyrene-d12             | 264                 | 260   | 60                   | 35                  | 264   | 236                 |
| Dibenzo[a,h]anthracene         | 278.2               | 276.2 | 40                   | 35                  | 125   | 124.1               |
| Indeno[1,2,3-cd]pyrene          | 276.3               | 274.3 | 40                   | 40                  | 138.1 | 137.1               |
| Benzo[g,h,i]perylene           | 276.1               | 274.1 | 35                   | 45                  | 138   | 137                 |
| Benzo[g,h,i]perylene-d12       | 288                 | 284   | 40                   | 40                  | 144   | 142                 |

2. Experimental Design, Materials and Methods

Sediments sampling were carried out during the International Siberian Shelf Study Expedition (ISSSS-2020) on the research vessel “Akademik Keldysh” in October 2020. One sample was taken from each station. The samples were taken from 12.5 to 308.5 m depth. The top 0–2 cm layers of the grabbed sediments were carefully collected into solvent-rinsed air-tight glass containers, with a stainless-steel spatula which was washed with acetonitrile in advance. All samples were placed into solvent-rinsed air-tight glass containers, which were stored at −10°C until further analysis. Before analysis, samples were dried at 40 °C for 72 h, homogenized in a planetary ball mill and sieved through a 125 μm stainless steel sieve.

Two grams of dry sediment samples were extracted by pressurized liquid extraction (ASE 350, Dionex, Sunnyvale, USA) with a mixture of reagent grade (>99%, Panreac, Barcelona, Spain) dichloromethane (DCM) and acetone (3:1, v/v) at 175 °C and 10.5 MPa. The extraction was carried out in a 10 mL stainless steel cell in two cycles (5 min each) in static mode with flush volume percentage of 100% according to [1]. The obtained extracts were concentrated to 1 mL in a high-purity (> 99.99%) nitrogen stream using Reacti-Therm evaporator (Thermo scientific, Waltham, USA). The mixture of deuterated internal standards (acenaphthylene-d₈, benzo[ghi]perylene-d₁₂, fluoranthene-d₁₀, naphthalene-d₈, phenanthrene-d₁₀, pyrene-d₁₀) purchased from Sigma Aldrich (Steinheim, Germany) was added prior to extraction to obtain the final concentrations of 20 ng mL⁻¹ in analyzed solution. Clean up procedure was carried out in Pasteur pipette filled with activated copper and silica as described [2].

Silica gel was activated by heating at 180 °C for 8 h, copper filings were activated by a 7M HCl solution and consecutively rinsed with distilled water, acetone and n-hexane respectively, prior to use. Activated copper (1 cm) was placed at the base of the column to remove elemental sulfur. A silica gel suspension made up in n-hexane filled into pipette to a height of 5 cm. Sorbent was flushed with 20 mL of hexane/DCM mixture (3:1, v/v). Extracts were concentrated again in a nitrogen stream to 1 mL prior to analysis. Each sediment sample was prepared, extracted and analyzed in three repetitions with further averaging and calculation of standard deviations.

PAH standards were purchased from Supelco (Bellefonte, USA). A multi-component stock solution of 18 target PAHs in benzene/DCM mixture (1:1, v/v) containing 2000 μg mL⁻¹ of each analyte was used in quantification procedure for the obtaining the calibration solutions with concentrations 1–100 ng mL⁻¹ by consecutive dilutions with the solvent and addition of internal standards mixture to a concentration of 20 ng mL⁻¹.
Agilent 7890A gas chromatography system equipped with an Agilent 7000 MS/MS triple quadrupole mass spectrometry detector (Agilent, Santa Clara, USA) and autosampler was used for determination of PAHs. Agilent DB-XLB capillary column (30 m × 0.25 mm, 0.1μm film thickness) was used for chromatographic separation. The 1-μL samples were injected in pulsed split-less mode (injection pulse pressure 50 psi for 0.9 min, 30 mL min⁻¹ purge flow to split vent for 0.75 min) through an ultra-inert liner with a glass wool. The inlet temperature was 300 °C. Helium (6.0 grade) was used as the carrier gas in a constant pressure (25 psi) mode. The oven temperature program was as follows: 50 °C for 3 min, 40 °C min⁻¹ ramp to 150 °C, 15 °C min⁻¹ ramp to 280 °C (held for 2 min), 40 °C min⁻¹ ramp to 340 °C (held for 3 min). The total run time was 20.7 min. After each run 1 min backflush at 340 °C was used to clean the GC column from low volatile contaminants. The mass spectrometry detection was performed with electron ionization (70 eV) in multiple reaction monitoring (MRM) mode with MRM transitions taken from literature [3]. Collision energies were optimized for each MRM transition in automatic regime to achieve maximum signal intensity. The collision (nitrogen) and quenching (helium) gas flows were 1.5 mL min⁻¹. The transfer line was kept at 320 °C. The ion source and quadrupole analyzer temperatures were fixed at 320 °C and 150 °C, respectively.

Limits of detection (LODs) and quantification (LOQs) were calculated as the lowest concentrations of analytes having clear discerned peaks with signal-to-noise ratios (S/N) of 3 and 10, respectively. The obtained values were approximately 1 and 3 ng g⁻¹, respectively. Recoveries were estimated for all samples by comparison of chromatographic peak areas for the deuterated surrogate standards in extracts and calibration solution. The obtained mean recoveries ranged from 50 to 75% (lower values in this range are characteristic of PAHs with lowest molecular weights, e.g. naphthalene). Quantitative analysis was performed by internal standard calibration method. Each deuterated internal standard was used for quantification of analytes closest to it in retention times. If the determined concentrations were below LOQ, the values of ½ LOQ were assigned for calculation of total sum of PAHs (Σ18PAHs). Since two analytes - B[bb]F and B[k]F - were not chromatographically separated, they were quantified as a sum.

Total organic carbon and black carbon were analyzed according to the method [4]. For removal of organic carbon, 50 mg of each sample was weighed into 2 ml glass vial. Samples were oxidized at 375 °C for 24 h in the presence of air. For removal of inorganic carbon to the cooled samples 50 μl of water and 50 μl of 1 M HCl were added. Then samples were acidified for 1 h at room temperature. After cooling another 50 μl of 1 M HCl was added, followed by 30 min of cooling. Vials were dried at 60 °C in an oven. Steps were repeated until complete removal of carbonates. Approximately 100 μg was taken for the analysis on an EA3000 CHNS-analyzer (EuroVector, Pavia, Italy)

**Ethics Statement**

The authors declare that there are no ethical issues with the data presented and the methods used.

**CRediT Author Statement**

**Dmitry Lakhmanov**: Investigation, Writing – original draft; **Aleksandr Kozhevnikov**: Formal analysis, Resources; **Sergey Pokrishkin**: Software, Validation; **Igor Semiletov**: Funding acquisition; **Dmitry Kosyakov**: Project administration, Funding acquisition.

**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.
Data Availability

Data of polycyclic aromatic hydrocarbons concentration in the Siberian Arctic seas sediments (Original data) (Mendeley Data).

Acknowledgments

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