Data Article

Dataset of PAHs determined in home-made honey samples collected in Central Italy by means of DLLME-GC-MS and cluster analysis for studying the source apportionment

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A R T I C L E   I N F O

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Dataset link: Raw chromatogram files of honey samples analyzed by GC-MS/QqQ (Original data)

A B S T R A C T

This paper would like to show all the data related to an intensive field campaign focused on the characterization of the Polycyclic Aromatic Hydrocarbons (PAHs) composition profile in almost 60 honey samples collected in Central Italy. The analytical data here reported are the base for a study aimed to identify the pollution sources in a region. 22 PAHs were analyzed by means of ultrasound-vortex-assisted dispersive liquid-liquid micro-extraction (DLLME) procedure followed by a triple quadrupole gas chromatograph/mass spectrometer (GC-MS). A chemometrics approach has been carried out
Specifications Table

Subject: Food Science: Food Chemistry
          Analytical Chemistry
          Environmental Chemistry
          Pollution

Specific subject area: Chromatography, food quality, air quality monitoring, source apportionment

Type of data: Tables
          Figures
          Chromatograms

How the data were acquired: The data were acquired by a triple quadrupole gas chromatograph/mass spectrometer.

Data format: Raw
          Analyzed
          Filtered

Description of data collection: The honey samples were processed by an extraction protocol based on the ultrasound-vortex-assisted dispersive liquid-liquid micro-extraction (DLLME) procedure followed by a gas chromatography coupled with a triple quadrupole mass spectrometry. The analyses were carried out using a standard solution of perdeuterated PAH compounds.

Data source location: All the samples were collected in Central Italy, regions Latium and Molise; the analyses were carried out at the laboratories of the Institute of Atmospheric Pollution Research (IIA), National Research Council (CNR), in Rome. Belgrade and Serbia database was available in literature.

Data accessibility: The dataset is available on this article and can be found in Mendeley repository data:
          Passarella, Sergio; Guerriero, Ettore; Quici, Luisangela; Ianiri, Giuseppe; Cerasa, Marina; Notardonato, Ivan; Protano, Carmela; Vitali, Matteo; Russo, Mario V.; De Cristofaro, Antonio; Avino, Pasquale (2022), “Raw chromatogram files of honey samples analyzed by GC-MS/QqQ”, Mendeley Data, V1, doi: 10.17632/kn72vrxx7y71
          https://data.mendeley.com/datasets/kn72vrxx7y71

Related research article:
For an article which has been submitted:
          S. Passarella, E. Guerriero, L. Quici, G. Ianiri, M. Cerasa, I. Notardonato, C. Protano, M. Vitali, M.V. Russo, A. De Cristofaro, P. Avino, PAH source apportionment in home-made honey samples collected in Central Italy by means of chemometric approach, Food Chem. 382 (2022) 132361. doi: 10.1016/j.foodchem.2022.132361
          https://www.sciencedirect.com/science/article/abs/pii/S0308814622003235?via%3Dihub

Value of the Data

- The analytical procedure reported allows to investigate PAHs by perdeuterated compounds and DLLME-GC-MS analysis at trace levels
- Honey samples can be considered as a biomonitoring index in anthropogenic or natural areas, avoiding long and tedious sampling procedures
- Data can be useful for source apportionment of PAHs in relationship to different emissions for air quality studies
• Data can be used by other scientists for different chemometrics analysis in the food quality study

1. Data Description

The dataset reported here is related to the analytical procedure set up for analyzing 22 polycyclic aromatic hydrocarbons (PAHs) (Table 1) in honey samples adapted from Kazacic et al. [1].

The raw files of the gas chromatography coupled with mass spectrometry (GC-MS) data are available in a dedicated repository: all the chromatograms are deposited in the Mendeley one [2]. It should be noted that in the repository 62 chromatograms are deposited: the difference, i.e. 5 chromatograms, is due to samples #2997 and #2998 whose chromatographic runs were repeated three times, and to a toluene chromatogram reported (for checking the column clearness).

Under such analytical conditions 57 home-made honey samples were analyzed. For a preliminary analysis of the relations among PAHs, the Pearson’s correlation was performed: Table 2 shows the main correlations between PAHs with R above 0.6.

The simultaneous presence of 22 PAHs and 57 samples generates a problem of multivariate analysis. Before running the chemometric approach, the analysis of variance (ANOVA) was carried out by SPSS statistics software for Windows, version 25.0 (IBM Corp., Armonk, NY, USA). The results show what compounds with high concentration variability (i.e., high relative standard deviation, RSD) record high square mean values, and a significance value (or α level) equal to zero (< 0.01), i.e. BaA, BeP, Bb+jF, BghiP, BkF, IPy, Chr, BaP, DahA, Phe, DalP, DaiP, DahP, DaeP, Per. The main consideration regards the role of molecules at the highest molecular weight. In fact, this occurrence is responsible for the sample distribution in different clusters. The Cluster Analysis (CA), performed by means of SPSS software and based on non-hierarchical (k-means) technique, meaning that the grouping is built on Euclidean distance, was applied for determin-

| PAH                  | Acronym |
|---------------------|---------|
| Acenaphtene         | Acy     |
| Acenaphthylene      | Ace     |
| Anthracene          | Ant     |
| Benzo[a]anthracene  | BaA     |
| Benzo[a]pyrene      | Bb+jF   |
| Benzo[b+j]fluoranthene | BkF   |
| Benzo[e]pyrene      | BghiP   |
| Benzo[ghi]perylen   | BaP     |
| Benzo[k]fluoranthene| BeP     |
| Chrysene            | Chr     |
| Dibenzo[a,e]pyrene  | DahA    |
| Dibenzo[a,h]anthracene | DalP |
| Dibenzo[a,h]pyrene  | DaeP    |
| Dibenzo[a,i]pyrene  | DaiP    |
| Dibenzo[a,l]pyrene  | DahP    |
| Fluoranthene        | Fu      |
| Fluorene            | Fl      |
| Indeno[1,2,3-cd]pyrene | IPy |
| Naphthalene         | Na      |
| Perylene            | Phe     |
| Phenanthrene        | Py      |
| Pyrene              | Per     |

Table 1
List of the PAHs investigated and the related acronyms.
Table 2
Main correlations between PAHs, showing an R above 0.6. For acronyms: see Table 1. Table adapted from ref. [3].

| Correlations between 0.6-0.7 | Correlations between 0.7-0.8 | Correlations between 0.8-0.9 | Correlations > 0.9 |
|-----------------------------|-----------------------------|-----------------------------|--------------------|
| Ace-Acy                     | Fl-Acy                      | BeP-BkF                    | B(b+J)F-Chr        |
| Ant-Phe                     | Fl-Ace                      | BaP-Chr                    | lPy-B(b+J)F        |
| Py-Fu                       | BaP-B(b+J)F                 | DahA-BaA                   | BghiP-B(b+J)F      |
| BaA-Phe                     | lPy-Chr                     | IPy-B(b+J)F                | DahA-IPy           |
| B(b+J)F-Fu                  | lPy-BaP                     | BghiP-Chr                  | DahP-DaIP          |
| BaP-BkF                     | IPy-Per                     | DahP-Chr                   | DahP-DaIP          |
| Per-BaP                     | DahP-Per                    | DaeP-DaIP                  | DahP-DaIP          |
| DahA-Phe                    | BghiP-IPy                   | DahP-DaeP                  | DahP-DaeP          |
| DahA-Fu                     | DahP-DaeP                   | DahP-DalP                  | DahP-DalP          |
| DaeP-DahA                   |                             |                             |                    |

Table 3
Distance among center clusters.

| Cluster | 1     | 2     | 3     | 4     |
|---------|-------|-------|-------|-------|
| 1       | 60.21 | 63.54 | 58.04 |
| 2       | 60.21 | 27.53 | 27.60 |
| 3       | 63.54 | 27.53 | 27.04 |
| 4       | 58.04 | 27.60 | 27.04 |

Table 4
Number of samples (#) in each cluster.

| Cluster | # Sample | Quote % |
|---------|---------|---------|
| 1       | 1       | 2.0     |
| 2       | 3       | 5.9     |
| 3       | 41      | 80.4    |
| 4       | 6       | 11.8    |
| valid   | 51      | 100.0   |

ing the possible grouping among the honey samples [4]. First, 4 clusters were identified. Table 3 shows distance among center clusters: the greater the distance between the final centers of the clusters, the greater their dissimilarity.

On the other hand, Table 4 reports the number of samples in each cluster. It can be noted that cluster 1 is characterized by 1 sample (#41), cluster 2 by 3 samples (#32, #49, #50) and cluster 4 by 6 samples (#15, #19, #24, #25, #47, #53) whereas the cluster 3 is the most abundant containing 41 samples.

Tables 5–7 show the statistical data (in terms of mean, min, max values, standard deviation, RSD and 95 percentile) of each cluster (except for cluster 1).

A Principal Component Analysis (PCA) was applied for identifying the similarities among different datasets [5,6]. The chemometrics approach was carried out by open-access software, i.e. Tanagra [7]: the only condition considered was to have a dataset made of the same compounds. Following this statement, 15 PAHs were considered for the chemometrics treatment. The authors performed the PCA overall three datasets. In details, Table 8 shows the PCA applied to all the samples (i.e., 135 samples, divided in 51 from this study, in 61 from Serbia area [8] and in 23 from Belgrade area [9]).
Table 5  
Minimum, maximum and mean value (expressed as ng g⁻¹) along with sd, RSD% and 95 percentile (ng g⁻¹) of each PAH in cluster 2.

| PAH                     | # sample | Min     | Max     | Mean    | sd      | RSD%   | 95 percentile |
|-------------------------|----------|---------|---------|---------|---------|--------|---------------|
| Acenaphthene            | 3        | 0.021   | 0.17    | 0.10    | 0.08    | 75.7   | 0.17          |
| Acenaphthylene          | 3        | 0.056   | 0.20    | 0.13    | 0.07    | 55.9   | 0.19          |
| Anthracene              | 3        | 0.071   | 0.14    | 0.10    | 0.04    | 37.6   | 0.14          |
| Benzo[a]anthracene      | 3        | 0.023   | 0.63    | 0.40    | 0.33    | 82.3   | 0.62          |
| Benzo[a]pyrene          | 3        | 0.000   | 0.57    | 0.35    | 0.30    | 87.9   | 0.56          |
| Benzo[b+j]fluoranthene  | 3        | 0.000   | 0.35    | 0.21    | 0.18    | 88.3   | 0.34          |
| Benzo[e]pyrene          | 3        | 0.457   | 3.15    | 2.22    | 1.52    | 68.8   | 3.14          |
| Benzo[ghi]perylene      | 3        | 0.006   | 0.51    | 0.18    | 0.29    | 161.6  | 0.46          |
| Benzo[k]fluoranthene    | 3        | 0.787   | 1.82    | 1.38    | 0.53    | 38.5   | 1.79          |
| Chrysene                | 3        | 0.000   | 0.03    | 0.02    | 0.02    | 91.6   | 0.03          |
| Dibenzo[a,e]pyrene      | 3        | 0.009   | 0.73    | 0.25    | 0.42    | 165.4  | 0.66          |
| Dibenzo[a,h]anthracene  | 3        | 0.007   | 0.55    | 0.19    | 0.31    | 165.6  | 0.49          |
| Dibenzo[a,h]pyrene      | 3        | 0.001   | 1.00    | 0.34    | 0.57    | 171.5  | 0.90          |
| Dibenzo[a,i]pyrene      | 3        | 0.001   | 1.00    | 0.34    | 0.57    | 171.5  | 0.90          |
| Dibenzo[a,l]pyrene      | 3        | 0.000   | 1.00    | 0.34    | 0.57    | 170.6  | 0.90          |
| Fluoranthene            | 3        | 0.188   | 0.27    | 0.22    | 0.04    | 19.6   | 0.26          |
| Fluorene                | 3        | 0.034   | 0.35    | 0.19    | 0.16    | 84.2   | 0.34          |
| Indeno[1,2,3-cd]pyrene  | 3        | 0.003   | 0.52    | 0.33    | 0.28    | 86.1   | 0.51          |
| Naphthalene             | 3        | 0.026   | 0.69    | 0.30    | 0.35    | 116.4  | 0.64          |
| Perylene                | 3        | 0.000   | 0.64    | 0.21    | 0.36    | 169.6  | 0.57          |
| Phenanthrene            | 3        | 1.081   | 1.41    | 1.21    | 0.17    | 14.1   | 1.38          |
| Pyrene                  | 3        | 0.246   | 0.36    | 0.29    | 0.06    | 20.8   | 0.35          |

Table 6  
Minimum, maximum and mean value (expressed as ng g⁻¹) along with sd, RSD% and 95 percentile (ng g⁻¹) of each PAH in cluster 3.

| PAH                     | # sample | Min     | Max     | Mean    | sd      | RSD%   | 95 percentile |
|-------------------------|----------|---------|---------|---------|---------|--------|---------------|
| Acenaphthene            | 41       | 0.00    | 1.70    | 0.40    | 0.39    | 96.9   | 1.21          |
| Acenaphthylene          | 41       | 0.03    | 2.84    | 0.71    | 0.68    | 95.2   | 1.81          |
| Anthracene              | 41       | 0.02    | 0.14    | 0.08    | 0.03    | 37.9   | 0.13          |
| Benzo[a]anthracene      | 41       | 0.00    | 0.86    | 0.12    | 0.19    | 159.4  | 0.52          |
| Benzo[a]pyrene          | 41       | 0.00    | 0.25    | 0.14    | 0.11    | 130.3  | 0.12          |
| Benzo[b+j]fluoranthene  | 41       | 0.00    | 0.58    | 0.13    | 0.11    | 87.4   | 0.31          |
| Benzo[e]pyrene          | 41       | 0.01    | 0.62    | 0.11    | 0.14    | 127.8  | 0.45          |
| Benzo[ghi]perylene      | 41       | 0.00    | 0.42    | 0.07    | 0.10    | 140.6  | 0.26          |
| Benzo[k]fluoranthene    | 41       | 0.00    | 0.90    | 0.07    | 0.14    | 210.2  | 0.18          |
| Chrysene                | 41       | 0.01    | 0.46    | 0.09    | 0.09    | 98.0   | 0.25          |
| Dibenzo[a,e]pyrene      | 41       | 0.00    | 0.07    | 0.01    | 0.01    | 178.4  | 0.04          |
| Dibenzo[a,h]anthracene  | 41       | 0.00    | 0.22    | 0.02    | 0.04    | 209.7  | 0.09          |
| Dibenzo[a,h]pyrene      | 41       | 0.00    | 0.04    | 0.00    | 0.01    | 171.3  | 0.01          |
| Dibenzo[a,i]pyrene      | 41       | 0.00    | 0.04    | 0.00    | 0.01    | 171.3  | 0.01          |
| Dibenzo[a,l]pyrene      | 41       | 0.00    | 0.09    | 0.01    | 0.01    | 212.6  | 0.01          |
| Fluoranthene            | 41       | 0.12    | 0.44    | 0.22    | 0.06    | 27.8   | 0.30          |
| Fluorene                | 41       | 0.03    | 2.06    | 0.65    | 0.51    | 78.8   | 1.47          |
| Indeno[1,2,3-cd]pyrene  | 41       | 0.00    | 0.31    | 0.05    | 0.07    | 137.1  | 0.18          |
| Naphthalene             | 41       | 0.00    | 1.89    | 0.26    | 0.32    | 122.8  | 0.64          |
| Perylene                | 41       | 0.00    | 0.03    | 0.01    | 0.01    | 131.2  | 0.03          |
| Phenanthrene            | 41       | 0.70    | 1.49    | 1.09    | 0.18    | 16.4   | 1.46          |
| Pyrene                  | 41       | 0.19    | 0.86    | 0.45    | 0.19    | 41.6   | 0.78          |
Table 7
Minimum, maximum and mean value (expressed as ng g⁻¹) along with sd, RSD% and 95 percentile (ng g⁻¹) of each PAH in cluster 4.

| PAH               | # sample | Min     | Max     | Mean    | sd      | RSD%   | 95 percentile |
|-------------------|----------|---------|---------|---------|---------|--------|---------------|
| Acenaphthene      | 6        | 0.013   | 0.507   | 0.297   | 0.195   | 65.6   | 0.496         |
| Acenaphthylene    | 6        | 0.030   | 2.272   | 0.826   | 0.757   | 91.6   | 1.903         |
| Anthracene        | 6        | 0.028   | 0.111   | 0.064   | 0.034   | 52.1   | 0.109         |
| Benzo[a]anthracene| 6        | 0.069   | 0.975   | 0.592   | 0.402   | 67.9   | 0.953         |
| Benzo[a]pyrene    | 6        | 0.410   | 1.130   | 0.743   | 0.264   | 35.5   | 1.072         |
| Benzo[b+j]fluoranthe| 6   | 1.017   | 1.691   | 1.371   | 0.256   | 18.6   | 1.680         |
| Benzo[e]pyrene    | 6        | 0.584   | 1.193   | 0.884   | 0.204   | 23.0   | 1.147         |
| Benzo[ghi]perylen| 6        | 0.833   | 1.963   | 1.361   | 0.428   | 31.4   | 1.874         |
| Benzo[k]fluoranthe| 6        | 0.638   | 1.134   | 0.863   | 0.166   | 19.2   | 1.081         |
| Chrysenene        | 6        | 0.615   | 1.387   | 1.079   | 0.281   | 26.0   | 1.372         |
| Dibenzo[a,e]pyrene| 6        | 0.074   | 0.180   | 0.130   | 0.046   | 35.3   | 0.178         |
| Dibenzo[a,h]anthracene| 6   | 0.082   | 0.197   | 0.138   | 0.039   | 27.8   | 0.188         |
| Dibenzo[a,i]pyrene| 6        | 0.029   | 0.118   | 0.054   | 0.037   | 68.1   | 0.109         |
| Dibenzo[a,h]pyrene| 6        | 0.029   | 0.118   | 0.054   | 0.037   | 68.1   | 0.109         |
| Dibenzo[a,i]pyrene| 6        | 0.032   | 0.118   | 0.069   | 0.033   | 48.5   | 0.110         |
| Indeno[1,2,3-cd]pyrene| 6   | 0.660   | 1.434   | 1.039   | 0.292   | 28.1   | 1.386         |
| Fluoranthe| 6        | 0.183   | 0.524   | 0.334   | 0.115   | 34.5   | 0.488         |
| Fluorene          | 6        | 0.024   | 0.731   | 0.428   | 0.251   | 58.6   | 0.696         |
| Naphthalene       | 6        | 0.007   | 1.565   | 0.420   | 0.625   | 148.6  | 1.358         |
| Perylene          | 6        | 0.026   | 0.220   | 0.127   | 0.084   | 66.3   | 0.217         |
| Phenanthrene      | 6        | 0.856   | 1.386   | 1.095   | 0.175   | 15.9   | 1.326         |
| Pyrene            | 6        | 0.392   | 1.179   | 0.626   | 0.298   | 47.6   | 1.069         |

Table 8
PCA of all the samples investigated in this study along with the data collected in other papers [8,9].

| Component | Total | Variance % | Cumulative % | Extraction Sums of Squared Loadings |
|-----------|-------|------------|--------------|------------------------------------|
|           |       | Total      | Variance %   | Cumulative %                       |
| 1         | 8.865 | 59.100     | 59.100       | 8.865                              |
| 2         | 2.288 | 15.254     | 74.354       | 2.288                              |
| 3         | 1.425 | 9.501      | 83.855       | 1.425                              |
| 4         | 0.920 | 6.131      | 89.986       |                                    |
| 5         | 0.835 | 5.566      | 95.552       |                                    |
| 6         | 0.372 | 2.481      | 98.033       |                                    |
| 7         | 0.121 | 0.808      | 98.840       |                                    |
| 8         | 0.113 | 0.755      | 99.596       |                                    |
| 9         | 0.030 | 0.199      | 99.794       |                                    |
| 10        | 0.023 | 0.153      | 99.948       |                                    |
| 11        | 0.005 | 0.037      | 99.984       |                                    |
| 12        | 0.001 | 0.009      | 99.993       |                                    |
| 13        | 0.001 | 0.004      | 99.997       |                                    |
| 14        | 0.000 | 0.002      | 99.999       |                                    |
| 15        | 0.000 | 0.001      | 100.000      |                                    |

Fig. 1, obtained applying the PCA to all the common PAHs, shows the 3-D PCA-plot for identifying the different relevant contribution of each one whereas the Fig. 2 shows the PCA biplot applied to all the samples investigated in the three studies, using the two principal components.
Fig. 1. 3-D PCA-plot for each PAH using all the data. For acronyms: see Table 1. Figure modified from ref. [3].

Fig. 2. PCA score plot of the samples collected both in Belgrade and in Serbia and in Central Italy (this study) ("sample": data from Serbia area [8]; #"sample": this study; h"sample": data Belgrade area [9]). For acronyms: see Table 1. Figure modified from ref. [3].

2. Experimental Design, Materials and Methods

2.1. Honey sample collection

The analysis involved 57 home-made honey samples from different geographical locations in Central Italy. The sampling was carried out in maritime, hilly and mountainous areas; the samples were directly collected in the apiaries by local experts in the period from May to July. The samples were collected in different locations reported in Fig. 3; in each sampling site 5 samples were withdrawn every 15 days. For a better understanding of the PAH behavior in terms
of distribution and contamination, a comparison with other data present in literature was carried out. In particular, two papers were considered: they report a dataset of PAHs determined in honey samples collected in Serbia [8] and Belgrade [9] areas. These are the only papers showing a complete PAH profile.

2.2. DLLME procedure

The extraction was carried out by DLLME procedure [10]. 10 μL of the extraction standard solution of perdeuterated PAHs (10 ng μL⁻¹, L 429 IS, Wellington Laboratories) were added to 2.5 g of honey sample in acetone solution before shaking by vortexing for 40 seconds to favor the dissolution of the sample. The microextraction was performed using 150 μL of chloroform and the extraction process was favored by the formation of a macroemulsion by vortexing for 5 min and then by the formation of a microemulsion with the aid of an ultrasonic bath for 6 min. Subsequently, in order to facilitate the breaking of the emulsion and the recovery of the solvent, 10 g L⁻¹ of NaCl were added and then centrifuged at 4000 rpm for 30 minutes [11]: after, 1 μL was injected.

2.3. PAHs analysis by GC-MS

The instrumental analyses were performed by a triple quadrupole gas chromatograph/mass spectrometer (GC-MS) (Trace 1310 GC/TSQ 8000 Evo) (Thermo Fisher Scientific, Waltham, MA, USA) in electronic impact (EI) mode and the chromatographic separation was performed by a DB-XLB column (60 m × 0.25 mm, 0.25 μm I.D.) (Agilent Technologies, Santa Clara, CA, USA) with H₂ 3.00 mL min⁻¹ as the carrier gas. The PTV splitless injector was maintained at a constant temperature of 250 °C, mass transfer line temperature of 290°C and ion source temperature of 300°C. The oven was held at 60 °C for 1 min, then warmed 20 °C min⁻¹ until 200°C was reached, and held for 0 min, after was warmed at 7.0°C min⁻¹ until 275°C and held for 7 min, finally it was warmed at 18°C min⁻¹ until 325°C and held for 13 min. The analysis was performed in Selected Ion monitoring (SIM) and full scan mode: SIM time 0.215 s, full scan mode time 0.083 s and total scan mode time 0.300 s.

The first three components (Table 8), chosen because the eigenvalue is above 1 (Fig. 4) [12], describe 84 % of the whole dataset.
Fig. 4. Scree graph of the entire dataset, reporting the eigenvalue.

CRediT Author Statement

Pasquale Avino: Conceptualization; Giuseppe Ianiri, Ivan Notardonato: Investigation; Ettore Guerriero, Marina Cerasa: Formal analysis; Luisangela Quici, Sergio Passerella: Software; Mario Vincenzo Russo: Validation; Carmela Protano, Matteo Vitali: Data Curation; Pasquale Avino: Writing - Reviewing & Editing; Antonio De Cristofaro: Supervision.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Raw chromatogram files of honey samples analyzed by GC-MS/QqQ (Original data) (Mendeley Data)

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