Title

Improved polycyclic aromatic hydrocarbons and n-alkanes determination in speleothems through cleanroom sample processing

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Cave sediments extraction procedure and PAH results

Samples were freeze-dried and extracted by means of a Dionex Thermo Fisher ASE 350 accelerated solvent extractor at 100 °C and 1000 psi with a 1:1 mixture of dichloromethane and *n*-hexane (3 cycles, 5 min static). Extracts were concentrated under a gentle nitrogen stream. The instrumental analysis of PAHa was conducted as described in the main text for the calcite samples. Quantification was performed through the isotope dilution technique using ^{13}C-labeled internal standards (phenanthrene, acenaphthylene and benzo(*a*)pyrene) and corrected by the response factor. Results were blank corrected and divided by the samples weight. Values are in ng g^{-1}.

Table S1. Concentrations (ng g^{-1}) of target PAHs in cave sediments.

PAH	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10
Naphthalene	0.5	0.5	0.3	0.1	0.5	0.1	0.8	1	0.7	1
Acenaphthylene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Acenaphthene	nd	nd	nd	nd	nd	nd	nd	1.6	0.6	nd
Fluorene	nd	nd	0.8	nd	nd	nd	nd	0.4	nd	0.7
Phenanthrene	<lod	<lod	5	<lod	<lod	<lod	<lod	<lod	0.2	7.1
Anthracene	0.4	nd	nd	nd	nd	nd	nd	nd	nd	nd
Fluoranthene	<lod	<lod	1	<lod	0.1	<lod	0.2	<lod	0.0	0.8
Pyrene	<lod	<lod	0.4	<lod	<lod	<lod	0.1	<lod	<lod	0.3
Benzo(a)Anthracene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Chrysene	0.2	0.1	0.3	0.4	0.4	0.1	nd	0.1	0.1	0.2
Retene	<lod	0.1	nd	nd	0.7	1.9	nd	0.5	3	nd
Benzo(b)Fluoranthene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Benzo(k)Fluoranthene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Benzo(e)Pyrene	nd	nd	nd	nd	1	nd	nd	4	7	1
Benzo(a)Pyrene	0.3	0.4	nd	6	1	2	nd	3	6	<lod
Perylene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Benzo(ghi)Perylene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Indeno(1,2,3-cd)Pyrene	nd	nd	nd	nd	nd	nd	nd	nd	nd	2
Dibenzo(ah)Anthracene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd