## Data Article

# Dataset of pharmaceuticals and personal care products in a Mediterranean coastal wetland 

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#### Abstract

The dataset provides information on Pharmaceutical and Personal Care Products (PPCPs) detected in the Albufera Natural Park (Valencia, Spain), a typical Mediterranean coastal wetland. These PPCPs constitute an important group of organic pollutants highly representative of the human impact. The concentrations values measured in soil, sediment and water and the statistical relationship of contaminants between them and with the environmental parameters could help to understand their fate in different compartments. The data also reported the occurrence and removal efficiency (\%) for each contaminant in ten wastewater treatment plants (WWTPs), located in the surrounding area. This dataset could provide an idea on the effectiveness of WWTP treatments and the capacity of released PPCPs to affect the ecosystem. The extraction of analytes was based on solid-phase extraction (SPE) for water and solvent extraction followed by the previous SPE as clean-up for soil and sediment. Determination was carried out by high performance liquid chromatography tandem mass spectrometry (HPLC-MS/MS) with a triple-quadrupole.


[^0]The present dataset was analyzed within the article entitled: "Pharmaceuticals and personal care products in a Mediterranean coastal wetland: Impact of anthropogenic and spatial factors and environmental risk assessment" [1].
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## Specifications Table

| Subject | Pollution |
| :--- | :--- |
| Specific subject area | Pharmaceuticals and personal care products occurrence and fate in |
|  | Mediterranean coastal wetlands. |
| Type of data | Table |
| How data were acquired | Raw data were acquired via HPLC-MS/MS. The instrument was an Infinity 1260 |
|  | UHPLC system, coupled to mass spectrometry with a triple-quadrupole mass |
|  | detector 6410 (QqQ-MS) from Agilent Technologies (Santa Clara, CA, USA). The |
|  | ionization technique used was electrospray ionization (ESI). |
|  | Raw |
|  | Analyzed |
|  | Filtered |
|  | The mobile phase consisted of 2.5 mmol ${ }^{-1} \mathrm{NH}_{4} \mathrm{~F}$ in methanol (A) and 2.5 |
|  | mmol ${ }^{-1}$ NH |

## Value of the Data

- The contaminants monitoring of a Natural Park (Albufera) is needed to value the environmental risk and how it is related with anthropogenic pressures. In addition, statistical correlations show a relationship between studied PPCPs and the different matrices.
- National and international authorities, wastewater treatment plant managers and researchers can estimate removal, occurrence, transport and fate of PPCPs.
- The data of PPCPs occurrence in sediment, soil and water samples could serve as a knowledge base that allows the establishment of monitoring programs for the compounds that appear
with a higher frequency and establish a better assessment of the risks that exist for this natural space.
- The concentrations on each sampling point may help to understand the geophysical distribution of these contaminants and can be compared with other studies.


## 1. Data Description

The presented data were collected in the Albufera Natural Park (Valencia, Spain), which is included in the list of international wetlands (RAMSAR) (since 1990), in the Natura 2000 network as a Special Protection Area for Birds (SPA) under the Birds Directive, and is considered a Site of Community Importance (SCI) under Habitats Directive. This area of about 21000 hectares (ha) included a coastal lagoon fed by streams, rivers and irrigation channels, a sandy shoreline belt, rice paddies, vegetables and orange orchards in its most external part, where 43 sampling points and ten wastewater treatment plants (WWTPs), that discharged into irrigation channels that eventually end up in the park, were monitored. Strong anthropic pressure has already been noted in the area due to the proximity of Valencia city and its metropolitan area [3]. The occurrence of 32 pharmaceutical and personal care products (PPCPs) was investigated from November 2016 to February 2017. Detailed information of each sampling site is provided in the related article. Tables 1-2 shows the concentration of PPCPs in influent (i) and effluent (e) wastewater samples with relative date of collection. While, the removal efficiency (\%) for each compound was reported in Table 3. The occurrence of (32) water, (19) sediment and (33) soil samples was described in Tables 4-6. The data presented in Tables 1-2 and 4-6 are the average value of three method's replicates for each sample to check reproducibility. In the public repository (Mendeley Data) [4] have been published the additional tables (named table S. " $n$ "), that contain the results of each individual extraction as well as the average (Tables S1-S5) and the linearity ( $\mathrm{R}^{2}$ ), LOD, LOQ and matrix effect (ME) for each contaminant (Table S6-S7). The Statistical correlations between the studied PPCPs in soils, sediment and water were described in Tables 7-9. At last, statistical correlations between contaminants and intrinsic characteristics of all matrices were schematized in Tables 10-12. The intrinsic characteristics considered were temperature, pH , total soluble salts, dissolved $\mathrm{O}_{2}$ and redox potential in water, organic matter, carbonates, lime, clay and sand, pH , electric conductivity and cationic exchange capacity in sediments and organic matter, carbonates, sodium, potassium, magnesium and calcium, pH , electric conductivity and cationic exchange capacity in soil.

## 2. Experimental Design, Materials and Methods

The water characteristics were in situ measured using a portable Multiparameter Eutech Instrument CyberScan PCD 650 (Thermo Fisher Scientific, Basel, Switzerland). The soil and sediment characteristics were established in the laboratory using standard procedures. Organic matter was determined by oxidation with dichromate [5]. Carbonate was determined using the Bernard calcimeter [6] method and cationic exchanger capacity was calculated measuring sodium, potassium, magnesium and calcium by extraction with 1 M ammonium acetate solution and inductively coupled plasma optical emission spectroscopy) (ICP-OES) following the method of Rhoades [7]. Electric conductivity and pH were determined in the soil saturation extract with a pH -meter according to Richards [8] and finally, \% of lime, clay and sand were established using an hydrometer according to the Bouyoucos [9] method. The waste and surface waters $(200 \mathrm{~mL})$ were vacuum filtered by a $0.6-\mu \mathrm{m}$ glass fiber filter (GA-55, 90 mm - Advantec MFS, Dublin, CA, USA) and stored at $-20^{\circ} \mathrm{C}$ until the analysis. The sediments were lyophilized with a Virtis lyophilizer (SP Scientific, Gardiner, NY, USA) and the soil samples were sieved, and air-dried in the dark at $20^{\circ} \mathrm{C}$ to reduce the moisture content. The samples of water, soil and sediment were spiked with labeled internal standards to quantify the PPCPs present in the all

Table 1
Concentration of PPCPs ( $\mathrm{ng} \mathrm{L}^{-1}$ ) in the influents (i) of the WWTPs*.

|  | iAS | iCAT | iCAT | iPAL | iSAL | iPE | iPI | iPI | iPII | iPII | iPS | iQB | iSU |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Compound | 25/11/2016 | 22/11/2016 | 13/01/2017 | 14/12/2016 | 13/01/2017 | 13/01/2017 | 22/11/2016 | 13/01/2017 | 22/11/2016 | 13/01/2017 | 25/11/2016 | 14/12/2016 | 25/11/2016 |
| Alprazolam | 2.30 | n.d.** | 2.42 | 4.03 | 1.43 | 3.01 | 3.82 | 4.34 | 4.23 | 4.01 | 2.33 | 7.12 | 1.91 |
| Atenolol | 132 | 5.63 | 63.5 | 113 | 22.9 | 162 | 236 | 335 | 154 | 244 | 82.0 | 257 | 174 |
| Atorvastatine | 83.1 | 2.65 | 134 | 157 | 27.2 | 59.7 | 148 | 278 | 166 | 231 | 63.0 | 274 | 61.0 |
| Bezafibrate | 5.22 | 0.91 | 52.3 | 1.62 | 1.21 | 3.02 | 29.5 | 35.0 | 26.3 | 21.0 | 0.32 | 31.0 | 3.12 |
| BPA | 562 | 37.1 | $4.27 \times 10^{3}$ | 147 | 66.2 | 87.9 | 166 | 238 | 400 | 387 | 110 | $1.01 \times 10^{3}$ | 92.0 |
| Butylparaben | 5.43 | n.d. | 18.4 | 19.2 | 1.82 | n.d. | 13.6 | 33.0 | 1.92 | 10.8 | 9.14 | 4.62 | 6.73 |
| Caffeine | $8.70 \times 10^{3}$ | $9.78 \times 10^{3}$ | $9.36 \times 10^{3}$ | $6.08 \times 10^{3}$ | $2.46 \times 10^{3}$ | $5.24 \times 10^{3}$ | $9.99 \times 10^{3}$ | $1.32 \times 10^{3}$ | $6.95 \times 10^{3}$ | $8.36 \times 10^{3}$ | $4.34 \times 10^{3}$ | $18.07 \times 10^{3}$ | $4.3 \times 10^{3}$ |
| Chloramphenicol | 2.01 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.41 |
| Clofibric acid | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Codeine | 68.4 | 1.12 | 52.2 | 7.01 | 10.0 | 50.2 | 52.1 | 133 | 80.9 | 93.8 | 47.5 | 71.8 | 72.0 |
| Diclofenac | 324 | n.d. | 290 | 303 | 68.0 | 123 | 324 | 380 | 386 | 320 | 327 | 348 | 275 |
| Enelapril | 379 | n.d. | 283 | 63.8 | n.d. | n.d. | 288 | 281 | 243 | 141 | 119 | 510 | 127 |
| Ethylparaben | 25.0 | 4.34 | 174 | 87.2 | 5.32 | 3.03 | 87.2 | 43.0 | 0.52 | 1.71 | 5.21 | 43.0 | 73.0 |
| Etoricoxib | 9.31 | 1.32 | 17.4 | 14.8 | 6.03 | 6.94 | 14.9 | 15.0 | 18.5 | 22.0 | 12.0 | 36.1 | 7.42 |
| Flufenamic acid | 126 | 0.84 | 78.0 | 75.2 | 26.4 | 86.7 | 136 | 175 | 178 | 205 | 48.9 | 415 | 95.3 |
| Furosemide | 411 | n.d. | 354 | 198 | n.d. | 268 | 444 | 700 | 557 | 598 | 266 | 927 | 310 |
| Ibuprofen | $11.0 \times 10^{3}$ | $2.32 \times 10^{3}$ | $12.60 \times 10^{3}$ | $6.51 \times 10^{3}$ | $2.57 \times 10^{3}$ | $6.99 \times 10^{3}$ | $7.97 \times 10^{3}$ | $1.42 \times 10^{3}$ | $7.79 \times 10^{3}$ | $10.3 \times 10^{3}$ | $5.58 \times 10^{3}$ | $11.5 \times 10^{3}$ | $57.6 \times 10^{3}$ |
| Indomethacin | 6.03 | n.d. | 0.62 | n.d. | 1.03 | n.d. | 4.52 | 7.32 | n.d. | 3.82 | n.d. | n.d. | 0.72 |
| Lorazepam | 30.6 | 33.0 | 26.0 | 18.1 | 17.0 | 29.4 | 30.2 | 51.0 | 43.1 | 35.3 | 13.7 | 92.0 | 30.0 |
| Metformin | 190 | 3.62 | 338 | 323 | 47.9 | 286 | 256 | 410 | 260 | 321 | 206 | 319 | 135 |
| Methylparaben | 354 | 24.2 | 388 | 625 | 59.0 | 132 | 286 | $1.28 \times 10^{3}$ | 36.5 | 24.8 | 164 | 115 | 610 |
| Naproxen | $2.56 \times 10^{3}$ | 50.4 | $3.58 \times 10^{3}$ | 632 | 570 | $1.84 \times 10^{3}$ | $2.85 \times 10^{3}$ | $3.38 \times 10^{3}$ | $2.55 \times 10^{3}$ | $2.85 \times 10^{3}$ | $2.10 \times 10^{3}$ | $3.02 \times 10^{3}$ | $2.00 \times 10^{3}$ |
| Omeprazole | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | 270 | n.d. | n.d. | n.d. | n.d. | 158 |
| Paracetamol | $2.83 \times 10^{3}$ | $4.94 \times 10^{3}$ | $5.92 \times 10^{3}$ | $6.08 \times 10^{3}$ | $3.15 \times 10^{3}$ | $4.52 \times 10^{3}$ | $4.34 \times 10^{3}$ | $6.78 \times 10^{3}$ | $1.86 \times 10^{3}$ | $3.81 \times 10^{3}$ | $5.50 \times 10^{3}$ | $4.50 \times 10^{3}$ | $5.35 \times 10^{3}$ |
| Propylparaben | 188 | 11.7 | 395 | 288 | 39.9 | 319 | 373 | 627 | 111 | 224 | 290 | 134 | 275 |
| Salicylic acid | 779 | 167 | $2.01 \times 10^{3}$ | $1.61 \times 10^{3}$ | 250 | 394 | $1.41 \times 10^{3}$ | $2.58 \times 10^{3}$ | 237 | 187 | 730 | $1.49 \times 10^{3}$ | $1.76 \times 10^{3}$ |
| Simvastatin | $2.06 \times 10^{3}$ | n.d. | $1.67 \times 10^{3}$ | $1.30 \times 10^{3}$ | 186 | 606 | $1.28 \times 10^{3}$ | $2.02 \times 10^{3}$ | $1.31 \times 10^{3}$ | $2.03 \times 10^{3}$ | $1.60 \times 10^{3}$ | $2.05 \times 10^{3}$ | $1.75 \times 10^{3}$ |
| Thiamphenicol | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Tramadol | 569 | 153 | 551 | 297 | 138 | 419 | 587 | 859 | 681 | 750 | 220 | $1.05 \times 10^{3}$ | 416 |
| Triclocarban | 0.74 | 0.42 | n.d. | 0.41 | n.d. | 0.53 | 1.72 | 1.51 | 1.12 | 0.63 | 0.91 | 0.82 | 0.71 |
| Triclosan | 102 | 40.4 | 99.5 | 77.0 | 50.0 | n.d. | 308 | 410 | 56.2 | 133 | 156 | 153 | 104 |
| Warfarin | n.d. | n.d. | 0.33 | 0.54 | n.d. | 0.45 | n.d. | n.d. | 1.15 | n.d. | n.d. | 0.21 | n.d. |

* WWTPs: Pinedo 1 (PI), Pinedo 2 (PII), Port de Catarroja (CAT), Quart - Benàger (QB), Sueca (SU), Perelló-Sueca (PS), Perellonet (PE), Palmar (PAL), Saler (SAL) and Albufera Sud (AS)
${ }^{* *}$ n.d. $=$ not detected

Table 2
Concentration of PPCPs ( $n \mathrm{LL}^{-1}$ ) in the effluents (e) of the WWTPs*.

|  | eAS | eCAT | eCAT | ePAL | eSAL | ePE | ePI | ePI | ePII | ePII | ePS | eQB | eSU |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Compound | 25/11/2016 | 22/11/2016 | 13/01/2017 | 14/12/2016 | 13/01/2017 | 13/01/2017 | 22/11/2016 | 13/01/2017 | 22/11/2016 | 13/01/2017 | 25/11/2016 | 14/12/2016 | 25/11/2016 |
| Alprazolam | 3.20 | n.d.** | n.d. | 5.90 | 1.60 | 2.30 | 7.50 | 5.10 | 9.00 | 7.00 | 2.90 | 7.70 | 4.60 |
| Atenolol | 14.0 | 8.40 | 140 | 26.0 | 19.0 | 4.00 | 148 | 190 | 125 | 137 | 11.7 | 104 | 24.4 |
| Atorvastatine | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 152 | 143 | 52.9 | 146 | n.d. | 19.0 | 15.7 |
| Bezafibrate | 1.80 | 0.50 | 0.30 | 0.40 | 2.00 | 1.00 | 29.6 | 35.1 | 6.90 | 20.4 | 0.50 | 20.4 | 12.3 |
| BPA | 93.0 | 19.0 | n.d. | 35.8 | 11.0 | 41.0 | 181 | 165 | 65.3 | 240 | 71.0 | 221 | 83.0 |
| Butylparaben | 0.40 | n.d. | n.d. | n.d. | n.d. | n.d. | 0.20 | 0.20 | n.d. | 1.50 | n.d. | n.d. | n.d. |
| Caffeine | 8.80 | 5.50 | 43.0 | 149 | 28.9 | 36.0 | 15.3 | 72.0 | 40.4 | 151 | 26.8 | 455 | 40.0 |
| Chloramphenicol | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Clofibric acid | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Codeine | 14.1 | 1.40 | 0.20 | 7.10 | 3.50 | 7.30 | 50.1 | 76.1 | 53.6 | 96.7 | 24.5 | 32.8 | 31.0 |
| Diclofenac | 164 | n.d. | n.d. | 164 | 29.0 | 57.0 | 711 | 433 | 525 | 490 | 114 | 259 | 199 |
| Enelapril | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 83.0 | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ethylparaben | n.d. | n.d. | 0.20 | 0.30 | 0.50 | 0.40 | n.d. | 0.50 | n.d. | 1.00 | 0.60 | 4.00 | 0.30 |
| Etoricoxib | 8.00 | 2.60 | 1.40 | 19.5 | 3.70 | 4.70 | 25.7 | 24.7 | 30.2 | 22.5 | 13.4 | 22.1 | 27.7 |
| Flufenamic acid | 137 | 0.40 | n.d. | 95.8 | 22.0 | 65.0 | 350 | 200 | 277 | 200 | 45.0 | 328 | 187 |
| Furosemide | 134 | n.d. | n.d. | n.d. | 2.00 | 30.0 | 904 | 550 | 552 | 630 | 66.1 | 427 | 313 |
| Ibuprofen | n.d. | 499 | 231 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Indomethacin | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.80 | n.d. | 7.90 | n.d. | n.d. | n.d. |
| Lorazepam | 18.1 | n.d. | n.d. | 42.9 | 8.60 | 17.0 | 61.3 | 54.4 | 63.1 | 57.1 | 28.0 | 46.6 | 36.5 |
| Metformin | 6.50 | 2.00 | 4.40 | 9.90 | 1.10 | 8.00 | 66.3 | 34.9 | 18.9 | 24.0 | 15.6 | 23.4 | 26.5 |
| Methylparaben | 20.6 | n.d. | n.d. | 13.6 | 5.60 | 4.60 | 29.1 | 40.0 | 20.2 | 35.0 | 16.9 | 30.1 | 28.7 |
| Naproxen | 23.0 | n.d. | n.d. | 26.3 | n.d. | n.d. | 75.4 | n.d. | 90.7 | 146 | 26.1 | 318 | 205 |
| Omeprazole | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 30.0 | 14.0 | 25.6 | 23.0 | 4.00 | 16.0 | n.d. |
| Paracetamol | 11.3 | 2.00 | 19.0 | 34.8 | 49.0 | 39.0 | 18.1 | 37.0 | 16.6 | 15.4 | 24.1 | 37.0 | 70.0 |
| Propylparaben | 1.30 | n.d. | 1.70 | 5.30 | 4.10 | 5.00 | 5.80 | 10.0 | 3.70 | 8.70 | 3.00 | 6.20 | 2.30 |
| Salicylic acid | 1.40 | 58.4 | 108 | 380 | 310 | 248 | 207 | 345 | 205 | 325 | 124 | 580 | 212 |
| Simvastatin | n.d. | 245 | 80.0 | n.d. | n.d. | n.d. | n.d. | 35.0 | n.d. | n.d. | n.d. | n.d. | n.d. |
| Thiamphenicol | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 9.00 | n.d. | 14.8 | n.d. | n.d. | n.d. |
| Tramadol | 470 | n.d. | 3.50 | 576 | 120 | 467 | $1.10 \times 10^{3}$ | 994 | $1.28 \times 10^{3}$ | $1.30 \times 10^{3}$ | 273 | $11.6 \times 10^{3}$ | 666 |
| Triclocarban | n.d. | 0.40 | n.d. | 0.30 | 0.10 | 0.10 | 0.60 | 0.50 | n.d. | 0.4 | 1.20 | 0.40 | 0.50 |
| Triclosan | 24.0 | n.d. | n.d. | 22.6 | 4.30 | n.d. | 29.2 | 20.3 | 10.4 | 7.4 | n.d. | 14.0 | 26.3 |
| Warfarin | 0.50 | n.d. | n.d. | 0.30 | n.d. | n.d. | n.d. | n.d. | 0.80 | 0.7 | n.d. | 0.10 | 0.40 |

[^1]** n.d. $=$ not detected

Table 3
Removal efficiency (\%) for each PPCPs in each WWTPs: Pinedo 1 (PI), Pinedo 2 (PII), Port de Catarroja (CAT), Quart - Benàger (QB), Sueca (SU), Perelló-Sueca (PS), Perellonet (PE), Palmar (PAL), Saler (SAL) and Albufera Sud (AS).

|  | AS | CAT | CAT | PAL | EPE | PI | PI | PII | PII | PS | EQB |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Compound | 25/11/2016 | 22/11/2016 | 13/01/2017 | 14/12/2016 | 13/01/2017 | 22/11/2016 | 13/01/2017 | 22/11/2016 | 13/01/2017 | 25/11/2016 | 14/12/2016 | 13/01/2017 | 25/11/2016 |
| Alprazolam | -37 | /* | 100 | -47 | 22 | -99 | -18 | -115 | $-75$ | -26 | -9 | -12 | -146 |
| Atenolol | 89 | -51 | -120 | 77 | 98 | 37 | 43 | 19 | 44 | 86 | 60 | 17 | 86 |
| Atorvastatine | 100 | 100 | 100 | 100 | 100 | -3 | 49 | 68 | 37 | 100 | 93 | 100 | 74 |
| Bezafibrate | 66 | 51 | 99 | 72 | 66 | 1 | 1 | 74 | 3 | -67 | 34 | -57 | -291 |
| BPA | 83 | 49 | 100 | 76 | 53 | -9 | 31 | 84 | 38 | 35 | 78 | 83 | 10 |
| Butylparaben | 93 | 1 | 100 | 100 | 1 | 98 | 99 | 100 | 86 | 100 | 100 | 100 | 100 |
| Caffeine | 100 | 100 | 100 | 98 | 99 | 100 | 99 | 99 | 98 | 99 | 97 | 99 | 99 |
| Chloramphenicol | 100 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 100 |
| Clofibric acid | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| Codeine | 79 | -26 | 100 | -1 | 85 | 4 | 43 | 34 | -3 | 48 | 54 | 65 | 57 |
| Diclofenac | 49 | 1 | 100 | 46 | 54 | -119 | -14 | -36 | -53 | 65 | 26 | 57 | 28 |
| Enelapril | 100 | 1 | 100 | 100 | 1 | 100 | 70 | 100 | 100 | 100 | 100 | 1 | 100 |
| Ethylparaben | 100 | 100 | 100 | 100 | 87 | 100 | 99 | 100 | 42 | 89 | 91 | 90 | 100 |
| Etoricoxib | 14 | -108 | 92 | -31 | 32 | -72 | -65 | -63 | -2 | -12 | 39 | 39 | -275 |
| Flufenamic acid | -8 | 48 | 100 | -27 | 25 | -158 | -14 | -56 | 2 | 8 | 21 | 17 | -96 |
| Furosemide | 67 | 1 | 100 | 100 | 89 | -103 | 21 | 1 | -5 | 75 | 54 | ! | -1 |
| Ibuprofen | 100 | 79 | 98 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |
| Indomethacin | 100 | 1 | 100 | 1 | 1 | 100 | 76 | 1 | -108 | 1 | 1 | 100 | 100 |
| Lorazepam | 41 | 100 | 100 | -137 | 42 | -103 | -7 | -46 | -62 | -104 | 49 | 49 | -22 |
| Metformin | 97 | 44 | 99 | 97 | 97 | 74 | 91 | 93 | 93 | 92 | 93 | 98 | 80 |
| Methylparaben | 94 | 100 | 100 | 98 | 96 | 90 | 97 | 45 | -41 | 90 | 74 | 90 | 95 |
| Naproxen | 99 | 100 | 100 | 96 | 100 | 97 | 100 | 96 | 95 | 99 | 89 | 100 | 90 |
| Omeprazole | 1 | 1 | 100 | 1 | 1 | !** | 95 | ! | ! | ! | ! | 1 | 100 |
| Paracetamol | 100 | 100 | 100 | 99 | 99 | 100 | 99 | 99 | 100 | 100 | 99 | 98 | 99 |
| Propylparaben | 99 | 100 | 100 | 98 | 98 | 98 | 98 | 97 | 96 | 99 | 95 | 90 | 99 |
| Salicylic acid | 82 | 65 | 95 | 76 | 37 | 85 | 87 | 14 | -74 | 83 | 61 | -24 | 88 |
| Simvastatin | 100 | ! | 95 | 100 | 100 | 100 | 98 | 100 | 100 | 100 | 100 | 100 | 100 |
| Thiamphenicol | 1 | 1 | 1 | 1 | 1 | 1 | ! | 1 | ! | 1 | 1 | 1 | 1 |
| Tramadol | 17 | 100 | 99 | -94 | -11 | -89 | -16 | -88 | -74 | -24 | -10 | 13 | -60 |
| Triclocarban | 100 | 13 | 1 | 29 | 85 | 68 | 67 | 100 | 38 | -31 | 49 | ! | 26 |
| Triclosan | 76 | 100 | 100 | 71 | 1 | 91 | 95 | 82 | 94 | 100 | 91 | 91 | 75 |
| Warfarin | ! | 1 | 100 | 39 | 100 | 1 | 1 | 26 | ! | 1 | 71 | 1 | ! |

[^2]Table 4
Concentration of PPCPs ( $\mathrm{ng} \mathrm{L}{ }^{-1}$ ) in water samples (W. $\mathrm{n}^{\circ}$ ) of the Albufera Natural Park, Valencia, Spain.

| Compound | W. 1 | W. 4 | W. 5 | W. 6 | W. 7 | W. 10 | W. 11 | W. 12 | W. 14 | W. 15 | W. 16 | W. 17 | W. 18 | W. 19 | W. 20 | W. 21 | W. 22 | W. 23 | W. 26 | W. 27 | W. 29 | W. 30 | W. 31 | W. 32 | W. 33 | W. 34 | W. 36 | W. 37 | W. 38 | W. 39 | W. 40 | W. 41 | W. 42 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Alprazolam | 10.0 | 4.00 | n.d.* | 6.00 | n.d. | n.d. | n.d. | 1.00 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.00 | n.d. | 8.00 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Atenolol | 320 | 114 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 62.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 78 | n.d. | n.d. | n.d. | 92.0 | 52.0 | 221 | 72.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Atorvastatin | 1.00 | n.d. | n.d. | 1.00 | n.d. | n.d. | n.d. | n.d. | 21.0 | n.d. | n.d. | n.d. | n.d. | 1.00 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.00 | 1.00 | n.d. | 21.0 | 1.00 | 21.0 | 1.00 | 21.0 | n.d. | 1.00 | 21.0 | 1.00 | n.d. |
| Bezafibrate | 79.0 | 63.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.00 | n.d. | 2.00 | n.d. | n.d. | n.d. | 9.00 | n.d. | 75.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Bisphenol A | 185 | 145 | 150 | 120 | 48.0 | 65.0 | 75.0 | 56.0 | 70.0 | 25.0 | 51.0 | 48.0 | 72.0 | 45.0 | 140 | 57.0 | 31.0 | 12.0 | 19.0 | 115 | 140 | 190 | 115 | 158 | 60.0 | 205 | 197 | 54.0 | 150 | 158 | 130 | 81.0 | 150 |
| Butylparaben | n.d. | n.d. | 70.0 | 70.0 | 45.0 | n.d. | n.d. | n.d. | n.d. | 40.0 | n.d. | n.d. | 40.0 | 45.0 | n.d. | 40.0 | n.d. | n.d. | n.d. | 70.0 | n.d. | n.d. | 70.0 | n.d. | 40.0 | n.d. | 71.0 | n.d. | 70.0 | 70.0 | 42.0 | 42.0 | n.d. |
| Caffeine | 152 | 28.0 | 62.0 | 22.0 | 137 | 56.0 | 11.0 | 14.0 | 291 | 76.0 | 152 | 21.0 | 134 | 101 | 121 | 219 | 40.0 | 668 | 20.0 | 43.0 | 138 | 541 | 103 | 272 | 157 | 66.0 | 173 | 93.0 | 141 | 127 | 120 | 16.0 | 18.0 |
| Chloramphenicol | n.d. | n.d. | n.d. | 50.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 39.0 | 40.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 40.0 | n.d. | 50.0 | n.d. | n.d. | 50.0 | n.d. | n.d. | n.d. |
| Clofibric acid | 76.0 | 77.0 | 76.0 | 76.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 77.0 | n.d. | n.d. | n.d. | n.d. | 77.0 | 76.0 | 75.0 | 76.0 | 76.0 | n.d. | 75.0 | 76.0 | n.d. | 76.0 | 76.0 | 75.0 | n.d. | 80.0 |
| Codeine | 154 | 27.0 | n.d. | 26.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 8.00 | n.d. | n.d. | n.d. | 7.00 | 16.0 | n.d. | 21.0 | n.d. | n.d. | n.d. | 36.0 | 30.0 | 84.0 | 12.0 | n.d. | 39.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Diclofenac | 169 | 70.0 | 50.0 | 115 | 60.0 | 52.0 | 75.0 | 55.0 | n.d. | 75.0 | 90.0 | 67.0 | 72.0 | 63.0 | n.d. | 75.0 | 65.0 | 103 | 75.0 | n.d. | 45.0 | 95.0 | 65.0 | 82.0 | 78.0 | 66.0 | n.d. | 104 | 45.0 | 35.0 | 72.0 | 35.0 | 30.0 |
| Enalapril | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 4.00 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 8.00 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ethylparaben | n.d. | n.d. | 72.0 | 73.0 | 78.0 | 72.0 | n.d. | 82.0 | n.d. | 70.0 | n.d. | 70.0 | 70.0 | 70.0 | n.d. | n.d. | n.d. | 77.0 | 73.0 | 67.0 | n.d. | 75.0 | 66.0 | 67.0 | 80.0 | 79.0 | 70.0 | 76.0 | 65.0 | n.d. | 75.0 | 70.0 | n.d. |
| Etoricoxib | 18.0 | 4.00 | 3.00 | 8.00 | 1.00 | n.d. | 1.00 | 1.00 | n.d. | n.d. | 2.00 | n.d. | n.d. | 1.00 | 2.00 | 2.00 | n.d. | 2.00 | n.d. | 2.00 | 2.00 | 5.00 | 5.00 | 13.0 | n.d. | n.d. | 6.00 | 7.00 | 25.0 | 20.0 | 3.00 | 3.00 | 5.00 |
| Flufenamic Acid | 195 | 85.0 | 60.0 | 150 | n.d. | 1.00 | n.d. | n.d. | 1.00 | 2.00 | 4.00 | 2.00 | n.d. | n.d. | 61.0 | 7.00 | 1.00 | 15.0 | n.d. | n.d. | n.d. | 80.0 | 80.0 | 140 | 10.0 | n.d. | n.d. | 1.00 | n.d. | n.d. | n.d. | n.d. | n.d. |
| Furosemide | n.d. | 115 | n.d. | 74.0 | 48.0 | n.d. | n.d. | n.d. | n.d. | 70.0 | 90.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 65.0 | n.d. | n.d. | n.d. | 95.0 | 85.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ibuprofen | 90.0 | 85.0 | 20.0 | 35.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 144 | 60.0 | 217 | n.d. | n.d. | n.d. | 90.0 | n.d. | n.d. | 43.0 | n.d. | n.d. | n.d. |
| Indomethacin | 56.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 25.0 | n.d. | n.d. | n.d. | 24.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 50.0 | n.d. | 25.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Lorazepam | 88.0 | 15.0 | n.d. | 15.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 18.0 | n.d. | n.d. | n.d. | n.d. | 6.00 | n.d. | 31.0 | n.d. | n.d. | n.d. | 23.0 | 12.0 | 70.0 | 1.00 | n.d. | n.d. | 2.00 | n.d. | n.d. | n.d. | 5.00 | n.d. |
| Metformin | 251 | 66.0 | n.d. | 19.0 | 20.0 | 4.00 | 3.00 | 5.00 | 23.0 | 15.0 | 114 | 26.0 | 171 | 3.00 | 14.0 | 55.0 | 2.00 | 131 | 375 | 24.0 | 25.0 | 186 | 70.0 | 93.0 | 57.0 | 18.0 | 22.0 | 33.0 | 94.0 | 40.0 | 40.0 | 6.00 | 19.0 |
| Methylparaben | 75.0 | 75.0 | 107 | 73.0 | 82.0 | 74.0 | 75.0 | 80.0 | 77.0 | 75.0 | 79.0 | 72.0 | 75.0 | 74.0 | n.d. | 75.0 | n.d. | 82.0 | 75.0 | n.d. | 70.0 | 84.0 | 75.0 | 78.0 | 75.0 | 73.0 | 78.0 | 76.0 | 73.0 | n.d. | 75.0 | 82.0 | 74.0 |
| Naproxen | 195 | 145 | n.d. | 105 | n.d. | n.d. | n.d. | n.d. | 115 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 117 | 114 | 208 | 111 | n.d. | n.d. | 209 | 225 | 178 | 160 | n.d. | 86.0 | n.d. | n.d. | n.d. | 113 | 115 | n.d. |
| Omeprazole | n.d. | d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Paracetamol | 26.0 | n.d. | 5.00 | n.d. | 32.0 | 14.0 | 11.0 | 17.0 | 82.0 | 28.0 | 18.0 | n.d. | 32.0 | 31.0 | 11.0 | 40.0 | n.d. | 168 | n.d. | 25.0 | 33.0 | 75.0 | 17.0 | 37.0 | 49.0 | 43.0 | 47.0 | 23.0 | 23.0 | 25.0 | 15.0 | 23.0 | 26.0 |
| Propylparaben | n.d. | 88.0 | 75.0 | n.d. | 38.0 | 33.0 | 32.0 | 35.0 | 33.0 | 32.0 | 30.0 | 30.0 | 30.0 | 30.0 | 75.0 | 35.0 | 30.0 | 33.0 | 30.0 | 90.0 | 75.0 | 75.0 | 75.0 | 135 | 31.0 | n.d. | 75.0 | 31.0 | 75.0 | 70.0 | 32.0 | n.d. | 90.0 |
| Salicylic Acid | 99.0 | 88.0 | 75.0 | 113 | 690 | 602 | n.d. | n.d. | 840 | 282 | 249 | n.d. | 298 | 391 | 294 | 239 | 208 | 751 | 340 | 106 | 521 | 713 | 75.0 | 161 | 230 | 686 | 166 | 592 | 240 | 858 | 386 | 301 | 204 |
| Simvastatin | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Thiamphenicol | 35.0 | n.d. | n.d. | n.d. | n.d. | 5.00 | n.d. | 1.00 | n.d. | n.d. | 5.00 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 5.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 5.00 | 35.0 |
| Tramadol | $12.6 \times 10^{2}$ | 197 | 1.00 | 523 | n.d. | 11.0 | 13.0 | 12.0 | 3.00 | 5.00 | 18.0 | 9.00 | 33.0 | 7.00 | 7.0 | 84.0 | n.d. | 106 | n.d. | 22.0 | 27.0 | 333 | 171 | 695 | 89.0 | 3.00 | 50.0 | 24.0 | 56.0 | 42.0 | 18.0 | n.d. | 5.00 |
| Triclocarban | n.d. | 15.0 | 13.0 | n.d. | n.d. | 2.00 | n.d. | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | n.d. | n.d. | 5.00 | n.d. | 1.00 | n.d. | 14.0 | n.d. | 1.00 | 13.0 | n.d. | n.d. | n.d. | n.d. | 1.00 | 13.0 | 13.0 | 15.0 | 1.00 | 15.0 |
| Triclosan | 65.0 | 28.0 | n.d. | n.d. | n.d. | n.d. | 44.0 | 50.0 | 60.0 | 65.0 | n.d. | 51.0 | 55.0 | 48.0 | 15.0 | 72.0 | n.d. | 52.0 | 44.0 | n.d. | 25.0 | n.d. | 52.0 | 56.0 | 50.0 | 48.0 | 50.0 | n.d. | 45.0 | 31.0 | 35.0 | 65.0 | 20.0 |
| Warfarin | 65.0 | 65.0 | 65.0 | 64.0 | n.d. | 45.0 | n.d. | n.d. | n.d. | n.d. | 45.0 | n.d. | 48.0 | n.d. | 65.0 | 45.0 | n.d. | n.d. | 45.0 | n.d. | n.d. | 65.0 | 70.0 | 70.0 | 45.0 | 65.0 | n.d. | 46.0 | 65.0 | 65.0 | n.d. | 46.0 | n.d. |

[^3]Table 5
Concentration of PPCPs ( $\mathrm{ng} \mathrm{g}^{-1}$ ) in sediment samples (S. $\mathrm{n}^{\circ}$ ) of the Albufera Natural Park, Valencia, Spain.

| Compound | S. 5 | S. 9 | S. 10 | S. 15 | S. 16 | S. 17 | S. 18 | S. 23 | S. 26 | S. 30 | S. 33 | S. 34 | S. 35 | S. 36 | S. 37 | S. 38 | S. 39 | S. 40 | S. 41 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Alprazolam | n.d.* | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Atenolol | n.d. | 3.0 | n.d. | n.d. | 2.0 | 5.0 | 4.0 | 4.0 | 3.0 | 5.0 | 5.0 | 3.0 | 1.0 | n.d. | 1.0 | 4.0 | n.d. | n.d. | 16 |
| Atorvastatin | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.0 | 1.0 | n.d. | 21 | 1.0 | 21 | n.d. | 1.0 | 21 | n.d. | 1.0 | 21 |
| Bezafibrate | n.d. | n.d. | n.d. | 3.0 | n.d. | n.d. | 3.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 3.0 | 3.0 | n.d. |
| Bisphenol A | n.d. | 15 | 20 | 5.0 | 14 | 7.0 | 10 | 21 | 7.0 | 20 | 18 | 20 | 2.0 | n.d. | 9.0 | 14 | 19 | 19 | n.d. |
| Butylparaben | 6.0 | 6.0 | n.d. | 6.0 | 7.0 | n.d. | 6.0 | n.d. | n.d. | 6.0 | 6.0 | n.d. | 6.0 | n.d. | 6.0 | 6.0 | 6.0 | 6.0 | n.d. |
| Caffeine | 6.0 | 9.0 | 8.0 | 10 | 7.0 | 5.0 | 8.0 | 8.0 | 6.0 | 7.0 | 6.0 | 9.0 | 6.0 | 4.0 | 5.0 | 4.0 | 6.0 | 10 | 7.0 |
| Chloramphenicol | n.d. | 4.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Clofibric acid | n.d. | 5.0 | n.d. | n.d. | n.d. | n.d. | 5.0 | 5.0 | 5.0 | n.d. | $4.0$ | 5.0 | $4.0$ | $4.0$ | $5.0$ | $4.0$ | $5.0$ | $5.0$ | $5.0$ |
| Codeine | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Diclofenac | 3.0 | 3.0 | 8.0 | 8.0 | 10 | 6.0 | 7.0 | 5.0 | n.d. | 3.0 | 6.0 | 6.0 | 6.0 | n.d. | 5.0 | 3.0 | 8.0 | 4.0 | 4.0 |
| Enalapril | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ethylparaben | n.d. | 17 | 15 | n.d. | 16 | n.d. | n.d. | 15 | 15 | 18 | 15 | 17 | 18 | 15 | n.d. | 15 | 17 | 15 | 18 |
| Etoricoxib | 1.0 | n.d. | 1.0 | n.d. | n.d. | 6.0 | n.d. | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | n.d. | 2.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| Flufenamic Acid | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 3.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 |
| Furosemide | 10 | 9.0 | 9.0 | 12 | 14 | 22 | 12 | 22 | 9.0 | 13 | 15 | 48 | 10 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 10.0 |
| Ibuprofen | 47 | 13 | $10 \times 10^{1}$ | 24 | 22 | 30 | 35 | 70 | 22 | 7.0 | 23 | 29 | 63 | n.d. | 38 | 8.0 | n.d. | 28 | 4.0 |
| Indomethacin | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Lorazepam | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Metformin | 4.0 | 2.0 | 1.0 | 1.0 | 5.0 | 3.0 | 1.0 | 4.0 | 1.0 | 2.0 | 2.0 | 1.0 | 2.0 | 1.0 | 1.0 | 1.0 | 2.0 | 1.0 | 1.0 |
| Methylparaben | n.d. | 13 | 13 | n.d. | 14 | n.d. | 13 | n.d. | 17 | 18 | 18 | 18 | 19 | n.d. | 13 | n.d. | 14 | 14 | n.d. |
| Naproxen | n.d. | n.d. | n.d. | 2.0 | n.d. | n.d. | n.d. | 31 | 5.0 | n.d. | 8.0 | n.d. | n.d. | 2.0 | 4.0 | 7.0 | 7.0 | 10.0 | n.d. |
| Omeprazole | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Paracetamol | n.d. | 6.0 | n.d. | 2.0 | 2.0 | 1.0 | n.d. | n.d. | 33 | n.d. | 1.0 | n.d. | n.d. | 2.0 | n.d. | 6.0 | 6.0 | 3.0 | n.d. |
| Propylparaben | 4.0 | 2.0 | n.d. | 2.0 | n.d. | n.d. | 2.0 | 4.0 | n.d. | 2.0 | 9.0 | 9.0 | 9.0 | 9.0 | 12 | 9.0 | 9.0 | 9.0 | 9.0 |
| Salicylic Acid | 21 | 18 | 25 | 32 | 28 | 24 | 23 | 23 | 19 | 23 | 18 | 23 | 19 | 19 | 16 | 15 | 18 | 21 | 27 |
| Simvastatin | 8.0 | 21 | 21 | 13 | n.d. | 29 | 12 | n.d. | n.d. | 17 | n.d. | 12 | n.d. | n.d. | n.d. | 18 | n.d. | n.d. | 4.0 |
| Thiamphenicol | 12 | n.d. | 11 | 9.0 | 10 | 9.0 | 11 | 11 | 12 | 14 | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. |
| Tramadol | n.d. | n.d. | 1.0 | n.d. | 1.0 | 2.0 | 2.0 | 2.0 | n.d. | 13 | 2.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | 1.0 |
| Triclocarban | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 7 | 9 | 15 | n.d. | 8.0 | 10 | 5.0 | 5.0 | 12 |
| Triclosan | 10 | 14 | 8.0 | 7.0 | 8.0 | 8.0 | 13 | 18 | 9.0 | 17 | 8.0 | 9.0 | 8.0 | 8.0 | 8.0 | 8.0 | 9.0 | 8.0 | 8.0 |
| Warfarin | 8.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 8.0 | 8.0 | 8.0 | 8.0 | 9.0 | 8.0 | 8.0 |

* n.d. $=$ not detected

Table 6
Concentration of PPCPs ( $\mathrm{ng} \mathrm{g}^{-1}$ ) in soil samples (So. $\mathrm{n}^{\circ}$ ) of the Albufera Natural Park, Valencia, Spain.

| Compound | So. 1 | So. 2 | So. 3 | So. 4 | So. 5 | So. 6 | So. 7 | So. 8 | So. 9 | So. 10 | So. 11 | So. 12 | So. 13 | So. 14 | So. 15 | So. 16 | So. 17 | So. 20 | So. 23 | So. 24 | So. 25 | So. 26 | So. 29 | So. 30 | So. 31 | So. 32 | So. 33 | So. 34 | So. 35 | So. 41 | So. 42 | So. 43 | So. 44 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Alprazolam | n.d.* | 67 | n.d. | 67 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d | n.d. | n.d | n.d. | n.d. | n.d. | n.d | n.d | n.d. |
| Atenolol | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.0 | 1.0 | n.d. | 21 | 1.0 | 21 | n.d. | 1.0 | 21 | n.d. | 1.0 | 21 | 1.0 | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | 21 | n.d. | n.d. | n.d | n.d | 1.0 |
| Atorvastatin | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Bezafibrate | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Bisphenol A | 9.0 | 6.0 | 3.0 | 9.0 | 10 | 4.0 | 8.0 | 4.0 | 7.0 | 6.0 | 10 | 4.0 | 3.0 | 4.0 | 2.0 | 3.0 | 2.0 | 3.0 | 1.0 | 2.0 | 2.0 | 7.0 | 2.0 | 9.0 | 4.0 | n.d. | 3.0 | 15 | 8.0 | 9.0 | 11 | 7.0 | 9.0 |
| Butylparaben | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Caffeine | n.d. | 22 | 24 | 4.0 | 1.0 | 24 | n.d. | 3.0 | 24 | n.d. | 22 | 3.0 | 2.0 | 23 | 1.0 | 22 | 1.0 | 22 | 3.0 | 1.0 | 26 | 23 | 23 | n.d. | 23 | 1.0 | 23 | 3.0 | 22 | 16 | 13 | n.d. | 2.0 |
| Chloramphenicol | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d | n.d. |
| Clofibric Acid | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | d. | n.d. | .d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Codeine | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 7.0 | n.d. | n.d. | n.d. | n.d. | n.d. | 7.0 |
| Diclofenac | 2.0 | 3.0 | 1.0 | 1.0 | n.d. | 1.0 | 1.0 | 2.0 | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.0 | n.d. | 2.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 3.0 | n.d. | 2.0 | 2.0 | n.d. | n.d. | 2.0 | 1.0 | n.d. |
| Enalapril | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Ethylparaben | 3.0 | n.d. | n.d. | n.d. | 2.0 | n.d. | n.d. | 3.0 | n.d. | n.d. | n.d. | n.d. | n.d. | .d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 3.0 | n.d. | n.d. | 2.0 | n.d. | n.d. | n.d. | n.d. | n.d. | 3.0 | n.d. |
| Etoricoxib | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | 51 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d | n.d. | n.d. |
| Flufenamic Acid | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | .d. | d. | .d. | n.d. | n.d. | .d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Furosemide | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d | n.d. |
| Ibuprofen | 2.0 | 4.0 | 10 | n.d. | 7.0 | 4.0 | 3.0 | n.d. | 3.0 | n.d. | n.d. | n.d. | 3.0 | n.d. | n.d. | .d. | n.d. | n.d. | n.d. | n.d. | n.d. | 7.0 | n.d. | n.d. | n.d. | n.d. | 8.0 | n.d. | 4.0 | n.d. | 76 | 3.0 | n.d. |
| Indomethacin | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | .d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Lorazepam | n.d. | n.d. | 62 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 62 | n.d. | n.d. | 62 | n.d. | d. | n.d. | .d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d | n.d. |
| Metformin | n.d. | n.d. | n.d. | 47 | 47 | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | 47 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 48 | n.d. | n.c. | n.d. | n.d. | n.d. | n.d. | n.d. | 47 | n.d. |
| Methylparaben | n.d. | 3.0 | 2.0 | 2.0 | 2.0 | 3.0 | 3.0 | 2.0 | 2.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 4.0 | n.d. | n.d. | 2.0 | n.d. | n.d. | 2.0 | 2.0 | 2.0 | n.d. | n.d. |
| Naproxen | 1.0 | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | 1.0 | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. |
| Omeprazole | n.d. | n.d. | n.d. | 2.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 4.0 | n.d. | 4.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 3.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Paracetamol | n.d. | n.d. | 30 | n.d. | n.d. | 1.0 | n.d. | n.d. | 30 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 30 | 30 | 31 | n.d. | n.d. | 30 | n.d. | n.d. | n.d. | n.d. | 30 | n.d. | n.d. | 31 | n.d. | n.d. | n.d. | n.d. |
| Propylparaben | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.0 | n.d. | 2.0 | n.d. | n.d. | 1.0 | n.d. | n.d. | 1.0 | 1.0 | n.d. | n.d. | n.d. | 3.0 | n.d. | 21 | 22 | n.d. | 3.0 | n.d. | n.d. | 3.0 | 2.0 | 2.0 | 11 | 4.0 | n.d. |
| Salicylic Acid | 9.0 | n.d. | n.d. | n.d. | 2.0 | n.d. | n.d. | n.d. | 3.0 | n.d. | 2.0 | 2.0 | 20 | 2.0 | 6.0 | n.d. | 5.0 | n.d. | 2.0 | 1.0 | n.d. | n.d. | n.d. | 3.0 | n.d. | n.d. | n.d. | 6.0 | 18 | n.d. | n.d. | n.d. | 5.0 |
| Simvastatin | n.d. | n.d. | n.d. | d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d | 1.0 | n.d. | n.d | n.d. | n.d. |
| Thiamphenicol | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 3.0 |
| Tramadol | n.d. | n.d. | n.d. | 2.0 | n.d. | 1.0 | 60 | n.d. | 60 | n.d. | n.d. | n.d. | n.d. | n.d. | 60 | n.d. | 60 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 60 | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | 60 | n.d. | 60 |
| Triclocarban | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 2.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| Triclosan | 4.0 | 3.0 | n.d. | n.d. | n.d. | 1.0 | n.d. | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 4.0 | n.d. | n.d. | 1.0 | n.d. | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | 5.0 | n.d. | n.d. | n.d. |
| Warfarin | 1.0 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |

* n.d. $=$ not detected


## Table 7

Statistical correlations between the studied pharmaceuticals in soils (** Significant correlation at level of $\mathrm{P}=0.01$. * Significant correlation at level of $\mathrm{P}=0.05$ ).





Table 8
$\underline{\text { Statistical correlations between pharmaceuticals in sediments (** Significant correlation at level of } \mathrm{P}=0.01 \text {, * Significant correlation at level of } \mathrm{P}=0.05 \text { ). }}$

|  | Alpraz | Aten | Atorv | BZF | BisA | BPN | Caf | CPL | ClorA | Cod | DFC | Etor | EPB | FlufA | Furo | Ibup | Met | MPN | OPZ Pmol | PPN | SalA | SVTN | TPL | Tram | TCBN | TCSN | War |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Alpraz |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  | -.377* |
| Aten |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Atorv | . $454 * *$ |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| BZF |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| BisA | -.363* | .363* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| BPN |  |  |  | .371* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Caf |  |  |  | .522** | .375* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| CPL |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| ClorA |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Cod |  |  |  |  |  |  | .459** | .750** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| DFC |  |  |  | .365* |  |  | .385* |  | -.337* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Etor |  | .349* |  |  |  | -.516** | -.366* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| EPB |  |  | .347* |  | .402* |  |  |  | .329* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| FlufA |  | .453** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Furo |  | . $334 *$ |  |  | .334* | -.401* |  |  |  | .408* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Ibup |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Met |  |  |  |  |  |  |  |  | -.416** |  | .363* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| MPN |  |  |  |  | . $465^{* *}$ |  |  |  |  |  |  | -.332* | .370* | .330* |  |  |  |  |  |  |  |  |  |  |  |  |  |
| OPZ |  |  |  |  | .375* |  |  |  |  |  |  |  |  |  |  | .376* |  |  |  |  |  |  |  |  |  |  |  |
| Pmol |  |  |  |  |  |  |  |  |  |  | -.458** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| PPN |  |  | .469** |  |  |  |  |  | . $638^{* *}$ |  |  |  |  |  |  |  | -.329* |  |  |  |  |  |  |  |  |  |  |
| SalA | .325* |  |  |  |  |  | . $572 * *$ |  | -.561** |  | .515** |  |  |  |  |  |  |  |  | -.497** |  |  |  |  |  |  |  |
| SVTN |  | .373* |  |  |  |  |  | .341* | -. $456^{* *}$ |  |  | .393* |  |  |  |  |  |  |  | -.504** |  |  |  |  |  |  |  |
| TPL |  |  | -.497** |  |  |  |  |  | -.652** | -. $354 *$ |  |  | -.330* |  |  | .362* | . $454 * *$ |  |  | -.821** | .468** |  |  |  |  |  |  |
| Tram |  | .461** |  |  |  |  |  |  | -.357* |  |  |  |  | .830** |  |  |  |  |  |  |  |  | .428** |  |  |  |  |
| TCSN |  | . $482^{* *}$ |  |  | . $374 *$ |  |  | .327* |  |  |  |  |  | .644** |  |  | .350* |  |  |  |  |  | .453** | .614** | -.409* |  |  |
| War | -.377* | .395* | -.425** |  | .427** |  | .452** |  |  |  | .339* |  |  |  |  |  | .545** |  |  | -. 581 ** |  |  | .492** |  | -. 581 ** | .497** |  |

Alpraz: Alprazolam. Aten: Atenolol. Atorv: Atorvastatin. BZF: Bezafibrate. BisA: Bisphenol A. BPN: Butylparaben. Caf: Caffeine. CPL: Chloramphenicol. Clor A: Clofibric Acid. Cod: Codeine. DFC: Diclofenac. Etor: Etoricoxib. EPB: Ethylparaben. Fluf A: Flufenamic Acid. Furo: Furosemide. Ibup: Ibuprofen. Met: Metformin. MPN: Methylparaben. OPZ: Omeprazole. Pmol: Paracetamol. PPN: Propylparaben. SalA: Salicylic acid. SVTN: Simvastatin. TPL: Thiamphenicol. Tram: Tramadol. TCBN: Triclocarban. TCSN: Triclosan. War: Warfarin.

## Table 9

Statistical correlations between the studied pharmaceuticals in waters (** Significant correlation at level of $\mathrm{P}=0.01$, * Significant correlation at level of $\mathrm{P}=0.05$ ).

|  | Alpraz | Aten | Atorv | BZF | BisA | BPN | Caf | CPL | ClorA | Cod | DFC | Etor | EPB | FlufA | Furo | Ibup | IMTN | LZM | Met | MPN | Nap | Pmol | PPN | TPL | Tram |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Aten | .830** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Atorv |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| BZF | .858** | .899** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| BisA | .383* |  |  | .374* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| BPN |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| CPL |  |  |  |  |  | . $526 * *$ |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| ClorA |  |  |  |  | .906** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Cod | .871** | .929** |  | .830** | .423* |  |  |  | .358* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| DFC | .584** | .598** |  | . 442 ** |  |  |  |  |  | .550** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Etor | .469** | .410* |  | .410* | . $532 * *$ |  |  |  | .538** | .472** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| FlufA | .908** | .773** |  | .745** | . $454{ }^{* *}$ |  |  |  | .503** | .828** | .530** | .416* |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Ibup |  |  |  |  | .422* |  |  |  | .498** | . $372{ }^{*}$ |  |  |  | .393* | .368* |  |  |  |  |  |  |  |  |  |  |
| IMTN | .356* | .521** |  |  |  |  |  |  |  | .589** | .421* |  |  | . 450 ** |  | .510** |  |  |  |  |  |  |  |  |  |
| LZM | .870** | . 956 ** |  | . $848^{* *}$ |  |  | .380* |  |  | .935** | .624** | .445** |  | .803** |  |  | .456** |  |  |  |  |  |  |  |  |
| Met |  | .477** |  |  |  |  |  |  |  | .439* | .476** |  |  |  |  |  |  | .461** |  |  |  |  |  |  |  |
| MPN |  |  |  |  |  |  |  |  |  |  | . 376 |  | .377* |  |  |  |  |  |  |  |  |  |  |  |  |
| Nap | .462** | .609** |  | .463** |  |  | . 528 ** |  |  | .593** | .401* |  |  | . $550 * *$ | .370* | .395* | . 466 ** | .586** | .435* |  |  |  |  |  |  |
| Pmol |  |  |  |  |  |  | . $874^{* *}$ |  |  |  |  |  |  |  |  |  |  |  |  |  | .345* |  |  |  |  |
| PPN |  |  |  |  | .427* |  |  |  | . $554 * *$ |  | -.418* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| SalA |  |  | .354* |  |  |  | .513** |  |  |  |  |  |  |  |  |  |  |  |  |  |  | . $572 * *$ |  |  |  |
| TPL | . $406{ }^{*}$ | .475** |  | . 372 * |  |  |  |  |  | .510** |  |  |  |  |  |  | . $377 *$ | .475** |  |  |  |  |  |  |  |
| Tram | .951** | .890** |  | . $808 * *$ | .401* |  |  |  | .371* | .953** | .641** | .514** |  | .899** |  |  | . $534 * *$ | .924** | .423* |  | .546** |  |  | . $494 * *$ |  |
| TCBN |  |  |  |  |  | .435* |  |  | . $535{ }^{* *}$ |  |  |  |  |  |  |  |  |  |  |  |  |  | .480** |  |  |
| TCSN |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  | .387* |  |  |  |  |  |  |  |  |
| War | .412* | .386* |  | .374* | .448** |  |  |  | .401* | .362* |  | .473** |  | .570** |  |  |  | .364* |  |  |  |  |  |  | .419* |

Aten: Atenolol. Alpraz: Alprazolam. Atorv: Atorvastatin. BZF: Bezafibrate. BisA: Bisphenol A. BPN: Butylparaben. CPL: Chloramphenicol. ClorA: Clofibric Acid. Cod: Codeine. DFC: Diclofenac. Etor: Etoricoxib. FlufA: Flufenamic Acid. Ibup: Ibuprofen. IMTN: Indomethacin. LZM: Lorazepam. Met: Metformin. MPN: Methylparaben. Nap: Naproxen. Pmol: Paracetamol. PPN: Propylparaben. SalA: Salicylic acid. TPL: Thiamphenicol. Tram: Tramadol. TCBN: Triclocarban. TCSN: Triclosan. War: Warfarin.

Table 10
Statistical correlations between pharmaceuticals and intrinsic soil characteristics (** Significant correlation at level of $\mathrm{P}=0.01$. * Significant correlation at level of $\mathrm{P}=0.05$ ).

|  | pH | EC | $\mathrm{CO}_{3}$ | OM | Na | K | Mg | Ca | CEC |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Alpraz | $-.380^{*}$ | $.410^{*}$ |  |  |  | $.451^{* *}$ |  |  |  |
| BisA |  | $.358^{*}$ | $.421^{*}$ | $.372^{*}$ |  |  |  |  |  |
| Ibup |  |  |  | $.444^{* *}$ |  |  |  |  |  |
| MPN | $-.376^{*}$ |  |  |  |  | $-.363^{*}$ |  |  |  |
| Nap |  |  | $-.352^{*}$ |  |  |  |  |  |  |
| PPN |  | $.354^{*}$ |  |  | $.446^{* *}$ |  |  |  |  |
| TPL |  |  |  | $.414^{*}$ |  |  |  |  |  |
| TCSN |  | $396^{*}$ |  |  |  |  |  |  |  |
| War |  |  |  |  |  |  |  |  |  |

EC: Electric conductivity. $\mathbf{C O}_{\mathbf{3}}=$ : Carbonates. $\mathbf{O M}$ : Organic Matter. Na: Sodium. K: Potasium. Mg: Magnesium. Ca: Calcium. CEC: Cation Exchange Capacity.
Alpraz: Alprazolam. BisA: Bisphenol A. Ibup: Ibuprofen. MPN: Methylparaben. Nap: Naproxen. PPN: Propylparaben. TPL: Thiamphenicol. TCSN: Triclosan. War: Warfarin.

Table 11
Statistical correlations between pharmaceuticals and intrinsic sediment characteristics (** Significant correlation at level of $P=0.01$, * Significant correlation at level of $P=0.05$ ).

|  | OM | $\mathrm{CO}_{3}=$ | Sac | SandT | pH | EC | CEC |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Alpraz |  |  |  |  | -.459** |  |  |
| Aten |  |  |  |  |  |  |  |
| Atorv |  |  |  |  |  |  |  |
| BZF |  |  |  |  |  |  |  |
| BisA | .408* |  |  |  |  |  |  |
| BPN |  |  |  |  | .355* |  |  |
| Caf |  |  | -. $484^{* *}$ | .555** |  | -.325* |  |
| CPL |  |  |  |  |  |  |  |
| ClorA | .348* | .379* |  |  |  |  |  |
| Cod |  |  |  |  |  |  |  |
| DFC |  |  |  |  |  |  |  |
| Etor |  |  |  |  |  | .384* |  |
| EPB |  |  |  |  |  |  |  |
| FlufA |  |  |  |  | . $378 *$ | -.398* |  |
| Furo |  | -.382* |  |  |  |  |  |
| Ibup | -.334* |  | -. 366 * | .345* |  |  | -.335* |
| Met |  |  |  |  |  |  |  |
| MPN |  |  |  |  | . 426 ** | -.579** | -.399* |
| OPZ |  |  |  |  |  | -. $344^{*}$ |  |
| Pmol |  |  |  |  | .348* |  |  |
| PPN | . 512 ** | .477** | .565** | $-.490^{* *}$ |  | .510** | .368* |
| SalA | -.501** | -.614** | -.612** | . $582{ }^{* *}$ |  |  |  |
| SVTN |  |  |  |  | -.345* |  | -.328* |
| TPL |  | -.586** | -. $470^{* *}$ | .406* |  | -. $401 *$ |  |
| Tram |  |  |  |  |  |  |  |
| TCSN |  |  |  |  |  |  |  |
| War |  | -.402* |  |  |  | -.407* |  |

OM: Organic Matter. $\mathbf{C O}_{3}=$ : Carbonates. Sac: Lime + clay fractions. SandT: Total sand fraction. EC: Electric conductivity. CEC: Cation Exchange Capacity.
Alpraz: Alprazolam. Aten: Atenolol. Atorv: Atorvastatin. BZF: Bezafibrate. BisA: Bisphenol A. BPN: Butylparaben. Caf: Caffeine. CPL: Chloramphenicol. Clor A: Clofibric Acid. Cod: Codeine. DFC: Diclofenac. Etor: Etoricoxib. EPB: Ethylparaben. Fluf A: Flufenamic Acid. Furo: Furosemide. Ibup: Ibuprofen. Met: Metformin. MPN: Methylparaben. OPZ: Omeprazole. Pmol: Paracetamol. PPN: Propylparaben. SalA: Salicylic acid. SVTN: Simvastatin. TPL: Thiamphenicol. Tram: Tramadol. TCSN: Triclosan. War: Warfarin.

Table 12
Statistical correlations between the studied pharmaceuticals and intrinsic characteristics of waters (** Significant correlation at level of $P=0.01$, * Significant correlation at level of $P=0.05$ ).

|  | pH | T | EC | TDS | Rest | NaCl | DO\% | $\mathrm{Cl}^{-}$ | $\mathrm{NO}_{2}{ }^{-}$ | NO3 $=$ | SO4 $=$ | Na | K | Mg | Ca |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Alpraz | -. $4466^{* *}$ |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Aten | -.414* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Atorv |  |  |  |  |  |  | .354* |  |  |  |  |  |  |  |  |
| BisA |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Caf |  |  | -.410* |  |  | -.365* |  |  |  |  |  | -.388* |  | -.385* |  |
| ClorA |  |  |  | -.355* |  | -.345* |  |  |  |  | -.393* |  |  |  | -.361* |
| Cod | -. $458{ }^{* *}$ |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Enal |  |  |  |  |  |  |  |  | .721** |  |  |  |  |  |  |
| EPB |  |  |  |  |  |  | .371* |  |  |  |  |  |  |  |  |
| FlufA | -.398* |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Furo |  |  | -.361* |  | .381* |  |  |  |  |  |  | -.377* |  |  |  |
| IMTN |  |  |  |  |  |  |  |  | .473** |  |  |  |  |  |  |
| LZM | -.477** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Met |  |  |  |  |  |  |  |  |  | .694** |  |  |  |  |  |
| Nap |  |  |  |  |  |  | .359* |  | .387* |  |  |  |  | -.480** |  |
| Pmol |  | -.354* | -.356* |  |  |  |  |  |  |  |  |  |  |  |  |
| SalA |  | -.402* | -.387* |  |  |  |  |  |  |  |  | -.373* |  |  |  |
| TPL | -.420* |  |  |  |  |  |  |  |  |  |  | . $364 *$ | .736** |  |  |
| Tram | -.491** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| TCBN |  |  |  |  | .440* |  |  |  |  |  |  |  |  |  | -.355* |

T: temperature ( ${ }^{\circ} \mathrm{C}$ ). EC: Electric Conductivity ( $\mathrm{dS} / \mathrm{m}$ ). TDS: Total Dissolved Solids ( $\mathrm{mg} / \mathrm{L}$ ). Rest: Resistivity ( $\Omega$ ). DO\%: Dissolved Oxygen (\%). $\mathbf{C l}^{-}$: Chlorides ( $\mathrm{mg} / \mathrm{L}$ ). $\mathbf{N O}_{\mathbf{2}}{ }^{-}$: Nitrites. $\mathbf{N O}_{\mathbf{3}}{ }^{=}$: Nitrates ( $\mathrm{mg} / \mathrm{L}$ ). $\mathbf{S O}_{\mathbf{4}}=$ : Sulfates ( $\mathrm{mg} / \mathrm{L}$ ). Alpraz: Alprazolam. Aten: Atenolol. Atorv: Atorvastatin. Caf: Caffeine. ClorA: Clofibric Acid. Cod: Codeine. EPB: Ethylparaben. FlufA: Flufenamic Acid. Furo: Furosemide. IMTN: Indomethacin. LZM: Lorazepam. Met: Metformin. Nap: Naproxen. Pmol: Paracetamol. SalA: Salicylic Acid. TPL: Thiamphenicol. Tram: Tramadol. TCBN: Triclocarban.
environmental compartments. A previous solid-liquid extraction was performed for solid samples ( 1 g ), with the use of 15 mL of a mix containing Milli-Q water, Mcllvaine-EDTA buffer and methanol $(\mathrm{MeOH})$ in equal parts. The mixture was homogenized for 5 min by vortex agitation, sonicated for 10 min , and centrifuged for 6 min at 3000 rpm and $10^{\circ} \mathrm{C}$. The supernatant was separated and diluted with Milli-Q water to 200 mL . Then, the dilution was treated such as water extraction procedure.

The clean-up that involves PPCPs isolation and concentration were performed by Solid Phase Extraction (SPE). Two methods employing different cartridges Strata-X and Strata-X-CW (Phenomenex, $33 \mu \mathrm{~m}, 200 \mathrm{mg} / 6 \mathrm{~mL}$ ) characterized by a polymeric reversed and polymeric weak cationexchange stationary phase, respectively, were used. Strata-X was activated with $6 \mathrm{~mL} \mathrm{MeOH}, 6$ mL Milli-Q water and with 6 mL 2 mM Sodium Dodecyl Sulphate (SDS) solution (SDS method). While, Strata-X-CW was activated without SDS solution (WC method). The analytes were eluted with 6 mL of MeOH and 3 mL of $\mathrm{MeOH}-\mathrm{DCM}(50: 50 \mathrm{v} / \mathrm{v})$ for reversed phase and with 6 mL of $\mathrm{MeOH}-\mathrm{NH}_{4} \mathrm{OH}(95: 5 \mathrm{v} / \mathrm{v})$ for weak cation-exchange phase by gravity. The eluates were evaporated at $40^{\circ} \mathrm{C}$ and redissolved to 1 mL with mobile phase before injection.

Alprazolam, atorvastatin, caffeine, chloramphenicol, diclofenac, flufenamic acid, furosemide, ibuprofen, omeprazole, paracetamol and thiamphenicol were detected by WC method in water, sediment and soil matrix. While, atenolol, bezafibrate, butylparaben, clorfibric acid, enalapril, ethylparaben, etoricoxib, indomethacin, lorazepam, metformin, methylparaben, naproxen, propylparaben, salicylic acid, simvastatin tramadol, triclocarban, triclosan, warfarin by SDS method. Only bisphenol A and codeine were determined by both methods, SDS in water matrix and WC in sediment and soil.

Instrumental analysis was performed by 1260 Infinity UHPLC (ultra-high-performance liquid chromatography) system coupled to mass spectrometry with a triple quadrupole mass detector ( 6410 QqQ-MS) from Agilent Technologies (Santa Clara, CA, USA). The electrospray ionization (ESI) was applied in negative and positive mode. The mobile phase consisted of MeOH (solvent A) and water (solvent B) both with $\mathrm{NH}_{4} \mathrm{~F}$ at $2.5 \mathrm{mmol} \mathrm{L}{ }^{-1}$ for negative mode and MeOH (sol-
vent A) and water (solvent B) with $0.1 \%$ formic acid in both solutions for positive mode. The calibration curves used to quantify the environmental contaminants were prepared in $\mathrm{H}_{2} \mathrm{O}$ -$\mathrm{MeOH}(70-30)$ and in solvent with SDS to obtain correct quantification of those compounds.

The qualitative and quantitative analysis of each chromatogram were performed by MassHunter Workstation (version 10.0 Software) supplied by Agilent Technologies. The statistical relationship of the different contaminants between them and with the environmental parameters in the three matrices selected (water, soil and sediment) was carried out by Statistical package IBM SPSS (version 26.0).

The limits of detection (LODs) and limits of quantification (LOQs) were estimated experimentally, spiking blank samples, at the lowest concentration ( $10 \mathrm{ng} \mathrm{g}^{-1}$ and $50 \mu \mathrm{gL}^{-1}$ ), with the PPCPs pre-extraction and estimating the analyte concentration able to provide a signal-to-noise ratio of 3 and 10 respectively. The LODs ranged from 1.65 and $16.65 \mathrm{ngL}^{-1}$ in waste and surface water, and from 0.33 and $6.67 \mathrm{ng} \mathrm{g}^{-1}$ dry weight (d.w.) in sediment and soil. Particularly, to established the matrix effects (ME) two eight point calibration curves (10, 25, 50, 75, 100, 250, $500,1000 \mathrm{ng} / \mathrm{mL}$ ) were compared: (i) one prepared in solvent (that is $\mathrm{H}_{2} \mathrm{O}-\mathrm{MeOH}(70-30)$ or this mixture with SDS depending on the method) and (ii) the other prepared in blank matrix extract, also redissolving the extract in $\mathrm{H}_{2} \mathrm{O}-\mathrm{MeOH}(70-30)$ or this mixture with SDS. Then, the ME was calculated according to the formula:

$$
\begin{equation*}
M E(\%)=\left(\frac{\text { Slope of calibration curve in matrix }}{\text { Slope of calibration curve in solution }}-1\right) \times 100 \tag{1}
\end{equation*}
$$

This information together with other experimental information were described in the Sadutto [2] work.

## 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.

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[^1]:    ** WWTPs: Pinedo 1 (PI), Pinedo 2 (PII), Port de Catarroja (CAT), Quart - Benàger (QB), Sueca (SU), Perelló-Sueca (PS), Perellonet (PE), Palmar (PAL), Saler (SAL) and Albufera Sud (AS)

[^2]:    * "/" means that compound was not detected in influent and effluent wastewater samples **"!" means that compound was detected only in effluent wastewater sample.

[^3]:    * n.d. = not detected

