



## Data Article

# Preliminary dataset of emerging contaminants in surface water, bottom water, porewater, and sediment: Urban and aquaculture impacts in Coliumo bay and Cauahue Channel in the central and southern coast of Chile



Pedro A. Inostroza<sup>a,b,\*</sup>, Yolanda Soriano<sup>c</sup>, Eric Carmona<sup>d</sup>,  
Martin Krauss<sup>d</sup>, Werner Brack<sup>d,e</sup>, Thomas Backhaus<sup>a,b</sup>,  
Renato A. Quiñones<sup>f,g</sup>

<sup>a</sup> Institute for Environmental Research, RWTH Aachen University, Aachen, Germany

<sup>b</sup> Department of Biological and Environmental Sciences, University of Gothenburg, Gothenburg, Sweden

<sup>c</sup> Food and Environmental Safety Research Group of the University of Valencia (SAMA-UV), Desertification Research Centre (CIDE) CSIC-GV-UV, Valencia, Spain

<sup>d</sup> Department Exposure Science, Helmholtz Centre for Environmental Research – UFZ, Leipzig, Germany

<sup>e</sup> Department of Evolutionary Ecology and Environmental Toxicology, Goethe University Frankfurt/Main, Frankfurt/Main, Germany

<sup>f</sup> Interdisciplinary Center for Aquaculture Research (INCAR), University of Concepcion, Concepción, Chile

<sup>g</sup> Departamento de Oceanografía, Universidad de Concepción, Concepción, Chile

## ARTICLE INFO

## Article history:

Received 28 April 2024

Revised 29 May 2024

Accepted 30 May 2024

Available online 5 June 2024

## ABSTRACT

Synthetic organic chemicals, including pesticides, pharmaceuticals, and industrial compounds, pose a growing threat to marine ecosystems. Despite their potential impact, data on the co-occurrence of these contaminants in multiple compartments, including surface water, bottom water, porewater, and sediment in the marine environment remains limited. Such information is critical for assessing coastal chemical status, establishing environmental quality benchmarks, and conducting comprehensive environmental risk assessments. In this study, we describe a multifaceted monitoring campaign targeting pesticides, pharmaceuticals, surfactants, additives, and plasticizers among other synthetic chemicals

\* Corresponding author at: Institute for Environmental Research, RWTH Aachen University, Aachen, Germany.  
E-mail address: [pedro.inostroza@rwth-aachen.de](mailto:pedro.inostroza@rwth-aachen.de) (P.A. Inostroza).

Dataset link: [Dataset of chemicals of emerging concern detected in the marine environment in central and northern Patagonia in Chile \(Original data\)](#)

**Keywords:**

Target screening  
Pesticides  
Pharmaceuticals  
Marine pollution  
LVSPE

in four sampling sites. One site was located in the small Coliumo bay affected by urban settlements and tourism in central-south and additionally, we sampled three sites, Caucahue Channel, affected by urban settlements and salmon farming in northern Patagonia in Chile. Surface water, bottom water, porewater, and adjacent sediment samples were collected for target screening analysis in LC- and GC-HRMS platforms. Our results show the detection of up to 83 chemicals in surface water, 71 in bottom water, 101 in porewater, and 244 in sediments. To enhance data utility and reuse potential, we provide valuable information on the mode of action and molecular targets of the identified chemicals. This comprehensive dataset contributes to defining pollution fingerprints in coastal areas of the Global South, including remote regions in Patagonia. It serves as a critical resource for future research including marine chemical risk assessment, policymaking, and the advancement of environmental protection in these regions.

© 2024 The Author(s). Published by Elsevier Inc.

This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>)

## Specifications Table

Subject	Pollution
Specific subject area	Synthetic organic pollutants occurring in coastal areas
Type of data	Table Analyzed, Filtered
Data collection	The data were acquired via gas and liquid chromatography-high-resolution mass spectrometry. Target screening was conducted for 861 chemicals using an UltiMate 3000 LC system (Thermo Scientific, Germany) coupled to a hybrid quadrupole-Orbitrap MS (Q Exactive Plus, Thermo Scientific, Germany) with a heated electrospray ionization (ESI) source. A retrospective analysis was applied to 150 of the 861 target chemicals. More hydrophobic analytes, comprised of 36 chemicals, were re-evaluated using a TRACE 1310 GC system (Thermo Scientific, Germany) coupled to a quadrupole-Orbitrap MS (Q Exactive, Thermo Scientific, Germany) equipped with a Thermal Desorption Unit (TDU-2; Gerstel, Mülheim, Germany) and a cooled injection system (CIS; Gerstel). Data were stored: Institute for Environmental Research, RWTH Aachen University City/Town/Region: Aachen, Nordrhein-Westfalen Country: Germany
Data source location	Data were collected in one sampling campaign conducted in November 2021 in the following locations: Latitude and longitude (WGS84): Coliumo (36.531639 S 72.951917 W), Tubildad (42.124361 S 73.470944 W), Aucar (42.162694 S 73.480833 W) Montolin (42.207639 S 73.390611 W)
Data accessibility	Repository name: zenodo Data identification number: <a href="https://zenodo.org/records/11199330">10.5281/zenodo.11199330</a> Direct URL to data: <a href="https://zenodo.org/records/11199330">https://zenodo.org/records/11199330</a>

## 1. Value of the Data

- We report environmental concentrations of emerging chemicals, including pesticides, pharmaceuticals, surfactants, additives, and plasticizers among other synthetic chemicals, in coastal areas of central-southern Chile and Northern Patagonia, Chile.
- The dataset includes multi-compartment measurements (surface water, bottom water, porewater, and sediments) from each sampling site.

- The reported data can be used for the definition of pollution profiles and for the establishment of environmental quality standards.
- The data can be used by environmental risk assessors for risk prediction and by local authorities to develop mitigation strategies and environmental surveillance programs.

## 2. Background

Emerging contaminants, such as pesticides, pharmaceuticals, personal care products, and industrial chemicals, are ubiquitous in aquatic environments, often co-occurring at concentrations ranging from nanograms to micrograms per litre in both water and sediment samples. Despite frequent monitoring in marine environments, knowledge gaps persist regarding the dynamics of these chemicals between environmental matrices, including surface water, bottom water, porewater, and sediments, particularly in remote coastal areas impacted by urban and industrial activities in Patagonia. This study aims to characterize emerging contaminants across four coastal sites within all aforementioned environmental compartments to provide a comprehensive, multidimensional assessment of chemical pollution.

## 3. Data Description

The dataset originates from surface water, bottom water, porewater, and superficial sediment samples collected from four distinct sites affected by anthropogenic activities in the central-southern coast and northern Patagonia of Chile.

The dataset is reported in tabular format and is available in both Rdata (RDS) and tab-separated values (TSV) formats. The dataset can be accessed at Inostroza et al. [1]. For each reported substance, the dataset includes essential identifiers such as the CAS Registry Number (CAS RN), the International Chemical Identifier (InChI), and its hashed InChIKey. To complement the dataset, chemical classes and sub-classes, based on their intended use, are included (e.g., Class 1: pharmaceutical, Class 2: antihypertensive, Class 3: beta blocker). Additionally, the mode of action (MoA) information (broad and specific) as well as molecular target sites of each chemical was retrieved from Kramer et al. [2]. The RDS and TSV files contain the columns defined in Table 1, while Table 2 provides an overview of the micropollutants detected and quantified.

**Table 1**

Columns and its respective description in the dataset.

Columns	
Chemical_name	Name of the chemical
CAS_number	CAS Registry Number use as chemical identifier
InChIKey	Textual identifier for chemical substances
Screening_method	Screening platform used for analysis
Class_1	Use category (e.g., pharmaceutical, pesticide, biocide, etc.)
Class_2	Sub-use category (e.g., antibiotic, herbicide, plasticizer, etc.)
Class_3	Sub-use category (e.g., benzodiazepine, organophosphate, etc.)
MoA_broad	Mode of action of the chemical
MoA_specific	Specific Mode of action of the chemical
Molecular_target	Molecular target known to interact with the chemical
Matrix	Environmental compartment
Sites	Sampling sites
MDL	Method detection limit in ng/L
Quant	Measured environmental concentrations in ng/L
units	Unit of the measurements (ng/L for water and ng/g for sediment)

**Table 2**

Summary of detected and quantified chemicals per environmental matrix.

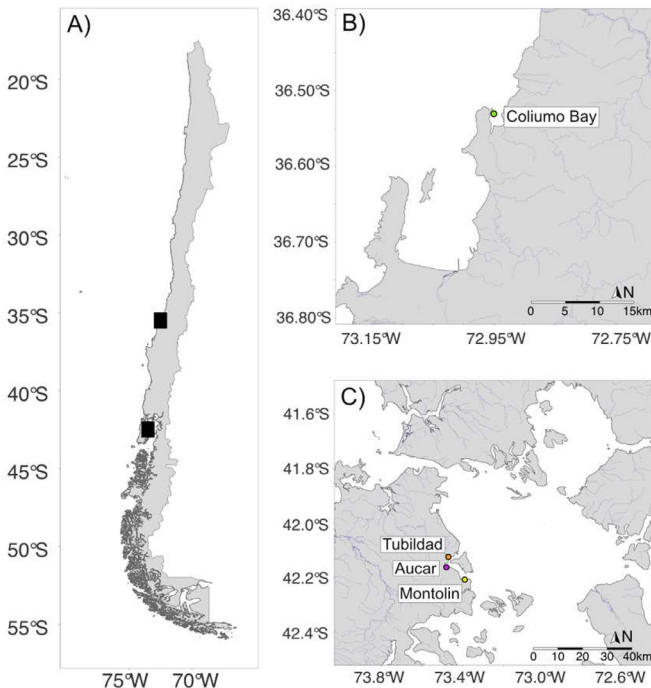
Sites	Surface water		Bottom water		Porewater		Sediment	
	Detected	Quantified	Detected	Quantified	Detected	Quantified	Detected	Quantified
Coliumo	83	47	71	40	85	51	244	224
Tubildad	73	40	66	22	101	51	238	207
Aucar	73	44	61	27	89	43	244	210
Montolin	76	42	58	21	91	41	238	202

## 4. Experimental Design, Materials and Methods

### 4.1. Sampling Design and Sample Collection

Samples were collected from four sampling sites along central-southern coast of Chile and northern Patagonia's coast, Chile, in October 2021 (spring season) (Fig. 1A). No rain was recorded before, during, and after the sampling. Sampling sites were selected based on the type of land use in the surrounding area. We primarily focused on small urban settlements featuring wastewater treatment plants discharges towards the marine environment. In addition, we selected sites hosting industrial activities such as salmon farming. This allowed us to characterise coastal sites with different levels of anthropogenic impact.

Coliumo Bay, a small bay in south-central Chile, is home to approximately 7,500 people and serves as a popular summer tourist destination. Additionally, the bay supports a small fishing fleet and receives both treated and untreated wastewater discharges. It is important to note that there is no salmon farming in Coliumo Bay or the surrounding area (Fig. 1B).



**Fig. 1.** (A) Location of sampling sites along the Chilean coast. (B) Sampling site in Coliumo Bay. (C) Sampling sites in the northern Patagonia (Caucahue Channel). All sampling sites are color-coded.

Tubildad, Aucar, and Montolin are located in northern Patagonia, specifically within the Caucahue Channel (Fig. 1C). This channel is subject to significant influence from aquaculture activities, including 37 mussel farms, 13 salmon farms, and five seaweed farms [3]. It is also impacted by agricultural activities, along with the discharge of untreated and treated urban and industrial wastewater. Further details on the geographical coordinates of the sampling sites are given in Table 3 and environmental parameters for each of the sites is provided in Table 4.

Surface water samples were collected from 2 meters below the surface using a Niskin bottle (20 L, General Oceanics). Water was transferred to 20 L glass bottles, previously cleaned with methanol, and filtered using an on-site large-volume solid phase extraction (LV SPE) device (MAXX Mess-und Probenahmetechneik GmbH, Rangendingen, Germany) (Fig. 2). The LV SPE enabled the filtering of 50 L per sampling and has been previously employed in marine sampling [4]. A detailed description of the LV SPE sampler, method development, and extraction recoveries can be found in Schulze et al. [5]. LV SPE cartridges were conditioned using a HPLC pump at a flow of 10 ml/min with 200 mL ethyl acetate followed by 200 mL methanol and 100 mL water (LC-MS grade) and stored in the fridge at 4°C before sampling. After sampling, all cartridges were dried up with nitrogen for 60 min and freeze-dried to remove remaining water. The analytes were eluted from each cartridge with 100 mL ethyl acetate, 100 mL methanol, 100 mL methanol with 1.0 vol% formic acid, and 100 mL methanol with 2 vol% of 7N ammonia in methanol. The volume was reduced using rotary evaporation (40°C water bath) and a gentle stream of nitrogen. Subsequently, all extracts were finally re-dissolved in LC-methanol at a relative enrichment factor (REF) of 1000, and the remaining extracts were stored at -20 °C until

**Table 3**

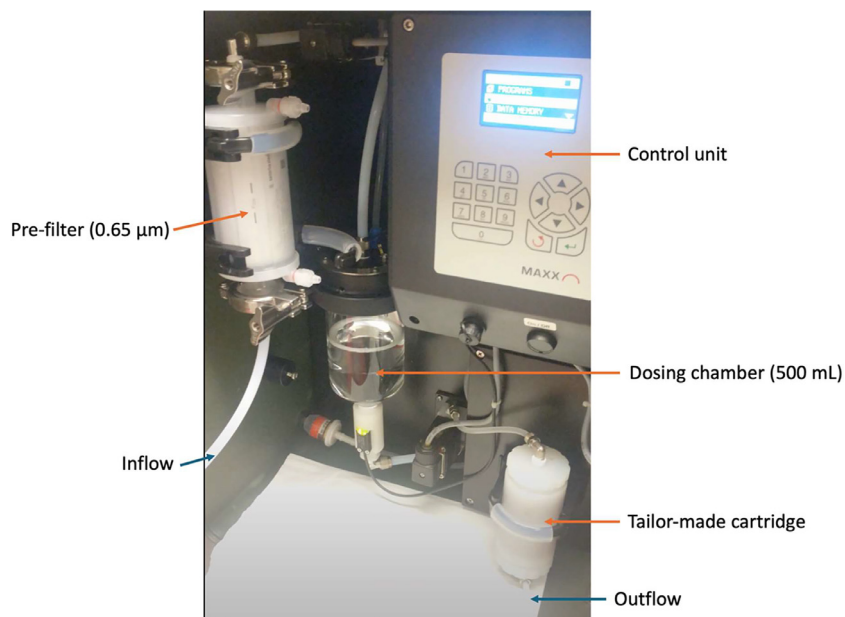
Additional sampling site information. Geographic coordinates in decimal degree (WGS84).

Sites	Latitude (S)	Longitude (W)
Coliumo	36.531632	72.951917
Tubildad	42.124361	72.470944
Aucar	42.162694	73.480833
Montolin	42.207639	73.390611

**Table 4**

Environmental parameters per sampling sites. Measurements were conducted using a multi-parameter probe 137 (Hannah model HI 9829, United Kingdom). Conductivity in ms/cm; temperature in celsius; redox potential in mVolt.

Sites	pH	Conductivity	Temperature	Redox
Coliumo-water	7.8	43.77	17.1	-
Colium-bottom	7.6	-	16.8	102.1
Coliumo-sediment (-1 cm)	7.1	-	16.4	-65.2
Coliumo-sediment (-3 cm)	6.8	-	16.5	-66.7
Coliumo-sediment (-5 cm)	6.7	-	16.3	-58.8
Tubildad-water	8.4	46.99	13.4	-
Tubildad-bottom	7.8	-	13.3	155.7
Tubildad-sediment (-1 cm)	6.9	-	14.3	-78.2
Tubildad-sediment (-3 cm)	6.7	-	14.9	-77.2
Tubildad-sediment (-5 cm)	7.0	-	14.2	-175.6
Aucar-water	8.5	44.72	16.7	-
Aucar-bottom	8.4	-	19.2	170.5
Aucar-sediment (-1 cm)	7.6	-	18.6	129.3
Aucar-sediment (-3 cm)	7.5	-	18.1	-0.7
Aucar-sediment (-5 cm)	7.6	-	17.9	-50.6
Montolin-water	8.4	46.80	12.6	-
Montolin-bottom	7.8	-	13.9	96.2
Montolin-sediment (-1 cm)	7.1	-	13.7	-52.1
Montolin-sediment (-3 cm)	7.5	-	13.8	-57.3
Montolin-sediment (-5 cm)	7.4	-	13.7	-52.4



**Fig. 2.** Picture of the LV SPE device. Main parts are pointed by orange arrows and flow direction is pointed out in blue. Detailed explanations and scheme of the LV SPE cartridge in Schulze et al. [5].

further analysis. Subsequent liquid chromatography (LC) and gas chromatography (GC) analyses followed established analytical protocols [4,6,7]. To ensure sample integrity, the remaining extracts were stored at  $-20\text{ }^{\circ}\text{C}$  for future bioassays.

Surface sediment samples were collected using a cylindrical piston corer equipped with a 6 cm diameter plastic core tube (USC 06000, UWITEC GmbH, Austria). The top 4-5 cm were sliced using a stainless-steel cutting plate previously cleaned with methanol. Samples were immediately stored in aluminum boxes in the dark at  $-20\text{ }^{\circ}\text{C}$  in a portable freezer until extraction.

Bottom water and porewater were collected using the cylindrical piston corer. The corer was predrilled at 3 cm intervals and taped before each sampling. Rhizons ( $0.45\text{ }\mu\text{m}$  pore size, Rhizosphere Research Products, Netherlands) were inserted at 3 cm intervals in the predrilled holes to collect bottom and porewater samples. These samples were stored in 5 mL brown vials with headspace (i.e., leaving some air in the vial) and kept at  $-20\text{ }^{\circ}\text{C}$ .

#### 4.2. Reagents and Chemicals Used for the Target Analysis of CECs

Methanol (LC grade, Honeywell (USA)), ethyl acetate (LC-MS grade, from Honeywell), formic acid (98–100 %, from Merck), 7N Ammonia in Methanol (from Honeywell), water (LC-MS grade from Fisher Optima LC/MS, from Fisher Chemical), dichloromethane (LC grade, from Merck) and acetone (LC grade, from Honeywell) were used. GC-grade ethyl acetate was purchased from Merck (Darmstadt, Germany). The solvents used for extraction and instrumental analysis were all of LC-MS grade. For structural confirmation and quantification, analytical standards with at least of 90 % purity were obtained from various suppliers.

Description of the isotope-labelled internal standards for GC and LC in Table 5 and Table 6, respectively. All blanks (device and sampling) were treated in the same manner as environmental samples.

**Table 5**

Internal standards - isotope-labelled compounds for quantitative GC-HRMS screening analysis.

Name	m/z	Formula
PCB-180- <sup>13</sup> C <sub>12</sub>	407.24	C <sub>12</sub> H <sub>3</sub> Cl <sub>17</sub>
Hexachlorobenzene- <sup>13</sup> C <sub>6</sub>	290.74	C <sub>6</sub> Cl <sub>6</sub>
Acenaphthene-D <sub>10</sub>	164.27	C <sub>12</sub> H <sub>10</sub>
Phenanthrene-D <sub>10</sub>	188.29	C <sub>14</sub> H <sub>10</sub>
Perylene-D <sub>12</sub>	264.38	C <sub>20</sub> H <sub>12</sub>
Chrysene-D <sub>12</sub>	240.36	C <sub>18</sub> H <sub>12</sub>
Naphthalene-D <sub>8</sub>	136.22	C <sub>10</sub> H <sub>8</sub>

**Table 6**

Internal standards - isotope-labelled compounds for quantitative LC-HRMS screening analysis.

Name	LC mode	m/z	RT [min]	Formula
Monoisobutylphthalate-D <sub>4</sub>	ESI_neg	225.1070	10.6	C <sub>12</sub> H <sub>14</sub> O <sub>4</sub>
4-Nitrophenol-D <sub>4</sub>	ESI_neg	142.0448	5.7	C <sub>6</sub> H <sub>5</sub> NO <sub>3</sub>
Tricosan-D <sub>3</sub>	ESI_neg	289.9627	13.4	C <sub>12</sub> H <sub>7</sub> Cl <sub>3</sub> O <sub>2</sub>
Mecoprop-D <sub>3</sub>	ESI_neg	216.0512	11.5	C <sub>10</sub> H <sub>11</sub> ClO <sub>3</sub>
Laurylsulfate-D <sub>25</sub>	ESI_neg	290.3048	24.4	C <sub>12</sub> H <sub>25</sub> O <sub>4</sub> S
Bezafibrate-D <sub>4</sub>	ESI_neg	364.1259	11.6	C <sub>19</sub> H <sub>20</sub> ClNO <sub>4</sub>
Acesulfame-D <sub>4</sub>	ESI_neg	166.0118	1.5	C <sub>4</sub> H <sub>4</sub> NO <sub>4</sub> S
Hydrochlorothiazide- <sup>13</sup> C-D <sub>2</sub>	ESI_neg	301.9773	1.7	C <sub>7</sub> H <sub>8</sub> ClN <sub>3</sub> O <sub>4</sub> S <sub>2</sub>
Bentazone-D <sub>6</sub>	ESI_neg	245.0872	9.7	C <sub>10</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub> S
Cyclamate-D <sub>11</sub>	ESI_neg	189.1234	4.1	C <sub>6</sub> H <sub>13</sub> NO <sub>3</sub> S
Creatinine-D <sub>3</sub>	ESI_pos	117.0850	0.6	C <sub>4</sub> H <sub>7</sub> N <sub>3</sub> O
Diazinon-D <sub>10</sub>	ESI_pos	315.1711	12.8	C <sub>12</sub> H <sub>21</sub> N <sub>2</sub> O <sub>3</sub> PS
Benzophenone-3-D <sub>5</sub>	ESI_pos	234.1173	12.3	C <sub>13</sub> H <sub>10</sub> O
p-Toluene-sulfonamide-D <sub>4</sub>	ESI_pos	193.0933	5	C <sub>7</sub> H <sub>9</sub> NO <sub>2</sub> S
Cotinine-D <sub>3</sub>	ESI_pos	180.1211	0.9	C <sub>10</sub> H <sub>12</sub> N <sub>2</sub> O
Diglyme-D <sub>6</sub>	ESI_pos	141.1392	2.4	C <sub>6</sub> H <sub>14</sub> O <sub>3</sub>
Chlormequat-D <sub>9</sub>	ESI_pos	131.1296	0.7	C <sub>5</sub> H <sub>13</sub> Cl <sub>2</sub> N
Carbamazepine-D <sub>10</sub>	ESI_pos	247.1650	9.8	C <sub>15</sub> H <sub>12</sub> N <sub>2</sub> O
Atrazine- <sup>13</sup> C <sub>3</sub>	ESI_pos	219.1111	10.2	C <sub>8</sub> H <sub>14</sub> ClN <sub>5</sub>
Benzotriazole-D <sub>4</sub>	ESI_pos	124.0807	3.9	C <sub>6</sub> H <sub>5</sub> N <sub>3</sub>
Carbendazim-D <sub>4</sub>	ESI_pos	196.1019	2.2	C <sub>9</sub> H <sub>9</sub> N <sub>3</sub> O <sub>2</sub>
Tri-n-butylphosphate-D <sub>27</sub>	ESI_pos	294.3414	13.3	C <sub>12</sub> H <sub>27</sub> O <sub>4</sub> P
DEET-D <sub>7</sub>	ESI_pos	199.1822	10.4	C <sub>12</sub> H <sub>17</sub> NO
Metolachlor-D <sub>6</sub>	ESI_pos	290.1788	12.2	C <sub>15</sub> H <sub>22</sub> ClNO <sub>2</sub>
Isoproturon-D <sub>3</sub>	ESI_pos	210.1680	10.4	C <sub>12</sub> H <sub>18</sub> N <sub>2</sub> O
Diclofenac-D <sub>4</sub>	ESI_pos	300.0491	12.5	C <sub>14</sub> H <sub>11</sub> Cl <sub>2</sub> NO <sub>2</sub>
Caffeine-D <sub>3</sub>	ESI_pos	198.1065	5.6	C <sub>8</sub> H <sub>10</sub> N <sub>4</sub> O <sub>2</sub>
Clarithromycin-D <sub>3</sub>	ESI_pos	751.5030	10.3	C <sub>38</sub> H <sub>69</sub> NO <sub>13</sub>
Desisopropylatrazine-D <sub>5</sub>	ESI_pos	179.0855	4.6	C <sub>5</sub> H <sub>8</sub> ClN <sub>5</sub>
Decyltrimethylammonium-D <sub>30</sub>	ESI_pos	230.4256	9.4	C <sub>13</sub> H <sub>30</sub> N
Atenolol-D <sub>7</sub>	ESI_pos	274.2143	1.1	C <sub>14</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub>
Progesterone-D <sub>9</sub>	ESI_pos	324.2883	12.6	C <sub>21</sub> H <sub>30</sub> O <sub>2</sub>
Verapamil-D <sub>6</sub>	ESI_pos	461.3281	9	C <sub>27</sub> H <sub>39</sub> ClN <sub>2</sub> O <sub>4</sub>
Bezafibrate-D <sub>4</sub>	ESI_pos	366.1405	11.6	C <sub>19</sub> H <sub>20</sub> ClNO <sub>4</sub>
Sulfamethoxazole-D <sub>4</sub>	ESI_pos	258.0845	6.4	C <sub>10</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub> S
Tebuconazole-D <sub>9</sub>	ESI_pos	317.2089	12.6	C <sub>16</sub> H <sub>22</sub> ClN <sub>3</sub> O
Imidacloprid-D <sub>4</sub>	ESI_pos	260.0847	6.6	C <sub>9</sub> H <sub>10</sub> ClN <sub>5</sub> O <sub>2</sub>

#### 4.3. Bottom, Porewater, and Sediment Preparation and Extraction Procedures

First, bottom and porewater samples were filtered using Whatman GF/F 55 mm filters (GE Healthcare). Solid-Phase Extraction (SPE) was performed on these samples using Chromabond HR-X cartridges (6 mL, 200 mg sorbent, Macherey-Nagel, Düren, Germany) with a Promochrom SPE-03 automated device. SPE process blanks were included in parallel.

Before sample loading, SPE cartridges were conditioned with 5 mL of ethyl acetate (10 mL/min) followed by 5 mL of methanol (10 mL/min). After sample extraction, cartridges were washed with 1 mL of ultra-pure water and dried for approximately 90 min using N<sub>2</sub>. Sample extracts were eluted with the following solvent fractions at a flow rate of 5 mL/min: 5 mL ethyl acetate, 5 mL methanol, 4 mL methanol with 1 % formic acid, and 4 mL methanol with 2 % 7N ammonia. The extracts were evaporated under nitrogen flow to approximately 500 µL, filtered (PTFE, 0.2 µm pores size, 13 mm diameter), and then evaporated to dryness. Extract residues were reconstituted in 0.5 mL of methanol.

Sediment samples were extracted by Pressurized Liquid Extraction (PLE) with a mixture of acetone and ethyl acetate (1:1 v/v) using an Accelerated Solvent Extraction (ASE) 200 device (Dionex). The raw extracts were concentrated under a nitrogen stream and the solvent was exchanged for dichloromethane. The extracts were then evaporated to near dryness using a Xcel-Vap nitrogen evaporator, and finally adjusted to 0.5 mL with dichloromethane. The extracts were transferred to 2 mL glass vials and stored at -20°C until purification.

Sediment cleanup was performed by flash chromatography using an Agilent 1200 binary pump and a pre-packed chromatography column (Chromabond Flash RS 4 g SiOH column, 10.6 cm × 12.4 mm, Macherey-Nagel, Düren, Germany). The column was first conditioned with dichloromethane and the extracts were eluted with dichloromethane and methanol, which were collected in separate fractions.

For the GC-HRMS analysis, half of the dichloromethane fraction was concentrated under a nitrogen stream and re-dissolved in ethyl acetate. The second half of the dichloromethane fraction was combined with half of the methanol fraction, concentrated under a nitrogen stream and re-dissolved in methanol for LC-HRMS analysis. Processing blanks were prepared with hydromatrix and processed the same way as the samples. All samples were filtered (pore size 45 µm) and stored in a freezer at -20°C until analysis.

#### 4.4. Target Chemical Screening in LC-HRMS and GC-HRMS

Monitoring of emerging chemicals, such as pesticides, pharmaceuticals, and industrial chemicals, is limited in coastal areas of South America, particularly in Chile [8,9]. Therefore, we employed a target list of 861 chemicals, based primarily on those commonly detected in European freshwater systems. However, it's important to note that the same target list was previously used for surface waters collected along the Swedish coast [4]. Furthermore, the classification system for these chemical classes is predominantly European-centric.

The target screening was conducted using an UltiMate 3000 LC system (Thermo Scientific) coupled with a hybrid quadrupole-Orbitrap MS (QExactive Plus, Thermo Scientific) featuring a heated electrospray ionization (ESI) source. A retrospective analysis, as outlined by Muschket et al. [10], was applied to 150 out of the 861 target chemicals. Furthermore, an additional evaluation using a TRACE 1310 GC system (Thermo Scientific) coupled with a hybrid quadrupole-Orbitrap MS (QExactive GC, Thermo Scientific) was performed for the more hydrophobic analytes (36 chemicals). This GC system was equipped with a Thermal Desorption Unit (TDU-2; Gerstel, Mülheim, Germany) and a cooled injection system (CIS; Gerstel). More detailed information on the settings for LC-HRMS and GC-HRMS can be found in [4,7]. A set of method-matched calibration and internal standards, consisting of 7 (GC) and 38 (LC) isotope-labelled compounds (Tables 5 and 6), were used for quantification.

ProteoWizard (V 2.1.0) was used to convert LC-HRMS raw data into mzML format [11]. Subsequently, peak detection, sample alignment, and target compound annotation were performed using MZmine (V 2.40.1) [12] as detailed in Beckers et al. [13]. We used the R package {MZquant} (V 0.7.22) to perform blank correction, calibration, and then quantification of the annotated target compounds. Blank peak elimination and blank intensity thresholds were calculated according to Machate et al. [7].

For the quantification of GC-HRMS detected compounds, the software TraceFinder 4.1 (Thermo Scientific) was used for further evaluation. Method-matched calibration standards were



used in a series ranging from 0.5 to 5000 ng/L. These calibration standards were treated in the same way as the samples. The target compounds were quantified using the internal standards with the nearest retention time following Nanusha et al. [6] and Machate et al. [7]. The method detection limits (MDLs) were determined according to US-EPA procedure [14] and all chemicals below MDLs are reported as detected as well as above the respective MDLs were grouped as quantified. Finally, raw data is available upon request.

## Limitations

The chemical target list for micropollutant analysis in this study was primarily based on chemicals commonly detected in streams and rivers in European contexts. Besides, the classification of chemical classes is predominantly European-centric. The classification of modes and mechanisms of action relates for the vast majority of chemicals to the intended target organisms or close phylogenetic relatives. Furthermore, our study is based on a single sampling campaign conducted in late spring. Therefore, our findings provide only a one-time chemical fingerprint of the studied area.

## Ethics Statement

The proposed data does not involve any human subjects, animal experiments, or data collected from social media platforms.

## Data Availability

[Dataset of chemicals of emerging concern detected in the marine environment in central and northern Patagonia in Chile \(Original data\) \(zenodo\).](#)

## CRedit Author Statement

**Pedro A. Inostroza:** Conceptualization, Methodology, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review & editing, Visualization; **Yolanda Soriano:** Methodology, Validation, Formal analysis, Writing – review & editing; **Eric Carmona:** Methodology, Validation, Formal analysis, Writing – review & editing; **Martin Krauss:** Methodology, Validation, Investigation, Writing – review & editing; **Werner Brack:** Investigation, Resources, Writing – review & editing; **Thomas Backhaus:** Resources, Writing – review & editing, Funding acquisition; **Renato A. Quiñones:** Conceptualization, Resources, Investigation, Writing – review & editing.

## Acknowledgements

We thank Oliver Alarcon and Vicente Villalobos for fieldwork support, Margit Petre and Jörg Ahleim (UFZ) for LVSPE cartridge preparations. This work was supported by the FRAM Centre for Future Chemical Risk Assessment and Management at the University of Gothenburg and the Interdisciplinary Center for Aquaculture Research (INCAR, FONDAP-ANID, [Grants 1522A0004](#)). The QExactive Plus LC-HRMS and GC-HRMS used at UFZ are part of the major infrastructure initiative CITEPro (Chemicals in the Terrestrial Environment Profiler) funded by the Helmholtz Association.

## 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.

## References

- [1] P.A. Inostroza, Y. Soriano, E. Carmona, M. Krauss, W. Brack, T. Backhaus, R. Quiñones, Dataset of chemicals of emerging concern detected in the marine environment in central and northern Patagonia in Chile, (2024). [10.5281/zenodo.11080740](https://doi.org/10.5281/zenodo.11080740).
- [2] L. Kramer, T. Schulze, N. Klüver, R. Altenburger, J. Hackermüller, M. Krauss, W. Busch, Curated mode-of-action data and effect concentrations for chemicals relevant for the aquatic environment, *Sci. Data* 11 (2024) 60, doi:[10.1038/s41597-023-02904-7](https://doi.org/10.1038/s41597-023-02904-7).
- [3] M. Sobarzo, L. Bravo, C. Iturra, A. Troncoso, R. Riquelme, P. Campos, C. Agurto, Hydrodynamics of a channel occupied by the aquaculture industry in southern Chile: implications for connectivity between farms, *Aquac. Environ. Interact.* 10 (2018) 291–307, doi:[10.3354/aei00268](https://doi.org/10.3354/aei00268).
- [4] P.A. Inostroza, E. Carmona, Á. Arrhenius, M. Krauss, W. Brack, T. Backhaus, Target screening of chemicals of emerging concern (CECs) in surface waters of the Swedish West Coast, *Data* 8 (2023) 93 (Basel), doi:[10.3390/data8060093](https://doi.org/10.3390/data8060093).
- [5] T. Schulze, M. Ahel, J. Ahlheim, S. Ait-Aissa, F. Brion, C. Di Paolo, J. Froment, A.O. Hidasi, J. Hollender, H. Hollert, M. Hu, A. Kloß, S. Koprivica, M. Krauss, M. Muz, P. Oswald, M. Petre, J.E. Schollée, T.B. Seiler, Y. Shao, J. Slobodnik, M. Sonavane, M.J.F. Suter, K.E. Tollefsen, Z. Tousova, K.H. Walz, W. Brack, Assessment of a novel device for onsite integrative large-volume solid phase extraction of water samples to enable a comprehensive chemical and effect-based analysis, *Sci. Total Environ.* 581 (2017) 350–358, doi:[10.1016/j.scitotenv.2016.12.140](https://doi.org/10.1016/j.scitotenv.2016.12.140).
- [6] M.Y. Nanusha, M. Krauss, B.G. Sørensen, T. Schulze, B.W. Strobel, W. Brack, Occurrence of plant secondary metabolite fingerprints in river waters from Eastern Jutland, Denmark, *Environ. Sci. Eur.* 33 (2021) 25, doi:[10.1186/s12302-021-00464-4](https://doi.org/10.1186/s12302-021-00464-4).
- [7] O. Machate, D.S. Schmeller, A. Loyau, A. Paschke, M. Krauss, E. Carmona, T. Schulze, A. Moyer, K. Lutz, W. Brack, Complex chemical cocktail, containing insecticides diazinon and permethrin, drives acute toxicity to crustaceans in mountain lakes, *Sci. Total Environ.* 828 (2022) 154456, doi:[10.1016/j.scitotenv.2022.154456](https://doi.org/10.1016/j.scitotenv.2022.154456).
- [8] M.L. Tovar-Salvador, M.G. Pintado-Herrera, P.A. Lara-Martín, E. Bonnail, Occurrence, sources and environmental risk assessment of organic micropollutants in coastal sediments from the Atacama Region (Chile), *Sci. Total Environ.* 900 (2023) 165871, doi:[10.1016/j.scitotenv.2023.165871](https://doi.org/10.1016/j.scitotenv.2023.165871).
- [9] K. Pozo, C. Ahrendt, V. Gómez, C. Jacobsen, M. Torres, T. Recabarren, V. Oyanedel-Craver, O. Audy, P. Příbylová, J. Klánová, Novel flame retardants detected in marine plastic litter in coastal areas in central Chile, *Mar. Pollut. Bull.* 201 (2024) 116194, doi:[10.1016/j.marpolbul.2024.116194](https://doi.org/10.1016/j.marpolbul.2024.116194).
- [10] M. Muschket, W. Brack, P.A. Inostroza, L.M. Beckers, T. Schulze, M. Krauss, Sources and fate of the antiandrogenic fluorescent dye 4-methyl-7-diethylaminocoumarin in small river systems, *Environ. Toxicol. Chem.* 40 (2021) 3078–3091, doi:[10.1002/etc.5181](https://doi.org/10.1002/etc.5181).
- [11] J.D. Holman, D.L. Tabb, P. Mallick, Employing proteowizard to convert raw mass spectrometry data, *Curr. Protoc. Bioinform.* 46 (2014) 13.24.1–13.24.9, doi:[10.1002/0471250953.bi1324s46](https://doi.org/10.1002/0471250953.bi1324s46).
- [12] T. Pluskal, S. Castillo, A. Villar-Briones, M. Orešič, MZmine 2: Modular framework for processing, visualizing, and analyzing mass spectrometry-based molecular profile data, *BMC Bioinform.* 11 (2010) 1–11, doi:[10.1186/1471-2105-11-395](https://doi.org/10.1186/1471-2105-11-395).
- [13] L.M. Beckers, W. Brack, J.P. Dann, M. Krauss, E. Müller, T. Schulze, Unraveling longitudinal pollution patterns of organic micropollutants in a river by non-target screening and cluster analysis, *Sci. Total Environ.* 727 (2020) 138388, doi:[10.1016/j.scitotenv.2020.138388](https://doi.org/10.1016/j.scitotenv.2020.138388).
- [14] US Environmental Protection Agency (USEPA) Definition and Procedure for the Determination of the Method Detection Limit, Revision 2, U.S. Environmental Protection Agency, 2016.