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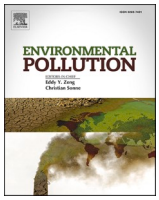

An optimal QuEChERS method for the determination of emerging contaminants by LC-MS/MS in the Antarctic bivalve *Adamussium colbecki*☆

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A B S T R A C T

Emerging contaminants (ECs) are anthropogenic and naturally occurring chemicals detected in various environmental matrices, including remote regions such as Antarctica. *Adamussium colbecki*, an Antarctic bivalve, is an excellent bioindicator for assessing contamination in polar marine ecosystems. However, its high protein (10–18 %) and lipid (2–10 %) content require an effective sample pre-treatment to minimise matrix interferences during contaminant analysis. In this study, an optimised QuEChERS extraction method was developed and validated to determine ECs in *A. colbecki* using high-performance liquid chromatography coupled with tandem mass spectrometry (HPLC-MS/MS). A multivariate experimental design approach was employed to improve analyte recovery (R%) and minimise matrix effect (ME%). Initially, a Plackett-Burman screening identified the most influential factors, which were subsequently optimised using a Doehlert design to construct quadratic models for response optimisation. The final method allowed to obtain acceptable recoveries (46–123 %) and satisfactory matrix effects (62–103 %) for 18 ECs, confirming its suitability for complex biological matrices. The optimised protocol was then applied to *A. colbecki* specimens collected during Antarctic campaigns in 2001, 2005, 2018, and 2019. Triclosan (TCS) was quantified in two samples and detected below the quantification limit in two others, while perfluorooctanoic acid (PFOA) and octyl-dimethyl p-aminobenzoic acid (OD-PABA) were identified at trace levels in 2005 samples. This study presents the first validated QuEChERS method for ECs analysis in *A. colbecki*, providing a reliable tool for long-term environmental monitoring and contamination assessment in the Antarctic marine ecosystem.

1. Introduction

Emerging contaminants (ECs) are chemical species, both anthropogenic and naturally occurring, found in different environmental compartments (Khan et al., 2022). Their spread can cause known or suspected harm to the environment and human health, yet they have not been subjected to international regulation (Lowther, 2014; Noguera-Oviedo and Aga, 2016). ECs encompass a wide range of categories, including pesticides, pharmaceuticals and personal care products (PPCPs), illegal drugs, plasticisers, hormones, microplastics (which can behave as collectors of ECs), and more (Richardson and Kimura, 2016; Sauvé and Desrosiers, 2014; Chouchene et al., 2022).

Even remote regions, such as Antarctica, have not remained free from anthropic activities and are no longer pristine (Olalla et al., 2020; Vecchio et al., 2025). Pesticides and other synthetic organic compounds have been detected in Antarctic animal tissue since the 1960s (Bargagli and Rota, 2024). These contaminants can enter the Antarctic environment through long-range transport (LTR) or direct input from aeroplanes, boats, and wastewater treatment plants at research stations (Lowther, 2014). The presence of ECs in Antarctica is expected to grow in the future, due to several factors, including the increasing number of scientific bases and visitors, and the low efficiency of the treatment plants (Olalla et al., 2020; MacKeown et al., 2024). The region's low temperatures, freezing water, and polar night periods reduce the degradation processes of contaminants, thereby increasing ECs' persistence (Olalla et al., 2020). Consequently, their accumulation or pseudo-persistence in the Antarctic environment may pose risks to living beings, including mutagenicity, genotoxicity, reproductive and developmental disorders, and immune system disruption in various species (Postigo et al., 2023).

Due to their high tolerance to a wide range of ambient conditions and their filter-feeding nature bivalves are commonly used to monitor marine contamination (Mwangi et al., 2016; Pizzini et al., 2015). *Adamussium colbecki*, the most widespread bivalve organism in the Antarctic coastal environment, is particularly suited for this role. This specie has been frequently used in various studies as a biomonitoring organism;

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already in 1992, Berkman and Nigro proposed it as a bioindicator for the study of trace metals around Antarctica(Berkman and Nigro, 1992). Later on, Chiantore et al. explored the role of this organism in circulation of organic matter in the coastal area of Terra Nova Bay (Chiantore et al., 1998). In the same area, Magi et al. employed *Adamussium colbecki* to assess the occurrence of the antifouling agent tributyltin and its degradation products (Magi et al., 2004). The bivalve was also employed for monitoring and retrospective studies on persistent organic pollutants (POPs) (Vecchio et al., 2025; Bonacci et al., 2009; Grotti et al., 2016; Pizzini et al., 2017). Endemic and circumpolar, *A. colbecki* is primarily found near Syowa Station, Stonington Islan, McMurdo Sound, and Terra Nova Bay (Chiantore et al., 1998). Its abundance in near-shore waters and its ability to accumulate contaminants through its filter-feeding behaviour make *A. colbecki* suitable for monitoring pollution in the Antarctic ecosystem (Pizzini et al., 2015; Caroli and Bottoni, 2010).

However, the complexity of the sample poses a significant challenge in interpreting biomonitoring results. *A. colbecki* samples need to be pretreated to eliminate the high concentration of proteins (10–18 %) and lipids (2–10 %) that can interfere with the detection of contaminants (Diallo et al., 2022). Common extraction techniques, such as accelerated solvent extraction (ASE), pressurised liquid extraction (PLE), and microwave-assisted solvent extraction (MASE) have limitations in handling these matrices (Diallo et al., 2022; Picot Groz et al., 2014; McEneff et al., 2013; Ribeiro et al., 2020; Perestrelo et al., 2019). The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) extraction method offers a preferable alternative due to its low cost, high efficiency, reliability, and potential ability to extract a wide range of analytes (Diallo et al., 2022; Picot Groz et al., 2014; Long et al., 2023; Kim et al., 2019; Rausch et al., 2021). This method, initially developed for the analysis of pesticides in food, involves an initial liquid-liquid partitioning followed by a clean-up of the extract using dispersive solid-phase extraction (d-SPE), to remove sources of potentially interfering compounds (Long et al., 2023; Kim et al., 2019). Despite the widespread use of the QuEChERS method for various analyte-matrix combinations, its application to the analysis of emerging pollutants in biological matrices from protected areas, such as polar regions, remains limited (Azcune et al., 2022). Our study contributes to addressing this gap by adapting and optimising the QuEChERS approach for the extraction of emerging contaminants in *A. colbecki* (Kim et al., 2019).

The sample treatment by QuEChERS for the subsequent analysis of ECs in *A. colbecki*, requires the optimisation of several parameters, including sample amount, extraction solvent, solvent pH, salt amount, type and amount of d-SPE sorbent, and dilution of extracts to reduce matrix effects (Diallo et al., 2022; Perestrelo et al., 2019). Given the high complexity and potential interaction between these parameters, a univariate approach is insufficient. Instead, experimental design is a valuable tool for optimising multiple parameters simultaneously (Diallo et al., 2022; Benedetti et al., 2019).

Herein, we present a comprehensive optimisation of the QuEChERS method for determining ECs in *A. colbecki* samples, analysed by high-performance liquid chromatography coupled to tandem mass-spectrometry (HPLC-MS/MS). A multivariate approach was exploited to optimise both the recovery (R%) and matrix effect (ME%) through two experimental designs. Firstly, independent variables such as the solvent/sample ratio, PSA and C18 amount, extraction and clean-up time, and centrifugation mode were investigated by a screening Plackett-Burman design. Then, a Doehlert design was used to study the variables previously identified as statistically significant, in order to provide quadratic models for response optimisation. Finally, the optimised procedure was applied to study environmental samples collected in Antarctica during Antarctic campaign of 2001, 2005, 2018, and 2019. Hence, the paper reports for the first time the development and validation of a QuEChERS method for the analysis of emerging contaminants in the Antarctic marine biota, using the bioindicator *A. colbecki*.

2. Materials and methods

2.1. Standards and reagents

The analytical standards were obtained from different suppliers. The following compounds were purchased from Sigma-Aldrich (St. Louis, MO, USA): paraxanthine (PRX), theophylline (TFL), carbamazepine (CBZ), hydrochlorothiazide (HCTZ), benzophenone-3 (BP-3), octyl dimethyl p-aminobenzoate (OD-PABA), ethyl hexyl methoxy cinnamate (EHMC), octocrylene (OC), perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), acesulfame K (ACS), sucralose (SCL), bisphenol A (BPA), estrone (E1), β-estradiol (E2), 17α-ethinyl estradiol (EE2), ibuprofen (IBU), gemfibrozil (GEM), furosemide (FRSM), metoprolol (MTPL), clenbuterol (CLBT), terbutaline (TRBT), atenolol (ATN), ethylexyl salicylate (EHS), nicotine (NCT), cocaine (COCA), omethoate (OMT), daminozide (DMNZ), 2,4-dichlorophenoxyacetic acid (2,4-D), chloramphenicol (CMPH), metformin (MTF), mepiquat (MPQ), chlormequat (CMQ), and triclosan (TCS). Caffeine (CAFF), ketoprofen (KET), naproxen (NAP), theobromine (THB) and diclofenac (DCF) were bought from Fluka Analytical (Saint Gallen, Switzerland), whereas salbutamol (SLBT) was purchased from Alfa Aesar (Haverhill, MA, USA). All standards were high purity grade (≥97 %). Stock standard solutions were prepared in methanol or methanol-water (1:1) mixture.

Magnesium sulfate was supplied from Carlo Erba Reagents (Rodano, MI, Italy) and sodium chloride from Sigma Aldrich (St. Louis, MO, USA). End-capped C18 bonded silica loose sorbent was obtained from Supelco (Bellefonte, PA, USA) while primary secondary amine (PSA) loose sorbents was sourced from Phenomenex (Torrance, CA, USA). Methanol (MeOH) and acetonitrile (ACN), both HPLC-MS grade, were purchased from Merck (Darmstadt, Germany). Ultrapure water was obtained from a Millipore Q-Gard system equipped with a Millipak 0.22 μm filter (Millipore, Watford, UK).

2.2. LC-MS/MS analysis

The analyses were performed using a 1200 SL Liquid Chromatograph coupled to an Agilent 6430 Triple Quadrupole mass spectrometer (MS) equipped with an Electrospray Ionization (ESI) ion source (Agilent Technologies, Santa Clara, CA, USA). The MS operating conditions were: drying gas N_2 (purity >98 %), temperature 300 °C, 11 L/min flow; nebuliser gas pressure 15 psi; capillary voltage 4000 V.

Chromatographic separation was achieved using a Kinetex® F5 analytical reversed-phase column (100 mm × 2.1 mm i.d.; 2.6 μm particle size, Phenomenex, Torrance, CA, USA). The separation and the analysis of target compounds were conducted using two distinct chromatographic methods, for analytes ionizing in positive and negative polarity, respectively. Details are present in Paragraph S1 of Supporting Information (Table S1 and Table S2).

Dynamic-multiple reaction monitoring (d-MRM) mode was employed to enhance the selectivity and sensitivity of the MS detection method. The most abundant MRM transition was used for quantification (quantifier) while additional transitions were used for qualitative confirmation (qualifier). Detailed MS conditions and MRM transitions are provided in SI (Table S3). The ratio between the peak area of the qualifier and the quantifier ion was used to confirm compound identities. In the cases of compounds for which only one relatable MRM transition was available, identification was instead based on the comparison of retention times with those of analytical standards, with full awareness of the limitations this may entail in terms of confirmation. Data acquisition, qualitative, and quantitative analyses were performed using The MassHunter 10.0 software from Agilent.

2.3. Experimental design

To optimise the QuEChERS procedure in terms of analyte recovery (R %) and matrix effect (ME%), a structured approach using Design of

Experiments (DOE) was adopted. The QuEChERS method involves an initial extraction step using an ACN: H_2O solvent mixture, followed by agitation and addition of salts ($MgSO_4$ and NaCl) to achieve phase separation. The organic fraction is then collected, and a dispersive solid-phase extraction (d-SPE) step is used for purification by adding $MgSO_4$ and clean-up sorbents (PSA and C18). In this study, six factors that could potentially affect the R% and the ME% of the analytes were initially examined at two levels (coded as −1 and +1), as detailed in Table 1. First, a screening was performed, followed by a response surface design to optimise the factors influencing the response. The Plackett-Burman design was used for the initial screening of variables. This design allows the simultaneous investigation of multiple factors to determine their significance and linear relation with the responses as well as to identify their ranges (Plackett and Burman, 1946). Specifically, this design is effective for examining k variables with a minimum of 4n experiments, where 4n is the smallest multiple of four greater than k. In this study, 8 experiments were necessary, but they were performed in replicate to increase the degrees of freedom. This allowed evaluating the significance of the variables and their main effects on R% and ME%.

The experiments of the Plackett-Burman are summarized in Table S4 and were performed in random order to avoid systematic errors due to time effect.

Four factors were identified as significant for most analytes during the screening phase and were subsequently studied using a Doehlert design (Doehlert, 1970). The studied factors included the solvent/sample ratio, PSA amount, and clean-up time, which were analysed at five, seven, and three levels, respectively (Table 2). A total of 15 experiments (Table S5), including three replicates at the central point, were conducted to evaluate the linear, quadratic, and interaction terms of the models (Sarabia et al., 2009). Again, the experiments were performed in random order. Extracts obtained from each experimental condition were analysed using HPLC-MS/MS and the key responses, R% and ME% were evaluated for each analyte.

R% was evaluated by normalizing the peak areas to the mass of the sample processed in each experiment, calculated using the following formula:

$$R(\%) = \frac{PA_B - PA_{NS}}{PA_A - PA_{NS}}$$

Where PA_B represents the peak area of the analyte in the sample spiked before sample treatment, PA_{NS} is the peak area of the non-spiked sample, and PA_A is the peak area of the analyte in the sample spiked after sample treatment. The recovery study was performed by spiking the samples at a concentration of 960 ng/g.

ME% was studied for each sample. The ME% was determined by analysing the extracts spiked with a known quantity of analytes (PA_A) and comparing the signal with the corresponding pure standard in MeOH:H_2O. The following formula was used to calculate ME%:

$$ME(\%) = \frac{PA_A - PA_{NS}}{PA_P}$$

Where PA_A and PA_{NS} are the same already defined, and PA_P is the peak area of the pure standard solution. The open-source software Chemometric Agil Tool (CAT), developed by the Chemistry Group of the Italian Chemical Society, was used for model computation using multiple linear regression (MLR), and for statistical evaluations (Leardi and Melzi).

Table 1
Factors investigated in the Plackett-Burman experimental design and selected levels.

Factor	−1 level	+1 level
V_{solv}/m_{sample} ratio (mL/mg)	0.06	0.12
Quantity of PSA (mg)	25	100
Quantity of C18 (mg)	25	100
Extraction time (min)	1	4
Clean-up time (min)	1	4
Shaking mode	Vortex	Ultrasounds

Table 2
Factors investigated in the Doehlert experimental design and selected levels.

Factor	Level	Value
V_{solv}/m_{sample} ratio (mL/mg)	−1	0.06
	−0.5	0.08
	0	0.10
	0.5	0.12
	1	0.14
Quantity of PSA (mg)	−0.866	10
	−0.577	25
	−0.289	40
	0	55.0
	0.289	70
	0.577	85
	0.866	100
Clean-up time (min)	−0.817	1
	0	2.5
	0.817	4

2.4. MLR model validation

Three-dimensional response surfaces and contour plots were generated using CAT software to visualise the influence of each variable on the responses and to identify optimal regions. The predictive capability of the experimental model was validated by estimating the response obtained in non-investigated regions of the experimental domain. The predicted values generated through CAT, were compared to the experimental values (taking into account the respective confidence intervals) obtained from the analysis of spiked samples processed using the optimised conditions.

Confidence intervals for predictions were obtained by multiplying the leverage by the experimental standard deviation, while for the experimental values they were determined by multiplying the standard deviation of the measurements by the tabulated Student's t-value. The consistency observed between the predicted and experimentally measured values was used to validate the accuracy of the models.

2.5. Optimised extraction procedure

The optimised QuEChERS consists of two-steps: an initial extraction of the analytes followed by a clean-up using PSA and C18 sorbents.

First, 100 mg of *Adamussium colbecki* were weighted in a 50 mL centrifuge tube and 12 mL of ACN: H_2O (50:50, v/v) were added. The tube was vortexed for 1 min. To allow a salting-out extraction, 3.4 g of $MgSO_4$ and 0.9 g of NaCl were added. The sample was vortexed again and centrifuged at 3500 rpm for 5 min to achieve phase separation between ACN and water.

Following centrifugation, 2 mL of the supernatant were subjected to d-SPE, using 300 mg of $MgSO_4$, 85 mg of PSA, and 100 mg of C18. The sample was vortexed for 1 min and centrifuged again at 3500 rpm for 5 min). An aliquot of 1 mL of supernatant was then evaporated under a nitrogen stream and reconstituted in 1 mL of MeOH: H2O (50:50, v/v). The extract was then filtrated through a 0.2 μm polytetrafluoroethylene (PTFE) filter, diluted 5-fold with MeOH: H_2O (50:50, v/v), and subsequently analysed by means of HPLC-MS/MS.

2.6. Method performance

The following analytical figures of merit were assessed: linearity, accuracy (expressed as percentage recovery), precision (evaluated in terms of repeatability), as well as limit of detection (LOD) and quantification (LOQ).

Method's accuracy was determined via recovery assay, using *A. colbecki* samples spiked with target analytes (n = 3 replicates) on two

separate days. Repeatability was calculated as relative standard deviation (RSD%) of replicate analyses (n = 3) conducted within a single day (intra-day precision), while inter-day RSD% values were determined across replicate samples (n = 6) analysed on two distinct days. Due to the limited availability of *A. colbecki* and the logistical constraints associated with the collection of Antarctic environmental samples, the number of replicates used for intra- and inter-day accuracy assessments was limited.

Linearity was confirmed by constructing calibration curves, plotting the peak areas of the analytes against their corresponding concentrations ranging from 0.1 to 20 ng/mL (0.1, 0.5, 1, 5, 10, and 20 ng/mL). The linearity of the method was confirmed by the coefficient of determination (R^2), ensuring robust quantification over the specified range.

Finally, the LOD was experimentally determined from procedural blanks, defined as the lowest analyte concentration distinguishable from matrix signal with a signal-to-noise ratio (S/N) of 3, calculated on the quantifier ion transition of each analyte. The LOQ was calculated as the lowest concentration yielding as a S/N ratio of 10, ensuring accurate and reliable quantification of analytes in the sample matrix.

2.7. Sample collection, pooling, and storage

Specimens of *A. colbecki* were systematically collected as part of the Italian National Antarctic Research Program (PNRA) and, more recently, by researchers from Jang Bogo Station, South Korea. Samples were collected ethically by researcher during the Antarctic campaigns. The sampling was conducted in Terra Nova Bay, an 80 × 30 km inlet in southwestern Ross Sea, bordered by Cape Washington to the north and Drygalski Ice Tongue to the south. This bay represents a continental shelf with an average depth of approximately 450 m, reaching a maximum depth of 1100 m in the Drygalski Basin (Grotti et al., 2016).

Sampling sites were located between 74°38′ to 74°43′ South Latitude and 164°02′ to 164°13′ East Longitude, spanning areas from the coast to the edge of a polynya, as shown in Fig. S5 in Supplementary Information (18). At sampling depths ranging from 10 to 30 m, the seafloor primarily consisted of granitic rock interspersed with softer substrates of coarse sands and gravels.

Four environmental samples, each consisting of pooled tissues from ten individuals, were analysed. Specimens collected in 2001 and 2005 were obtained by Italian researchers through manual scuba diving. More recent samples, collected in 2018 and 2019, were provided by South Korean scientists located in Jang Bogo Station. All organisms were carefully placed in polyethylene bags immediately after collection and stored at −80 °C until further processing.

In preparation for analysis, specimens were thawed at 4 °C, and each organism's body length and weight were measured. Soft tissues were carefully dissected, thoroughly rinsed with ultrapure water to remove residual detritus, homogenised and subsequently freeze-dried to preserve sample integrity.

3. Results and discussion

To obtain a procedure suitable for the extraction of a broad range of ECs the optimisation followed a two-step experimental design approach. The selected analytes (n = 40) were chosen to represent a broad spectrum of ECs, including perfluorinated compounds, UV filters, PPCPs, lifestyle compounds, and pesticides. The selection was based also on the frequence of occurrence in literature studies regarding water contaminants. The amount of salts used for phase separation during QuEChERS extraction was maintained, as preliminary experiments demonstrated that this quantity ensured effective phase separation (Benedetti et al., 2019). Similarly, the amount of $MgSO_4$ used during the clean-up was kept constant, as it effectively removed residual water without impacting analyte recovery or matrix effect.

Six experimental variables were evaluated: solvent volume to sample mass ratio (V_{solv}/m_{sample}), extraction time, amount of PSA and C18 clean-up sorbents, clean-up time and shaking mode. Due to the diverse nature of interfering species, a combination of clean-up sorbents was tested to achieve a broad-spectrum purification. While C18 was used to adsorb non-polar and medium-polar compounds, PSA was chosen for its effectiveness in removing fatty acids, organic acids, and polar pigments (Gao et al., 2015).

Optimal conditions were chosen by evaluating R% and ME% as responses, targeting at high recovery rates and matrix effects (for their definition see Materials and Methods) for the analytes under investigation. The optimal ME value is 100 %, at lower values the matrix influences the ionization of the analyte leading to a suppression of the signal, at values above 100 % an increase in the signal is observed.

3.1. Screening design results

The experimental domain for the screening design was selected to be broad enough to detect any possible impact of the factors on the response but narrow enough to minimise confounding effects among factors. Six factors were investigated using the Plackett-Burman design as reported in SI (Table S4), with ranges defined based on preliminary tests and supported by existing literature (Álvarez-Muñoz et al., 2019).

The linear models obtained exhibited substantial variability in both ME% and R% across the different analytes, with recovery explained variances (R^2_{adj}) ranging from 33.8 % to 85.3 % and matrix effect explained variances from 40.5 % to 90.8 % for 18 analytes. Detailed statistical parameters, including R^2_{adj}, coefficients values and confidence intervals, are provided in the Supporting Information.

Statistical analysis at the 95 % confidence level (p = 0.05) identified four factors as significant contributors in affecting the response: the V_{solv}/m_{sample} ratio, the PSA clean-up sorbent, the clean-up time, and the shaking mode. As an example, Fig. 1 illustrates the significance of each factor, along with the associated confidence interval, affecting the ME% and R% of OD-PABA.

The four key factors (V_{solv}/m_{sample} ratio, PSA sorbent amount, clean-up time, and shaking mode) consistently emerged as significant predictors for recovery and matrix effect across most of the analytes. Among these, the shaking mode, a qualitative factor, exhibited a negative coefficient and subsequently was fixed to vortex (coded level −1) for the following optimisation step. Indeed, a maximization of both responses was seeked.

With shaking mode fixed, the remaining three factors were further optimised through a quadratic experimental design and response surface methodology. The non-significant factors were set to practical values that best supported the experimental consistency and efficiency.

3.2. Doehlert design results and optimum selection

The application of Design of Experiments (DoE) combined with Response Surface Methodology (RSM) provides a robust approach to develop mathematical models that describe the experimental domain, enabling statistical predictions and identification of optimal conditions. Commonly employed designs in RSM include Box-Behnken, central composite, and Doehlert designs. In this study, the Doehlert design was selected. Although the Doehlert experimental matrices are neither orthogonal nor rotatable (Cerqueira et al., 2021), it was chosen due to its flexibility. In fact, it allows design expansion by adding variables and movement within the experimental space. This design also supports studying the variables at a different number of levels within a single matrix, facilitating focused evaluation of critical factors that may require more detailed assessment.

In this study, V_{solv}/m_{sample} ratio, PSA quantity, and clean-up time were studied at seven, five, and three levels, respectively. This level differentiation enabled careful examination of variables considered most influential. A revised experimental domain was assessed by considering the results of the screening, and the experimental design matrix outlining each experimental condition is presented in the

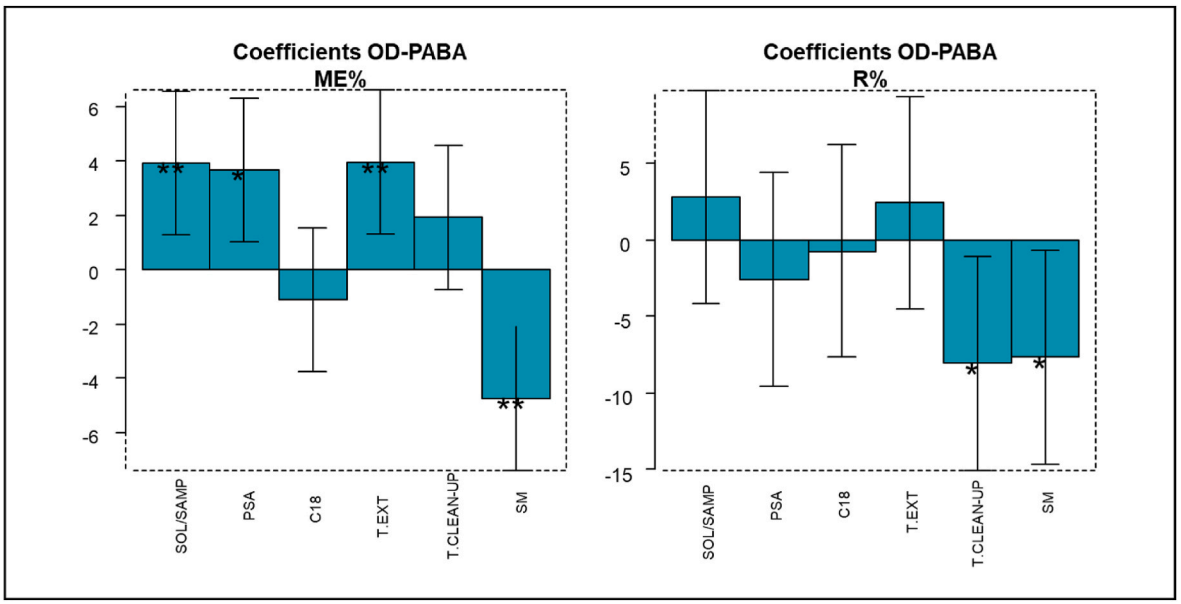

Fig. 1. Statistical significance of each factor affecting the ME% and R% of OD-PABA. One asterisk (*) indicates significance at a 95 % confidence level and two asterisks (**) indicate a 99 % confidence level.

Supporting Information (Table S5).

In terms of ME% significant explained variance was observed for 13 models within the chosen experimental domain (detailed results in Table S6 of the SI). For the remaining analytes, the ME% was satisfactory in all the domain, with the exception of COCA, which displayed consistently high ion suppression across the domain, likely due to interference from unidentified species.

For the analyte R%, significant explained variance was achieved for 12 models (results in Table S7 of the SI). For drugs such as NAP, DCF, FRSM, and the synthetic hormone E2, PSA quantity coefficient was found to be significant and negative, suggesting that PSA interact with those analytes, resulting in reduced recovery. For highly polar analytes, including MPQ, NCT, CMQ, and DMNZ, the models were not satisfactory; indeed, they exhibited poor recoveries in all conditions, likely due to distribution in the aqueous phase during the salting-out extraction. Finally, basic compounds (SLBT, ATN, MTPL and TRBT) showed variable recoveries ranging from 40 % to 60 %, which still seemed not to be significantly correlated with the variables in the considered domain.

The satisfactory models were then validated using the procedure in Section 2.4: validated models included R% for ACS, NAPR, GEM, and KET, and ME% for MTF, TRBT, and KET. However, recovery rates for some of these analytes were not sufficient to be included in the final procedure. Fig. 2 presents the response surfaces for OC, detailing its matrix effect and recovery. Complete response surfaces and contour plots for all analytes are available in Supporting Information.

For most analytes, the clean-up time best setting was at a coded level of −0.817, namely 1 min. Optimal recovery for analytes such as HCTZ, FRSM, TRBT, ATN, MTPL, OC, and EHS was achieved within a relatively broad parameter space, with PSA sorbent levels between −0.577 and 0.577 (25–85 mg), and recovery and matrix effect responses maximization agreeing within the range of −0.5 to 0.5 (0.08–0.12 mL/mg). Achieving the overall optimum for multiple responses required balancing the conditions identified as ideal for individual analytes. The final optimal conditions, presented in Fig. 3, were determined as 12 mL

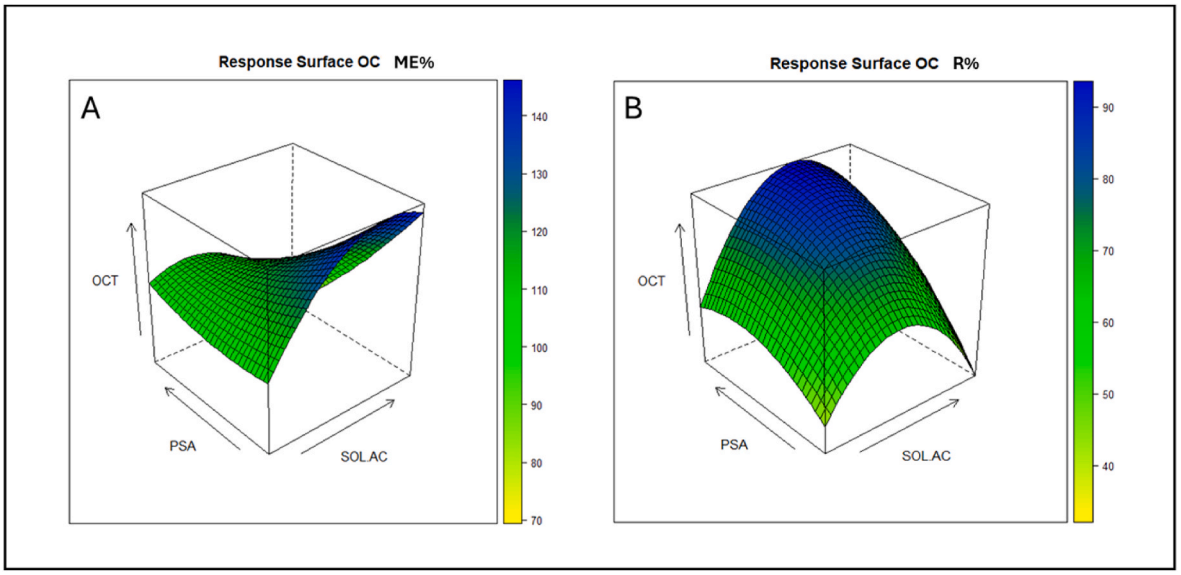

Fig. 2. Response surfaces for ME% (Figure A) and R% (Figure B) of OC.

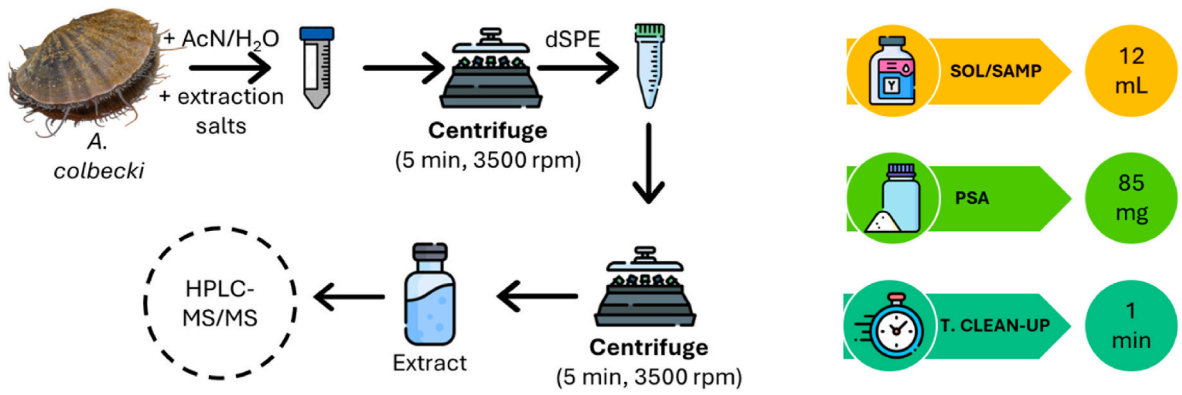

Fig. 3. Graphical overview of the QuEChERS method and the optimised parameters chosen for the extraction of 18 ECs from *A. colbecki* samples. Icons created by Freepik, sourced from Flaticon (https://www.flaticon.com/free-icons).

of solvent for 100 mg of *A. colbecki*, 85 mg of PSA sorbent, and a clean-up time of 1 min. Validation of these conditions was performed through three replicate experiments under optimal settings. The optimised experiment yielded satisfactory results of both R% and ME%, in line with model predictions, and even for analytes for which the model showed less satisfactory performance, such as PFOA and PFOS.

3.3. Method performance

Table 3 included the 18 analytes that met the established criteria in terms of good method performance. All analytes demonstrated satisfactory linearity, with determination coefficients (R^2) ranging between 0.991 and 0.999. Limits of detection and quantification were determined following the methodology outlined in Section 2.3.3. The calculated LOD and LOQ values for each analyte demonstrate the method's high sensitivity and suitability for detecting trace levels of analytes in complex matrices, except for EHS. Recovery percentages, most falling within the acceptable range of 60–123 %, indicated reliable accuracy of the method. Exceptions included SCL, DCF, and OMT, which exhibited recoveries outside the range, but were still maintained in the method. Intra-day precision, expressed as RSD% for three replicates, yielded values below 19.9 % for most analytes, except for OC (24 %) and EHS (38.4 %). For this latter analyte, low sensitivity might have affected precision. Inter-day precision, based on five replicates, showed RSD% values between 2.4 % and 12.5 % for most analytes, with exceptions observed for DCF (26.1 %), EHS (27.8 %), OD-PABA (33.3 %), and OC (39.3 %), highlighting a higher variability across separate analyses. These results reflect the method's generally acceptable precision. Matrix effects, yielded values between 70 % and 103 % for most analytes, indicating minimal ion suppression or enhancement in the analytical system; however, HCTZ exhibited the lowest ME value of 62 %. Specificity was then ensured by acquiring two MRM transitions (quantifier and qualifier) for each analyte and checking the ratio between their peak areas and retention times (Kruve et al., 2015).

Overall, these validation parameters confirm the method's reliability and robustness for most analytes, while highlighting a few areas for potential refinement, particularly concerning precision and recovery for specific compounds.

3.4. Analysis of environmental samples

The final analytical method integrated the optimised QuEChERS pretreatment, determined using a multivariate approach, with HPLC-MS/MS analysis employing a specific and precise MRM method for detecting 18 ECs of Table 3. Antarctic samples of *A. colbecki* were processed using the optimised procedure, and quantification was carried out using external calibration, thanks to the good accuracy and low ion suppression achieved. Indeed, due to the optimisation of ME%, which was negligible for 15 out of the 18 analytes, LC-MS signal normalisation was unnecessary. The three analytes with non-negligible ME% were not detected in the samples. The results of the quantification in the environmental samples are summarized in Table 4.

Table 3
Determination coefficient, matrix effect, recovery, relative standard deviation intra-day and inter-day, LOD and LOQ. (*) Results for this analyte should be interpreted with caution due to potential quantitative uncertainty.

Compound	R^2	ME (%) (n = 5)	R (%) (n = 5)	RSD (%) (Intra-day, n = 3)	RSD (%) (Inter-day, n = 6)	LOD (ng/g)	LOQ (ng/g)
OMT	0.999	94 ± 4	58 ± 7	13.8	2.4	7	20
TBR	0.994	87 ± 6	68 ± 6	2.1	4.4	41	123
CAFF	0.994	84 ± 3	90 ± 9	10.4	2.7	26	79
MTPL	0.999	94 ± 3	66 ± 7	2.5	9.7	120	401
CLBT	0.998	94 ± 5	73 ± 11	9.6	10.9	3	8
CBZ	0.999	103 ± 7	92 ± 15	11.3	10.5	3	8
BP-3	0.996	84 ± 4	77 ± 13	18.4	7.8	43	129
EHMC	0.997	89 ± 4	68 ± 6	9.9	3.8	51	154
OC	0.997	91 ± 14	84 ± 35	24	39.3	126	377
OD-PABA	0.997	93 ± 7	81 ± 27	11.3	33.3	5	14
EHS	0.994	72 ± 11	70 ± 29	38.4	27.8	947	2840
HCTZ	0.991	62 ± 8	123 ± 16	18.2	3.3	22	67
SCL	0.992	88 ± 7	59 ± 6	1.9	12.5	28	84
CMPH	0.999	98 ± 3	95 ± 9	14.3	5.3	6	19
PFOA	0.999	95 ± 5	102 ± 11	0.8	11.9	9	27
PFOS	0.996	84 ± 9	117 ± 26	19.9	21.0	7	22
DCF (*)	0.991	101 ± 3	46 ± 10	5.3	26.1	20	59
TCS	0.996	70 ± 5	91 ± 16	19.0	9.7	34	102

Table 4
Quantitation results, expressed as ng per g of dry weight of each sample of *A. colbecki*. The letters A and B indicate the two procedural replicates. Quantification results are expressed with their relative standard deviations, calculated on the instrumental replicates.

Analyte	AC 2001 A	AC 2001 B	AC 2005 A	AC 2005 B	AC 2018 A	AC 2018 B	AC 2019 A	AC 2019 B
OD-PABA	<LOD	<LOD	19 ± 6	<LOQ	<LOD	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOQ	<LOD	<LOD	<LOD	<LOD	<LOD
TCS	<LOD	<LOD	<LOQ	<LOQ	112 ± 42	129 ± 24	240 ± 3	198 ± 56

Among the detected contaminants, TCS, an antimicrobial agent, known to exert toxic effects on algae, micro-organisms, amphibians, and fish larvae even at low concentration (Emnet et al., 2015). Although its use in human hygiene biocides is no longer approved in the European Union since 2016, TCS may still be present in products used in international research stations operating in Antarctica (Sinicropi et al., 2022). TCS was quantified in two samples and detected at levels below the quantification limit in another. Both PFOA and OD-PABA were detected in *A. colbecki* samples collected in 2005 at levels below the quantification limit. These findings indicate the presence of these compounds in the Antarctic marine environment, albeit at trace levels. Although the possibility that prolonged storage may have affected the stability of some compounds cannot be completely excluded, the trace levels of analytes detected in the environmental samples are more likely indicative of the generally low contamination levels in Antarctic areas. Due to the logistical challenges of obtaining Antarctic samples, data on the presence of ECs in the region remain scarce, making a direct comparison difficult. However, our findings align with observations from prior studies regarding other matrices. For instance, Costa et al. reported quantifiable levels of TCS (3.8 ± 5.2 ng/g) in sediments from Ezcurra inlet, while TCS was detected at levels below the quantification limit in other locations within Admiralty Bay. Although Ezcurra inlet is geographically distant from Terra Nova Bay, these findings suggest the potential TCS contamination of Antarctic waters (Costa et al., 2024). TCS presence in Antarctic marine ecosystem likely originates from wastewater discharges from scientific bases. Proximity to international research bases may present a plausible source of contamination, particularly through wastewater discharge, which can introduce micropollutants into the environment. However, the contribution of long-range transport mechanisms, such as oceanic currents, cannot be excluded as alternative source (Zhao et al., 2012). The potential influence of nearby stations is supported by the spatial distribution of the sampling sites, as illustrated in Fig. S5 of the SI, which highlights the proximity of *A. colbecki* collection points to few research stations, including Mario Zucchelli and Jang Bogo. Studies, such as Emnet et al. conducted in 2015, have demonstrated TCS presence in effluents from wastewater treatment plants (WWTPs).

OD-PABA, a UV filter used as PCP, was also detected, even if below the quantification level. No studies to date have highlighted its presence in Antarctic seawater or studied its presence in sediments and species (MacKeown et al., 2024). However, OD-PABA has been detected in other non-Antarctic bivalves, such as *Mytilus galloprovincialis*, with reported concentrations of up to 833 ng/g dry weight (Picot Groz et al., 2014). This highlights the capacity of bivalves to bioaccumulate OD-PABA, suggesting similar potential in *A. colbecki*.

Finally, PFOA, a perfluoroalkyl substance (PFAS), was detected although below LOQ in *A. colbecki*. Previous studies, such as that by Cai et al., reported PFOA presence in Antarctic coastal seawater, with significant variability in concentrations depending on the location. The study attributed these variations to localised contamination, potentially from wastewater discharges (Cai et al., 2012). As a filter-feeding organism, *A. colbecki* likely accumulated PFOA directly from seawater.

The detection of ECs such as TCS, OD-PABA, and PFOA in *A. colbecki* underlines the vulnerability of Antarctic ecosystems to anthropogenic pollutants. These contaminants, even at trace levels, pose a potential threat to the biodiversity and ecological balance of the region. Continued monitoring is necessary to determine the environmental impact of human activities in Antarctica.

4. Conclusions

The analysis of complex matrices such as *A. colbecki* presents challenges due to high concentration of proteins and lipids, which can interfere with analyte recovery and introduce significant matrix effect. The aim of this study was to optimise R% and ME% through the DoE.

Using a multivariate approach, we conducted a total of 31 experiments, sixteen in an initial screening design to identify four significant factors, followed by fifteen experiments in a response surface design to optimise the quantitative variables. The mathematical models were validated and applied to determine the optimal conditions across the experimental domain. This is the first study applying a simple, fast, and cost-effective QuEChERS-based approach to a complex Antarctic biota sample, and the first one ever conducted to study ECs in *A. colbecki*. The optimised protocol demonstrated acceptable recoveries (46–123 %) and good matrix effects (62–103 %) for 18 ECs, proving the method's reliability for quantifying emerging contaminants in *A. colbecki* samples.

After the optimisation, Antarctic samples of *A. colbecki* were processed and quantification was performed. Among the studied analytes, TCS was detected in four samples, with concentrations ranging from 112 to 240 ng/g of dry weight in two of them. PFOA and OD-PABA were also found, below quantification level, in samples collected in 2005. This methodological advance offers a robust and easy-to-implement tool for future environmental monitoring, enabling standardised assessment of contaminants to detect trace levels of contamination that may pose a threat to the Antarctic unique marine biota. The findings from this study highlight the need for further research to build upon the methodological advantages presented here, particularly through the application of this approach to a wider range of contaminants and Antarctic species, and over extended temporal and spatial scales.

CRediT authorship contribution statement

Julia Gambetta Vianna: Writing – original draft, Validation, Methodology, Formal analysis, Data curation, Conceptualization. **Barbara Benedetti:** Writing – review & editing, Supervision, Methodology, Data curation, Conceptualization. **Jung-Keun Oh:** Writing – review & editing, Resources. **Marina Di Carro:** Writing – review & editing, Methodology, Conceptualization. **Emanuele Magi:** Writing – review & editing, Supervision, Project administration, Methodology, Conceptualization.

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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2025.126415.

Data availability

Data will be made available on request.

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