

Persistent contaminant baselines in dated sediments from a protected coastal lagoon in southeastern Uruguay

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Abstract

Purpose

Dated sediment cores establish contaminant baselines where long-term monitoring is unavailable. Nutrias Lagoon, within a protected area in southeastern Uruguay, is widely regarded as one of the region's few intact, low-impact South Atlantic coastal lagoon environments, yet this assumption has not been tested against persistent contaminants. This study assessed the occurrence and ecological relevance of polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), and total mercury (Hg).

Methods

A 65-cm sediment core collected in 2018 was dated using ^{210}Pb and ^{137}Cs . Seven PCB and seven PBDE congeners were analyzed by GC–MS/MS after QuEChERS extraction, and total Hg by direct mercury analysis. Profiles were interpreted against the chronology, mass accumulation rates, paleoenvironmental information, and selected sediment-quality screening values.

Results

Total Hg ranged from 56 ± 6 to $85 \pm 9 \mu\text{g kg}^{-1}$ dry weight and remained below the threshold effect level of $130 \mu\text{g kg}^{-1}$. PCB levels were < LOQ while PCB 52 occurred qualitatively in nine intervals, including five dated to 2001–2015 CE. None of the seven PBDE congeners were detected. Previously reported total PAHs remained below the aggregate benchmark, although some locally exceeded individual screening values. The contrasting profiles indicate heterogeneous, low-intensity contamination shaped by compound-specific behavior and sedimentary processes rather than a dominant source or event.

Conclusion

Nutrias Lagoon is a low-impact sedimentary reference rather than a pristine reference site. Its dated record provides a regional baseline while highlighting the need for compound-specific ecological interpretation and caution when attributing diffuse contaminant sources without direct source measurements.

Introduction

Sediment cores provide long-term archives of environmental change and are particularly useful for reconstructing contaminant histories in aquatic systems where instrumental monitoring is scarce or absent (Smol, 2019). Through sediment deposition, physical, chemical, and biological signals derived from the water body and its catchment can be preserved over time, allowing the reconstruction of environmental baselines and the identification of low-intensity anthropogenic pressures (Arocena, 2016; Smol, 2008). This approach is especially relevant in regions where information on historical contamination is limited and where most environmental assessments rely on recent monitoring data (Gros et al., 2023).

Persistent contaminants, including polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), mercury (Hg), and polycyclic aromatic hydrocarbons (PAHs), are of particular concern because of their persistence, toxicity, affinity for sediments, and potential for atmospheric transport (Campos et al., 2022; Cooke et al., 2020; Hung et al., 2013). Although some of these substances have been banned or restricted, they may continue to be detected in aquatic sediments due to legacy emissions, environmental redistribution, and diffuse regional or long-range transport (Azcune et al., 2022; UN Environment, 2019). In low-impact or protected areas, the detection of these contaminants is especially informative, as local point sources are generally absent or limited, making it possible to evaluate background levels and subtle regional contamination signals (Bolaños-Alvarez et al., 2024; Cooke et al., 2020; Muir & de Wit, 2010; Nipen et al., 2022).

Coastal lagoons are valuable but complex systems for this type of reconstruction. Their sedimentary records may integrate atmospheric deposition, catchment runoff, biological productivity, hydrological variability, sediment focusing, resuspension, and, in some cases, marine influence (Esteves & Monteiro Camargo, 2011; Last & Smol, 2001, 2002). Therefore, contaminant profiles in lagoon sediments should not be interpreted solely as direct records of emission intensity. Changes in sediment accumulation, grain-size distribution, organic matter content, freshwater–marine interactions, and extreme climatic events may also influence the concentration and preservation of contaminants (Smol, 2008). This is particularly relevant in shallow coastal lagoons, where small changes in hydrology or sediment dynamics can affect the temporal distribution of sediment-associated substances (Azcune, 2024).

In Uruguay, information on contaminants in aquatic environments is still limited and is mostly based on recent monitoring studies (Díaz et al., 2015; DINACEA et al., 2022; Griffero et al., 2019; Rodríguez-Bolaña et al., 2023). Paleoenvironmental reconstructions of organic contaminants and mercury remain scarce, despite the relevance of sediment archives for establishing baseline levels and assessing historical inputs. Southeastern Uruguay contains several coastal lagoons of high ecological and conservation value, many of them located within protected areas (Kruk et al., 2009; Ministerio de Ambiente, n.d.; Rodríguez-Gallego et al., 2003; UNESCO, 2001). These systems provide suitable conditions for evaluating whether low-impact environments record diffuse contaminant signals and for generating reference data for future regional assessments (Azcune et al., 2024; Blais et al., 2015; Smol, 2008; Valette-Silver, 1993).

Previous work on sediment core NUT1 from Nutrias Lagoon provided a validated chronological framework based on ^{210}Pb and ^{137}Cs , together with sedimentological, geochemical, and biological proxies (Azcune et al., 2024). That reconstruction identified changes in mass accumulation rate, particularly before and after the mid-1980s, as well as paleoenvironmental variations associated with regional precipitation and extreme events. PAH concentrations from this core have also been previously reported, showing detectable levels of selected compounds and an increase in lighter PAHs toward recent sediments (Azcune et al., 2024). These previously published PAH data provide contextual information for the present study, which extends the contaminant record of NUT1 through the independent determination of PCBs, PBDEs, and Hg in the same dated sediment sequence. By integrating these newly reported measurements with the established chronological and paleoenvironmental framework, this study provides a broader multi-contaminant baseline and enables comparison of the occurrence and temporal behavior of contaminant groups with contrasting sources, transport pathways, and environmental persistence.

The objective of this study is to evaluate baseline levels and low-intensity historical signals of PCBs, PBDEs, and total Hg in dated sediments from Nutrias Lagoon. Specifically, we aim to: (i) determine the occurrence of selected PCB and PBDE congeners and total Hg along the sediment core; (ii) interpret their distribution in relation to the available chronological and paleoenvironmental framework; (iii) assess whether the observed patterns are compatible with low-magnitude diffuse inputs in the absence of known local point sources, while considering sedimentary and hydrological controls; and (iv) compare the detected levels with sediment quality guidelines as a screening-level assessment of potential ecological relevance. By doing so, this work contributes to the development of regional baseline information for persistent contaminants in protected coastal lagoons of southeastern South America.

Materials and Methods

Study area

Nutrias Lagoon is a small, shallow coastal lagoon located in southeastern Uruguay (34°40'50.8" S, 54°17'42.0" W – Fig. 1), within the Laguna de Rocha Protected Landscape, which is part of the National System of Protected Areas (Ministerio de Vivienda Ordenamiento Territorial y Medio Ambiente, 2010). The lagoon has an area of 33.8 ha, a drainage basin of 44.7 ha, and a maximum depth of approximately 1.5 m (Kruk et al., 2009). Its catchment is predominantly covered by natural grasslands and scattered forest patches, with low-intensity livestock grazing as the main land use (Kruk et al., 2009). No urban settlements or industrial activities are located within the immediate basin, making the system suitable for evaluating baseline contaminant levels and diffuse environmental inputs (Azcune et al., 2024; de Álava et al., 2013).

Sediment core collection and subsampling

A 65 cm-long sediment core (NUT1) was collected in February 2018 from the deepest sector of Nutrias Lagoon (Fig. 1) using a manual push corer with an internal diameter of 15 cm. Immediately after collection, the core was sealed, refrigerated at 4°C, and transported to the laboratory. Subsampling was carried out under refrigerated conditions. The upper 30 cm were sectioned at 1 cm intervals, whereas the deeper part of the core was sectioned at 2 cm intervals. Sediment samples were stored under appropriate conditions until further analysis (Azcune et al., 2024). A total of 47 sediment intervals were analyzed.

Chronological and paleoenvironmental framework

The chronological framework used in this study was based on previously developed age-depth models for the same sediment core (Azcune et al., 2024). The chronology was obtained from ^{210}Pb and ^{137}Cs profiles and modeled using both the Constant Flux (CF) model (Appleby & Oldfield, 1978; Sanchez-Cabeza et al., 2012) and the Bayesian Plum model (Aquino-López et al., 2018, 2020). The $^{210}\text{Pb}_{\text{ex}}$ inventory was complete, allowing the application of the CF model. The total $^{210}\text{Pb}_{\text{ex}}$ inventory was $1841 \pm 59 \text{ Bq m}^{-2}$, and the estimated ^{210}Pb flux was $57 \pm 2 \text{ Bq m}^{-2} \text{ yr}^{-1}$, which is consistent with the atmospheric flux expected for the 30°–50°S latitudinal band. The chronology was validated using the ^{137}Cs fallout peak, which was recorded at 23–24 cm and assigned to 1966 ± 2 to 1969 ± 2 CE under the CF model (Azcune, 2024; Azcune et al., 2024).

Organic contaminant extraction and GC-MS/MS analysis

Acetone and n-hexane, both HPLC grade, were obtained from J.T. Baker. Anhydrous magnesium sulfate and sodium chloride were obtained from Merck. Certified reference material mixtures were purchased from AccuStandard (New Haven, CT, USA). The PCB standard solution (catalog no. AE-00059-R1-10ML) contained PCB 28, 52, 101, 118, 138, 153, and 180 in isooctane. The PBDE standard solution (catalog no. BDE-CSM) contained BDE 28, 47, 99, 100, 153, 154, 183, and 209 in isooctane:toluene (80:20, v/v). Triphenyl phosphate (TPP; Dr. Ehrenstorfer) was used as a surrogate compound.

PCBs and PBDEs were extracted from sediment samples using a modified QuEChERS procedure assisted by sonication (Anastassiades et al., 2003, 2007; Azcune et al., 2024; Pareja et al., 2022). For each sample, 10 g of raw sediment were placed in a 50 mL centrifuge tube and extracted with 10 mL of acetone:n-hexane (50:50, v/v). Samples were sonicated for 20 min. Subsequently, 4 g of MgSO_4 and 1 g of NaCl were added, and the tubes were manually shaken for 1 min and centrifuged for 10 min at 4000 rpm, corresponding to approximately $2600 \times g$, using a Sigma 2-6E centrifuge. An aliquot of 1 mL of the organic phase was filtered through a 0.22 μm PTFE filter and transferred to a chromatographic vial.

PCBs and PBDEs were analyzed using a Shimadzu TQ8040 GC-MS/MS system equipped with a Restek RXI-5MS capillary column (30 m $\times$ 0.25 mm i.d., 0.25 μm film thickness). Aliquots of 1 μL were injected in splitless mode at an injector temperature of 250°C. Helium was used as the carrier gas at a constant flow rate of 1 mL min^{-1} . The GC-MS/MS system was operated in multiple reaction monitoring mode, with ion-source and quadrupole temperatures of 230 and 150°C, respectively. Two MRM transitions per analyte were monitored for identification and quantification. Identification criteria was consistent with well-established SANTE 11312/2021 guidelines referring retention time matching with that of the corresponding matrix-matched calibration standard within ± 0.1 min, the chromatographic peaks obtained for both MRM transitions fully overlapped, and the relative MRM ion ratios in the sample was within $\pm 30\%$ of the average ratio obtained from calibration standards analyzed in the same sequence (European Commission, 2021).

Quantification was performed using five-point matrix-matched calibration curves prepared from the certified standard solutions described above. Limits of quantification were established as the lowest matrix-fortification level that fulfilled the recovery acceptance criteria described in the quality-assurance and quality-control section. For PCBs, the LOQs were 10 $\mu\text{g kg}^{-1}$ for all congeners except PCB 153, for which the LOQ was 1 $\mu\text{g kg}^{-1}$. For PBDEs, the LOQs were 1 $\mu\text{g kg}^{-1}$ for all congeners except BDE 183, for which the LOQ was 10 $\mu\text{g kg}^{-1}$. Signals that fulfilled the chromatographic and mass-spectrometric identification criteria but occurred below the corresponding LOQ were reported qualitatively as detected below the LOQ and were not assigned numerical concentrations. Although BDE 209 was present in the certified standard mixture, it was not included in the final determination because its MRM transitions could not be satisfactorily detected under the analytical conditions used.

Quality assurance and quality control for organic contaminants

Analytical quality assurance and quality control for PCB and PBDE determination included recovery assays, evaluation of matrix effects, surrogate recovery monitoring, matrix-matched calibration, and instrumental

stability checks throughout each analytical sequence. Recovery assays were performed in triplicate at different fortification levels. Performance was evaluated according to the SANTE criteria, with recoveries between 70% and 120% and relative standard deviations of $\leq 20\%$ considered acceptable (European Commission, 2021). Acceptable recoveries and precision were obtained at the fortification levels adopted as the LOQs, whereas performance at lower fortification levels was analyte-dependent (Table S1).

Matrix effects were assessed by comparing the slopes of calibration curves prepared in solvent with those of calibration curves prepared in sediment matrix, according to Cerqueira et al. (2014):

$$ME (\%) = 100 \times \frac{Slope_{matrix} - Slope_{solvent}}{Slope_{solvent}}$$

Based on the absolute ME value, matrix effects were classified as weak ($|ME| < 25\%$), moderate ($25\% \leq |ME| \leq 50\%$), or strong ($|ME| > 50\%$). Matrix-matched calibration was used for quantification to compensate for the observed matrix effects.

For PCB determination, TPP recoveries were within the established acceptance range throughout the analytical sequence, except for the sample corresponding to the 48–50 cm sediment interval, in which surrogate recovery was 0%. Consequently, PCB results from this interval were excluded from subsequent interpretation. Positive matrix effects were observed for all PCB congeners; the effect was moderate for PCB 101 and PCB 138 and strong for the remaining congeners. The end-of-sequence calibration check yielded recoveries of 87–94% relative to the initial calibration, indicating acceptable instrumental stability throughout the analytical run.

For PBDE determination, TPP recoveries remained within the established acceptance range throughout the analytical sequence, supporting adequate method performance. Positive matrix effects were observed for all PBDE congeners; the effect was weak for BDE 154 and moderate for the remaining congeners. BDE 209 was excluded from the final dataset because its diagnostic MRM transitions could not be satisfactorily detected under the analytical conditions used.

Mercury analysis

Total mercury concentrations were determined in dried and homogenized sediment samples using a DMA-80 direct mercury analyzer. The analytical method involved thermal decomposition of the sample under an oxygen atmosphere, followed by gold amalgamation, thermal desorption, and atomic absorption detection. Mercury concentrations were expressed as $\mu\text{g kg}^{-1}$ dry weight.

Quality assurance and quality control for Hg determination included analytical blanks and the certified reference material IAEA-456 marine sediment. The certified reference material was analyzed in seven replicates, yielding a mean recovery of 103% and a relative standard deviation of 1.84%, indicating satisfactory accuracy and precision.

Data processing and ecological thresholds

Temporal concentration profiles of PCBs, PBDEs, and Hg were interpreted according to the sediment chronology established for the core. Where applicable, measured concentrations were compared with

published sediment-quality guidelines, including the Threshold Effect Level (TEL) and Probable Effect Level (PEL), which were used as screening references for the potential occurrence of adverse biological effects on benthic organisms (Birch, 2011; Buchman, 2008; Long & MacDonald, 1998).

Previously published PAH concentrations from the same sediment core (Azcune et al., 2024) were also considered to provide an integrated interpretation of the occurrence and historical variation of different contaminant groups. These data were used to examine the co-occurrence of PAHs, PCBs, PBDEs, and Hg and to contextualize the overall contaminant profile recorded in the sediment sequence.

Results

The chronological framework previously established for core NUT1 was used to assign calendar ages to the sediment intervals analyzed in this study. Ages derived from the Constant Flux model (Appleby & Oldfield, 1978) were broadly consistent with those obtained using the Bayesian Plum model (Aquino-López et al., 2018) within their uncertainty ranges. The ^{137}Cs maximum recorded at 23–24 cm corresponded to 1966 ± 2 to 1969 ± 2 CE according to the Constant Flux model, supporting the chronology used for contaminant interpretation (Azcune, 2024; Azcune et al., 2024). Sediment mass accumulation rates remained relatively stable at approximately $0.091 \pm 0.003 \text{ g cm}^{-2} \text{ yr}^{-1}$ until the mid-1980s and increased to approximately $0.119 \pm 0.003 \text{ g cm}^{-2} \text{ yr}^{-1}$ after 1986. Additional accumulation maxima were recorded around 1914 ± 4 , 1956 ± 2 , 1964 ± 2 , and 1997 ± 1 CE (Fig. 2).

None of the seven PCB congeners included in the analytical method was quantified above its corresponding LOQ throughout the sediment core. PCB 52 was the only congener that produced signals fulfilling GC-MS/MS identification criteria, with qualitative detections recorded in the 2–3, 3–4, 4–5, 6–7, 7–8, 11–12, 25–26, 29–30, and 40–42 cm intervals. However, all detections remained below the LOQ and were therefore treated as qualitative occurrence data rather than quantitative concentrations. According to the Constant Flux chronology, one detection corresponded to 1920–1939, one to 1952–1962, one to 1960–1970, one to 1995–2000, and five to 2001–2015 CE (Fig. 3A). None of the seven PBDE congeners retained in the final analytical determination was detected in any sediment interval.

Total Hg concentrations ranged from 56 ± 6 to $85 \pm 9 \text{ } \mu\text{g kg}^{-1}$ dry weight, with an average concentration of approximately $75 \text{ } \mu\text{g kg}^{-1}$. The lowest concentrations occurred predominantly in the older section of the core, whereas values were slightly higher and comparatively stable in sediments deposited from approximately the mid-20th century onward. No pronounced concentration maxima were observed, and the overall variation remained limited relative to the analytical uncertainty (Fig. 3B). All measured Hg concentrations were below the Threshold Effect Level of $130 \text{ } \mu\text{g kg}^{-1}$ dry weight.

Discussion

The dated sediment record from Nutrias Lagoon indicates a low contaminant burden in a protected coastal system with no known urban or industrial point sources within its immediate catchment. None of the PCB congeners was quantified above the corresponding LOQ, no PBDEs included in the final analytical determination were detected, and total Hg concentrations remained within a relatively narrow range throughout the core. These findings support the use of Nutrias Lagoon as a reference system for evaluating persistent

contaminant occurrence in southeastern Uruguay. However, the protected status of the lagoon should not be equated with complete environmental isolation or pristine conditions. Protected and low-impact environments may still receive contaminants through atmospheric deposition, catchment runoff, regional redistribution, and other diffuse pathways (Azcune et al., 2022; Machate et al., 2023). The significance of the present record therefore lies not in demonstrating the absence of anthropogenic influence, but in establishing the magnitude and temporal variability of contaminant signals under conditions of limited local pressure.

Interpretation of these signals requires consideration of the sedimentary dynamics of the lagoon. Sediment cores preserve environmental information through accumulation, but measured concentrations do not necessarily represent direct records of changes in contaminant emissions or external loading. Variations in sediment supply, grain-size distribution, organic matter, hydrology, resuspension, and sediment focusing may alter contaminant concentrations and their preservation within the sediment column (Smol, 2008). This issue is particularly relevant in Nutrias Lagoon because the previously established chronology showed a change in mass accumulation rate around the mid-1980s, from approximately 0.091 to $0.119 \text{ g cm}^{-2} \text{ yr}^{-1}$, as well as several shorter accumulation maxima during the twentieth century (Azcune et al., 2024). An increase in sediment accumulation may dilute contaminant concentrations even if external inputs remain unchanged, whereas periods of reduced accumulation or enhanced focusing may produce relatively higher concentrations without a proportional increase in contaminant loading. Because contaminant fluxes were not calculated in the present study, concentration profiles must therefore be interpreted together with the chronological and sedimentary framework rather than as direct measures of historical emissions.

Total Hg showed the clearest measurable contaminant profile. Concentrations ranged from 56 ± 6 to $85 \pm 9 \text{ } \mu\text{g kg}^{-1}$ dry weight and were generally lower in the older part of the core, followed by a modest increase and comparatively stable values from approximately the mid-twentieth century onward. The profile lacked pronounced peaks, and much of its short-term variability was small relative to the analytical uncertainty. This pattern does not indicate a major episodic discharge or a strong local point source. Instead, it is compatible with low and relatively diffuse Hg inputs superimposed on changes in sediment accumulation and retention. Historical sediment records from other regions commonly show increases in Hg associated with twentieth-century anthropogenic emissions and subsequent atmospheric redistribution (Cooke et al., 2020). Dated sediment studies from Brazil and the Wider Caribbean have likewise demonstrated that Hg records may integrate local, regional, and atmospheric influences, with the relative contribution of each pathway depending on catchment characteristics, hydrology, sedimentation, and proximity to emission sources (Bolaños-Alvarez et al., 2024; Campos et al., 2022). In Nutrias Lagoon, the absence of known industrial and urban discharges makes diffuse atmospheric deposition and catchment transfer plausible pathways, but the available evidence does not permit source attribution.

The PCB results represent a qualitatively different type of record. PCB 52 was the only congener that fulfilled the identification criteria in selected sediment intervals, although all signals remained below the LOQ. These observations demonstrate that a low-level PCB signal was present in selected sediment intervals, but they do not provide quantitative concentrations or a basis for reconstructing a concentration trend. PCB 52 is a relatively low-chlorinated congener and is consequently more volatile and more readily transported through the atmosphere than highly chlorinated PCBs, making its occurrence environmentally plausible in a system lacking direct PCB sources (Hung et al., 2013; Montone et al., 2003; Muir & de Wit, 2010). Nevertheless, the

sedimentary detections should only be interpreted as evidence of low-level occurrence compatible with diffuse redistribution of legacy contaminants.

Five of the nine PCB 52 detections occurred in sediments assigned to the 2001–2015 CE interval, whereas only one detection was recorded in each of the earlier time ranges. Despite differences in sediment section thickness along the core, the chronological distribution therefore indicates a greater occurrence of low-level PCB 52 signals in the most recent sediments. This pattern may reflect environmental persistence and continued redistribution of PCBs following their widespread historical use (Melymuk et al., 2022). However, because all detections remained below the LOQ, the observed distribution should be interpreted as an increase in the frequency of qualitative occurrence signals rather than as evidence of increasing PCB 52 concentrations or depositional fluxes.

The absence of detectable PBDEs throughout core NUT1 indicates that these compounds did not constitute a measurable sedimentary contaminant signal in Nutrias Lagoon during the period represented by the record. This result is consistent with the protected and predominantly rural character of the catchment, where the absence of urban settlements, industrial activities, and waste-disposal sites limits the likelihood of substantial local PBDE inputs (Ohoro et al., 2021). In contrast to the measurable Hg profile and the qualitative occurrence of PCB 52, PBDEs showed no evidence of accumulation in the analyzed sediments. Although trace concentrations below the detection capability of the method cannot be excluded, the lack of detections supports the interpretation of Nutrias Lagoon as a low-impact reference system and provides a useful baseline for comparison with more urbanized or industrialized coastal environments.

The low contaminant burden recorded in Nutrias Lagoon contrasts with conditions reported for more impacted sectors of coastal and estuarine systems in the southwestern Atlantic region. In southern Brazil, Hg and hydrocarbon contamination has been documented in the Patos Lagoon estuary and adjacent coastal environments, particularly in areas influenced by urban sewage, industrial discharges, port activities, and other anthropogenic effluents (Medeiros et al., 2005; Mirlean et al., 2003; Neves et al., 2023). These regional studies do not demonstrate that contaminants detected in Nutrias Lagoon originated in southern Brazil, nor do they allow direct inference of transboundary transport. They do, however, show that substantial contaminant sources occur elsewhere within the broader southwestern Atlantic coastal region and that aquatic systems along this coast experience markedly different degrees of anthropogenic pressure. In this context, Nutrias Lagoon may provide a useful low-impact reference against which sedimentary records from more urbanized, industrialized, or intensively managed systems can be compared.

Previously published PAH data from the same core provide additional context for this interpretation. Unlike PCBs and PBDEs, several PAHs were quantified in the NUT1 record, and selected compounds showed temporal changes and occasional exceedances of sediment-quality screening values (Azcune et al., 2024). The different responses of PAHs, PCBs, PBDEs, and Hg demonstrate that contaminant groups should not be expected to produce synchronous or directly comparable sedimentary profiles. They differ in their sources, environmental persistence, transport mechanisms, partitioning behavior, degradation, and affinity for sedimentary organic matter (Cooke et al., 2020; Lohmann et al., 2007). The occurrence of detectable PAHs together with low Hg concentrations, qualitative PCB 52 signals, and non-detected PBDEs is therefore more appropriately interpreted as a heterogeneous contaminant record than as evidence of a single common source or contamination event. Changes in sediment accumulation, organic matter content, and hydrological

conditions documented in the NUT1 record may also contribute to the apparent correspondence or divergence among these profiles (Azcune et al., 2024). Differences in analytical sensitivity among contaminant groups should also be considered when comparing their sedimentary records.

An integrated comparison of contaminant occurrence in core NUT1 with the available sediment-quality screening values is presented in Table 1. This comparison should be interpreted as a screening-level assessment because the different benchmarks apply to specific compounds or contaminant sums and do not constitute site-specific predictions of ecological effects.

Table 1
Summary of contaminant occurrence in sediments of Nutrias Lagoon and comparison with selected sediment-quality screening values.

Contaminant group	Compound(s) detected	Maximum concentration (µg kg ⁻¹)	Ecotoxicological threshold (µg kg ⁻¹)	Sediment-quality guideline source	Screening-value assessment
PCBs (n = 7)	Only PCB 52 detected sporadically, always < LOQ	-	TEL (Σ _{PCBs}) = 34.1	NOAA (Buchman, 2008)	Exceedance could not be determined
PBDEs (n = 7)	Not detected	Not applicable	No screening value selected	–	Not assessed
Mercury (Hg)	Total Hg	85	TEL = 130	NOAA (Buchman, 2008)	No exceedance
PAHs (total)	Σ24 PAHs (9 detected; 6 > LOQ)	~ 1300	TEC (Σ _{PAHs}) = 1610	NOAA (Buchman, 2008)	No exceedance
PAHs (individuals)	Naphthalene	~ 200	TEL = 34.6	NOAA (Buchman, 2008)	TEL exceeded in upper layers
	Indeno[1,2,3-cd]pyrene	~ 220	LEL = 200	NOAA (Buchman, 2008)	Slight exceedance at depth
	Dibenzo[a,h]anthracene	~ 100	PEL = 135	CCME (1999)	No exceedance

LOQ, limit of quantification; TEL, threshold effect level; TEC, threshold effect concentration; LEL, lowest effect level; PEL, probable effect level. PAH concentrations and their temporal distribution were previously reported by (Azcune et al., 2024). Screening values represent screening-level benchmarks and should not be interpreted as site-specific toxicity thresholds.

From an ecological perspective, total Hg concentrations remained below the marine/estuarine TEL of 130 µg kg⁻¹ dry weight throughout the core, with a maximum concentration of 85 µg kg⁻¹. Within the sediment-quality guideline framework, adverse biological effects associated specifically with Hg would therefore be expected to occur only infrequently at the concentrations recorded in Nutrias Lagoon (Birch, 2011; CCME, 1999; Long & MacDonald, 1998). This comparison provides a useful screening-level assessment but should not be

interpreted as a site-specific prediction of ecological effects, because guidelines based on total contaminant concentrations do not fully account for local differences in contaminant bioavailability, sediment composition, benthic community sensitivity, exposure duration, or interactions among contaminants (Conder et al., 2014).

For PCBs, the TEL of $34.1 \mu\text{g kg}^{-1}$ applies to the sum of the same seven congeners determined in this study. However, $\Sigma 7\text{PCBs}$ could not be quantified because all congeners remained below their method-specific LOQs. PCB 52 was detected sporadically above the LOD but always below its LOQ of $10 \mu\text{g kg}^{-1}$, whereas the other six congeners were not detected under the applied analytical conditions. Based solely on the individual LOQs, the most conservative upper bound for $\Sigma 7\text{PCBs}$ would be $61 \mu\text{g kg}^{-1}$, which is higher than the TEL. Consequently, exceedance of the guideline cannot be formally excluded from the available quantitative data. Nevertheless, this upper bound is highly conservative because six of the seven congeners were not detected and, if present, occurred below their respective LODs. The results therefore indicate a low measurable PCB burden, although a definitive comparison with TEL would require a numerical upper-bound estimate based on the compound-specific LODs. Likewise, the absence of detected PBDEs indicates that the analyzed congeners did not produce a measurable signal under the applied analytical conditions, rather than demonstrating their complete environmental absence or the absence of ecological risk.

The PAH results provide a more differentiated ecological interpretation. The maximum concentration of $\Sigma 24$ PAHs, approximately $1300 \mu\text{g kg}^{-1}$, remained below the TEC of $1610 \mu\text{g kg}^{-1}$. However, the evaluation of individual compounds identified localized exceedances of screening values. Naphthalene reached approximately $200 \mu\text{g kg}^{-1}$ and exceeded its TEL of $34.6 \mu\text{g kg}^{-1}$ in the upper sediment layers, while indeno[1,2,3-cd]pyrene reached approximately $220 \mu\text{g kg}^{-1}$ and slightly exceeded its LEL of $200 \mu\text{g kg}^{-1}$ at depth. In contrast, the maximum dibenzo[a,h]anthracene concentration, approximately $100 \mu\text{g kg}^{-1}$, remained below its PEL of $135 \mu\text{g kg}^{-1}$ (Azcune et al., 2024; Buchman, 2008; CCME, 1999). Taken together, the sediment record indicates limited ecological concern associated with Hg and a low measurable burden of the analyzed PCB and PBDE congeners. Nevertheless, the exceedances observed for individual PAHs show that the absence of a total-PAH threshold exceedance does not preclude compound-specific ecological concern in particular sediment intervals.

Core NUT1 records a coherent low-impact contaminant profile. Total Hg remained below the selected sediment-quality screening value, PCB 52 occurred only as sporadic sub-LOQ signals, and none of the seven PBDE congeners retained in the final method was detected. When considered alongside the previously published PAH data, however, the record is heterogeneous rather than uniformly contaminant-free, because selected PAHs showed localized exceedances of screening values (Azcune et al., 2024). Nutrias Lagoon should therefore be regarded not as a pristine system, but as a protected lagoon with limited measurable contaminant inputs and no evidence of a dominant local point source. Its main contribution is to provide a dated reference against which sedimentary records from coastal lagoons exposed to stronger urban, industrial, or agricultural pressures can be evaluated.

Conclusions

The dated sediment record indicates that Nutrias Lagoon is a low-impact coastal system, but not an environmentally isolated or contaminant-free environment. The low-intensity and heterogeneous signals observed among contaminant groups do not support the occurrence of a dominant common source or a major

local contamination event. Instead, they are compatible with low-magnitude diffuse inputs, compound-specific transport and persistence, and subsequent redistribution within the lagoon. However, the available evidence does not support direct attribution to specific atmospheric, regional, or catchment sources.

From a sediment-quality perspective, the record does not indicate widespread concern associated with the contaminant groups evaluated. Total Hg remained below the selected screening value, and the total PAH burden remained below the corresponding benchmark. Nevertheless, localized exceedances for individual PAHs demonstrate that a low overall contaminant burden does not preclude compound-specific concern in particular sediment intervals. For PCBs, the available analytical data were insufficient to determine conclusively whether the total-PCB screening value was exceeded, whereas PBDEs could not be evaluated against a selected sediment-quality benchmark. The ecological interpretation should therefore remain compound-specific and be regarded as a screening-level assessment rather than as evidence of the absence of adverse effects.

The contrasting behavior of Hg, PCBs, PBDEs, and previously reported PAHs also demonstrates that contaminant histories in shallow coastal lagoons cannot be interpreted solely as records of changes in external inputs. Sediment accumulation, hydrological variability, resuspension, contaminant retention, and analytical sensitivity may influence the signals ultimately preserved in the sediment column. Consequently, chronological information must be interpreted together with sedimentary and paleoenvironmental processes when reconstructing contaminant histories in these systems.

Nutrias Lagoon should therefore be considered a low-impact sedimentary reference rather than a pristine reference site. Its main value lies in providing a dated benchmark against which future changes in sediment quality can be evaluated and sedimentary records from coastal lagoons exposed to stronger urban, industrial, or agricultural pressures can be compared. More broadly, this study shows that protected environments can provide valuable reference conditions for defining regional contaminant baselines and distinguishing subtle diffuse influences from the stronger signatures associated with direct anthropogenic sources.

Declarations

Declarations

All authors have read, understood, and have complied as applicable with the statement on “Ethical responsibilities of Authors” as found in the Instructions for Authors.

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Author Contribution

G.A. conceived the study, conducted the overall investigation, analyzed and interpreted the data, and wrote the original draft of the manuscript. N.G.G. and H.H. contributed to the development and implementation of the GC–MS/MS methodology and to the associated analytical work. C.M.A.-H. contributed to the mercury analysis. L.F. and A.P.P. supervised the research and critically reviewed and edited the manuscript. All authors reviewed and approved the final version of the manuscript.

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Data Availability

The data generated and analyzed during the current study are available from the corresponding author upon reasonable request.

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Figures

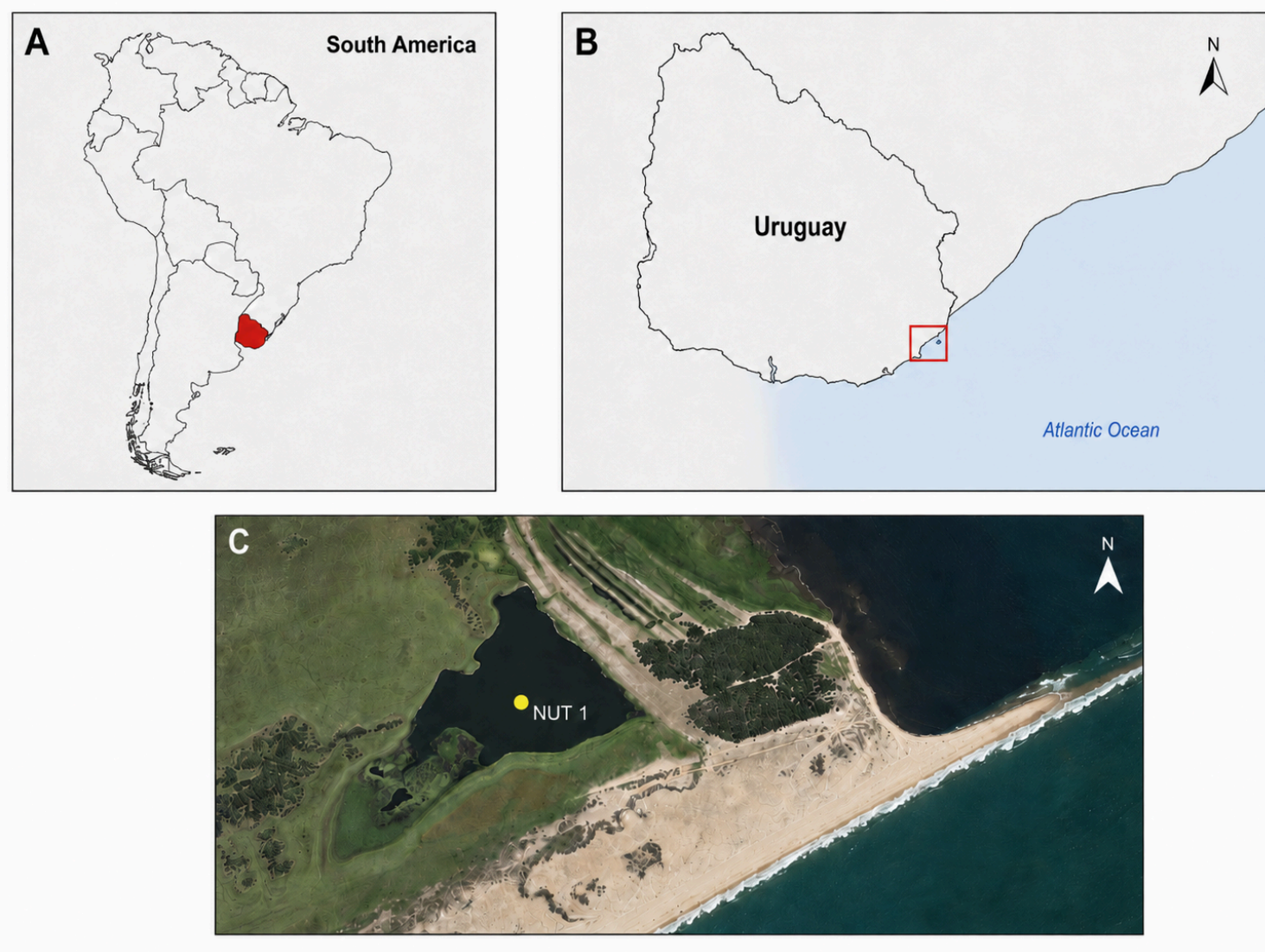


Figure 1

Study area geographic location.

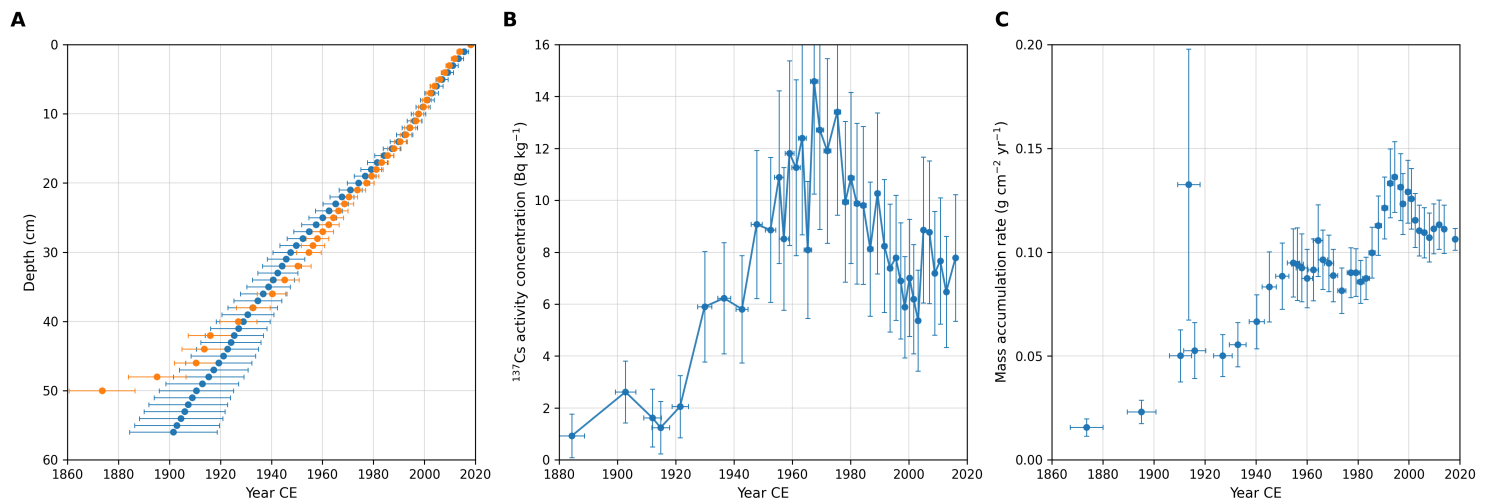


Figure 2

Chronological and sediment accumulation framework for sediment core NUT1. (A) Age–depth relationships obtained using the Constant Flux (CF) and Bayesian Plum models; (B) ^{137}Cs activity profile used to support the sediment chronology; and (C) mass accumulation rate as a function of calendar year. Chronological data and models were previously reported by Azcune et al. (2024) and were redrawn here to support the interpretation of contaminant profiles.

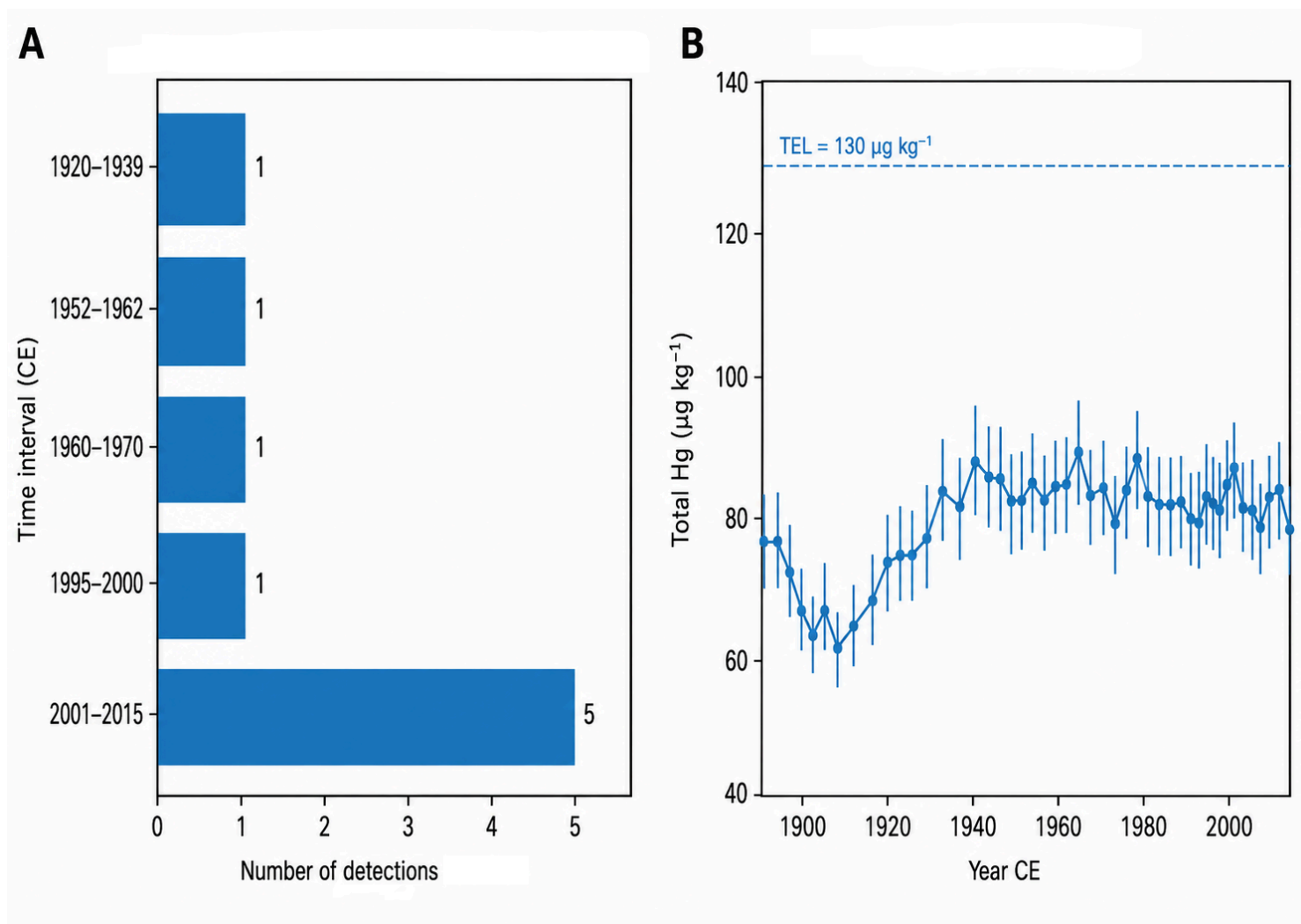


Figure 3

Occurrence of PCB 52 and temporal profile of total mercury in sediment core NUT1. (A) Number of PCB 52 detections grouped by calendar-year intervals according to the Constant Flux chronology. All PCB 52 signals were above the limit of detection but below the limit of quantification and are therefore presented as qualitative occurrence data. (B) Total mercury concentration as a function of calendar year, with analytical uncertainty bars and the Threshold Effect Level (TEL; $130 \mu\text{g kg}^{-1}$ dry weight) shown as a horizontal reference line.

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