

Past and present platinum contamination of a major European fluvial–estuarine system: Insights from river sediments and estuarine oysters



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ABSTRACT

Platinum Group Metals (PGM) are modern, technologically relevant elements for which (i) the anthropogenic cycle has outcompeted the natural cycles and (ii) environmental behavior, fate and impact are still widely unknown. Stripping voltammetry was used for accurate determinations of platinum (Pt) in historical records of river sediments and estuarine oysters from the Gironde fluvial–estuarine continuum (SW France) comprising the Lot River. Sediment cores from the Lot River, dated from 1952 to 2001, showed past Pt contamination due to former industrial (smelter) activities in the Lot River watershed. These samples revealed the phasing-out of a historical Pt contamination with Pt/Th (Thorium) values of $11 \times 10^{-5} \pm 0.79 \times 10^{-5}$ for the deepest part of the core which is clearly greater than the regional geochemical background value ($Pt/Th \sim 2.2 \times 10^{-5} \pm 0.68 \times 10^{-5}$). Wild oyster samples from the mouth of the Gironde Estuary collected from 1981 to 2013 showed Pt concentrations ranging from $0.80 \pm 0.01 \text{ pmol.g}^{-1}$ to $3.10 \pm 0.14 \text{ pmol.g}^{-1}$. Oyster samples have recorded the phasing-out of the smelter-related historical industrial Pt contamination and empirical modeling suggests the recent rise of a new source of Pt to the system. Temporal variations of Pt in oysters attributed to this recent source reflect the exponential increase of Pt demand for car catalytic converters, pointing towards the increasing importance of this emerging source to the aquatic system. Estuarine oysters prove to be suitable bioindicators for Pt contamination providing sensitive monitoring of emission variations over time. Furthermore, oysters may bioconcentrate Pt (bioconcentration factor, BCF $\sim 10^3$) and transfer this metal contamination to the higher food chain. These findings highlight the need for a deeper understanding of environmental Pt contamination, processes and possible adverse effects to biota.

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1. Introduction

Platinum (Pt), one of the rarest elements in the Earth's crust with a typical concentration of 2.6 pmol.g^{-1} (Rudnick and Gao, 2003), is a strategic commodity for industries in many countries (Reith et al., 2014; Cobelo-García et al., 2015) since its physical (density, resistance to corrosion) and chemical (e.g. catalyst) properties serve in many applications such as laboratory and medical equipment or (medical) drugs. The most extended application is the use of Pt-based vehicle catalytic converters which contain from 1 – 2 g for a small car to 12 – 15 g of Pt for a big truck (Tollefson, 2007), representing more than 70% of the total European Pt demand in 2013 (Johnson Matthey, 2014). Therefore these devices are recognized as a major source of Pt to the environment and

together with the burning of fossil fuels, mining and other industrial uses has led to a complete disturbance of the global Pt cycle (Sen and Peucker-Ehrenbrink, 2012). The significant recent increase of Pt concentrations in Antarctic snow (Soyol-Erdene et al., 2011) suggests large-scale atmospheric transport of Pt (Barbante et al., 2001) implying its global contamination. Major concerns on ecosystems and biota contamination arose few years after introducing car catalyst technology in Europe (e.g. Schäfer et al., 1999) and today anthropogenic Pt enrichment is recorded in all Earth compartments: atmosphere, water, sediments, and soils (e.g. Pawlak et al., 2014). Accordingly, Pt is considered an “emerging pollutant” for which it is necessary to develop innovative techniques providing reliable quantitative estimates of environmental pathways, loads, and concentrations (Rodrigues et al., 2009). To date, however, there is still a lack of data on environmental Pt concentrations mostly due to the relative difficulty to measure this element at low ambient levels. Recent work on Pt behavior in the Gironde Estuary has shown that estuarine reactivity along the salinity gradient is crucial for both the Pt distribution in the estuary and dissolved Pt export to the global ocean (Cobelo-García et al., 2014a). The net dissolved Pt addition due to estuarine mixing results in a higher export of dissolved Pt to the ocean than expected from the

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riverine input (Cobelo-García et al., 2014a). However, Pt sources and possible contamination of this system are still unknown. The present work aims at identifying past and present Pt contamination and sources by combining the first historical records of Pt in river sediments and estuarine oysters from the Gironde fluvial–estuarine system.

2. Material and methods

2.1. Study area

The Gironde Estuary (about 170 km in length), draining a 80,000 km² watershed, is a major European estuary considered a model for physical, hydrological and geochemical studies especially on trace element transport and reactivity in impacted watersheds (e.g. Lanceleur et al., 2011). The Lot–Garonne–Gironde fluvial–estuarine system is affected by multi-metal pollution originating from a common main point source being a former Zn-ore mining/metallurgic industry (e.g. 1842–1987: Cd, Zn, Cu, Pb; Audry et al., 2004a) on the Riou-Mort River near Decazeville (Fig. 1). After transport, trace metals can settle down and be deposited in river sediments where they accumulate under favorable hydraulic conditions provided by natural and reservoir lakes (Audry et al., 2004a). In 2001, two continuous sediment cores of about 1.4 m-long were collected using a manual corer consisting of a 10 × 10 cm rectangular Plexiglas tube. Sampling was realized in two hydroelectric reservoirs of the Lot River: the reservoirs of Marcenac and Cajarc located respectively upstream and downstream the Riou-Mort River i.e. the hypothetical Pt pollution source (Fig. 1). The sediment cores were sliced in thin horizontal sections at 1 cm resolution from the surface to 25 cm. The lower part was sampled with a 5 cm resolution. Immediately after recovery, each sliced section was centrifuged in order to remove pore waters and then sealed in sampling-bags under nitrogen. Sediment samples were dried at 50 °C to constant weight and then powdered and homogenized with an agate mortar

(Audry et al., 2004a). The second set of samples consisted of a historic series of wild oysters which, as suspension feeders, are exposed to both dissolved metals and metals bound to mineral particles such as phytoplankton and organic matter (Lanceleur et al., 2011) and therefore represent good bioindicators of their environment (Gunther et al., 1999). A time series (1981–2013) of mature (2 year old) wild-growing *Crassostrea gigas* Japanese oyster samples was extracted from the National Network for the Observation of Marine Environment Quality (RNO/ROCCH; i.e. the French Mussel-Watch; Ifremer.fr) specimen bank. Winter (February) oyster samples were chosen for the present work in order to avoid dilution of metal concentration due to variable soft-body weight during reproduction periods (Enríquez-Díaz et al., 2009). Individuals selected for this study originated from the mid-salinity range of the Gironde Estuary, at the La Fosse sampling site (salinity 15–20; Fig. 1), where highest trace metal concentrations are recorded in the same set of oyster samples (e.g. Cd and Ag; Lanceleur et al., 2011). A pool of fifteen individuals for each winter has been selected, lyophilized and grinded.

2.2. Analytical procedure

Samples were ashed in quartz crucibles at 800 °C during 3 h according to the heating scheme described by Nygren et al. (1990) and then digested using a mixture of 5 mL concentrated hydrochloric acid HCl and 3 mL of concentrated nitric acid HNO₃ (both Suprapur®, Merck) at 195 °C for 4 h. Then, samples were allowed to cool down, caps were removed and the acid was evaporated until near dryness. The residue was then redissolved adding 1 mL of concentrated sulfuric acid H₂SO₄ (TraceSELECT®, Fluka), and evaporated again until no fumes were observed (i.e. only H₂SO₄ was present). Cooled contents were then diluted with 0.1 M HCl. Sediment samples were syringe-filtered using PFA syringes (Savillex®) and 25 mm polyethersulfone membranes of 0.45 µm pore size (VWR®) prior to analyses. Platinum measurements

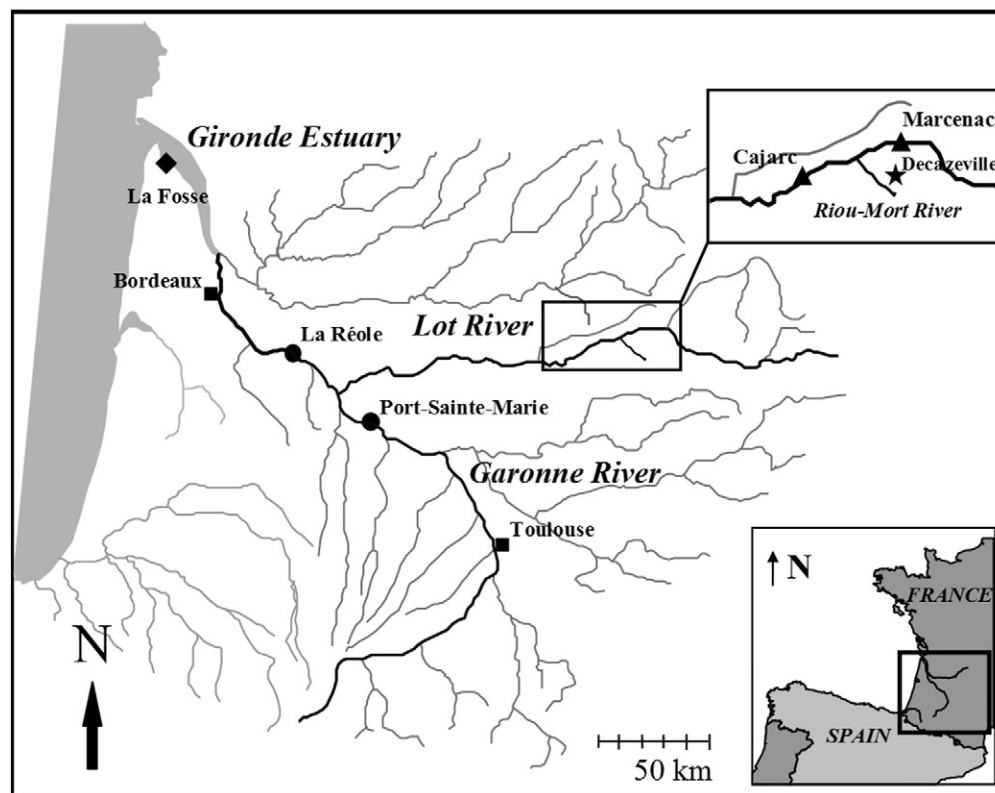


Fig. 1. The Gironde fluvial–estuarine continuum (SW France) with sampling locations: oyster samples were collected in the Gironde Estuary mouth at La Fosse (diamond) and sediment cores were retrieved in the Lot River in Cajarc and Marcenac (triangles). Suspended Particulate Matter (SPM) samples were collected in the Garonne River at the La Réole and Port Sainte Marie sites and analyzed in a previous study (round symbols, Cobelo-García et al., 2014a).

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