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Short Communication

## Fragrances in the seawater of Terra Nova Bay, Antarctica

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## ABSTRACT

Personal Care Products are emerging pollutants whose distribution in the Antarctic and remote environments is still largely unknown. Among PCPs, long-lasting and stable Fragrance Materials were selected to perform a first pilot study on their occurrence in the coastal surface seawater of Terra Nova Bay in the Ross Sea, Antarctica. Ambroxif, Amyl Salicylate, Benzyl Salicylate, Hexyl Salicylate, Lemonile and Okoumal were detected for the first time in Antarctic natural seawater, and reached total concentrations up to  $100 \text{ ng L}^{-1}$ . Treated discharges from the Italian research station Mario Zucchelli (MZS) contain FMs, however concentrations in nearby Tethys Bay increase during the seasonal melt of the sea ice and its snow cover: variability in emissions and distribution, as well as a contribution from atmospheric (long or short-range) transport were hypothesized.

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### 1. Introduction

The influence of the presence of humans in the Antarctic is of concern and is a heavily debated issue in environmental polar research. Far from the popular idea of a pure and pristine continent, the Antarctic environment is affected by the presence of pollutants deriving from both Long-Range Atmospheric Transport (LRAT) and local sources. Although contamination from Persistent Organic Pollutants (POPs) was recognized decades ago, there is a paucity of data about the presence of emerging pollutants, such as Personal Care Products (PCPs) in polar environments (Emnet et al., 2015). Recently the Scientific Committee on Antarctic Research (SCAR) identified the need to study how next-generation contaminants will affect Antarctic and Southern Ocean ecosystems as one of the highest priority scientific questions that researchers should aspire to answer in the next two decades and beyond (Kennicutt et al., 2014).

Fragrance Materials (FMs) are omnipresent ingredients of the majority of household and PCPs and despite their widespread use, little is known about the environmental fate of these compounds. The literature on the synthetic musks is an example of the concerns linked to the usage and discharge of FMs into the environment (Daughton and Ternes, 1999). Musks were also detected in Antarctica (Schiavone et al., 2009), in the Arctic (Xie et al., 2007) and in Alpine regions (Ferrario et al., 2017; Schmid et al., 2007), demonstrating that FMs could affect remote areas. Research stations and facilities constitute

local sources of contamination influencing the Antarctic environment (Hale et al., 2008; Vecchiato et al., 2015a, b). PCPs deriving from human activities were detected in the Antarctic Peninsula (Esteban et al., 2016) and in coastal seawater at distances up to 25 km from the McMurdo and Scott research stations (Emnet et al., 2015).

The present study was focused on 17 of the longest-lasting and most stable fragrance ingredients that are commercially available (Vecchiato et al., 2016). More precisely, the trade names (Givaudan®) of the selected FMs are: Amberketal, Ambroxif, Amyl Salicylate, Benzyl Salicylate, Bourgeonal, Dupical, Hexyl Salicylate, Isobutavan, Lemonile, Mefranal, Myraldene, Okoumal, Oranger Crystals, Pelargene, Peonile, Tridecene-2-Nitrile, Ultravaniil (Table S11). These FMs have in common a long persistence as fragrances (tenacity on blotter) ranging from a few weeks to months and can be used in commercial products which present very different and aggressive characteristics, such as liquid bleach and acid cleaners (eindex.givaudan.com). Their chemical stability and persistence as fragrances are possible indications of their persistence in the environment, and their semi-volatile features (vapour pressures ranging from 0.0004 to 1.19 hPa; Table S11) might promote their atmospheric transport and remote deposition. A similar process was hypothesized and experimentally demonstrated for musk FMs (Villa et al., 2014). The selected fragrances were recently found as contaminants in the Venice Lagoon: sewage outlets largely emit these FMs into the surface seawater, reaching total concentrations higher than  $10 \mu\text{g L}^{-1}$  in the innermost canals, however traces were found also in samples from background areas far from direct discharges (Vecchiato et al., 2016).

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This paper reported the first detection in environmental samples of most of the selected FMs.

Salicylates are the only FMs among the 17 selected with limited literature reporting their environmental occurrence. During the last years the consumption of Benzyl, Hexyl and Amyl Salicylates has grown rapidly because of their low prices (under \$5/kg), reaching in 2010 an estimated worldwide annual consumption of respectively 8500, 7000 and 6800 metric tons (Gaudin, 2014). For comparison purposes, in 2000 the worldwide use of polycyclic musks was 4000 metric tons (Villa et al., 2014). Benzyl Salicylate is also used as a UV-filter agent (Kameda et al., 2011) and is included in the EU list of allergenic fragrances (Heisterberg et al., 2011). Moreover it was recently found to have an oestrogenic activity comparable to Bisphenol A (BPA) (Zhang et al., 2012) and it gives oestrogenic responses in human breast cancer cells (Charles and Darbre, 2009). Benzyl and Hexyl Salicylates were measured in U.S., European and Japanese wastewater treatment plants (WWTPs) (Godayol et al., 2015; Kameda et al., 2011; Negreira et al., 2010; Simonich et al., 2002, 2000). Fewer data are reported about the occurrence of Benzyl Salicylate in river waters (Kameda et al., 2011; Negreira et al., 2010; Vila et al., 2016), while it was analyzed for in Japanese seawater samples, but not detected (Tashiro and Kameda, 2013). Benzyl Salicylate was also detected in indoor air (Lamas et al., 2010), due to its large use as a fragrance.

The aim of this work is to perform the first exploratory study to evaluate the occurrence and distribution of selected FMs in the coastal marine environment of Terra Nova Bay, Antarctica.

## 2. Materials and methods

Surface seawater samples (Table SI3) were collected during the austral summer of 2014–2015 in solvent-rinsed glass bottles at the sites shown in Fig. 1. Samplings started in mid-November through holes in the pack ice in Tethys Bay at TBa (74°42.052'S-164°02.267'E) and TBb (74°41.504'S-164°02.351'E) and ended in

mid-December, due to safety reasons as the pack was thinning. These samples therefore cover the period from the early to the advanced seasonal melting of the sea ice and of its snow cover. Later, sample TBa8 was collected from the shore facing TBa. For comparative purposes the drainage of the Wastewater Treatment Plant (WW) of the Italian research station Mario Zucchelli (MZS) and the receiving surface seawater of the Road Bay (RB1, RB2) were collected. About 70 people were permanently present in the base during the sampling period, with a mean daily production of desalted freshwater of about 11 m<sup>3</sup>, which results in a similar flow of treated wastewater.

Full analytical details of the method development were described previously (Vecchiato et al., 2016). Briefly, water samples were spiked with Phenanthrene <sup>13</sup>C as an internal standard (CLM-2451, Cambridge Isotope Laboratories Inc., Andover, MA USA) and extracted using 200 mg Oasis® HLB cartridges (Waters Corp., Milford, MA USA) previously conditioned with 10 mL of dichloromethane followed by 10 mL of ultrapure water (ELGA, High Wycombe, UK). Pesticide-grade dichloromethane, toluene and acetone (Romil Ltd., Cambridge, UK) were used. Samples were eluted with 1 mL of toluene followed by 15 mL of dichloromethane and were dried with an aliquot of Na<sub>2</sub>SO<sub>4</sub>. Volumes were reduced to 250 µL under a gentle nitrogen flow at 23 °C (Turbovap II®, Caliper Life Science, Hopkinton, MA, USA) and the instrumental analysis was conducted by GC–MS (7890A-5975C, Agilent Technologies, Avondale, USA) in Single Ion Monitoring (SIM) mode of two ions for quantification and confirmation purposes (Vecchiato et al., 2016). Since most of the 17 FMs are not available at analytical or reagent grade, all the following results are expressed as a concentration of the commercial product. Crude concentrations were corrected using the instrumental response factors and results are mean-blank corrected. Field blanks (n = 3) were conducted extracting 0.5 L of ultrapure water (ELGA) and the Method Detection Limit (MLD) was calculated as three times the standard deviation of the blank signal. Details are reported in Table SI2.

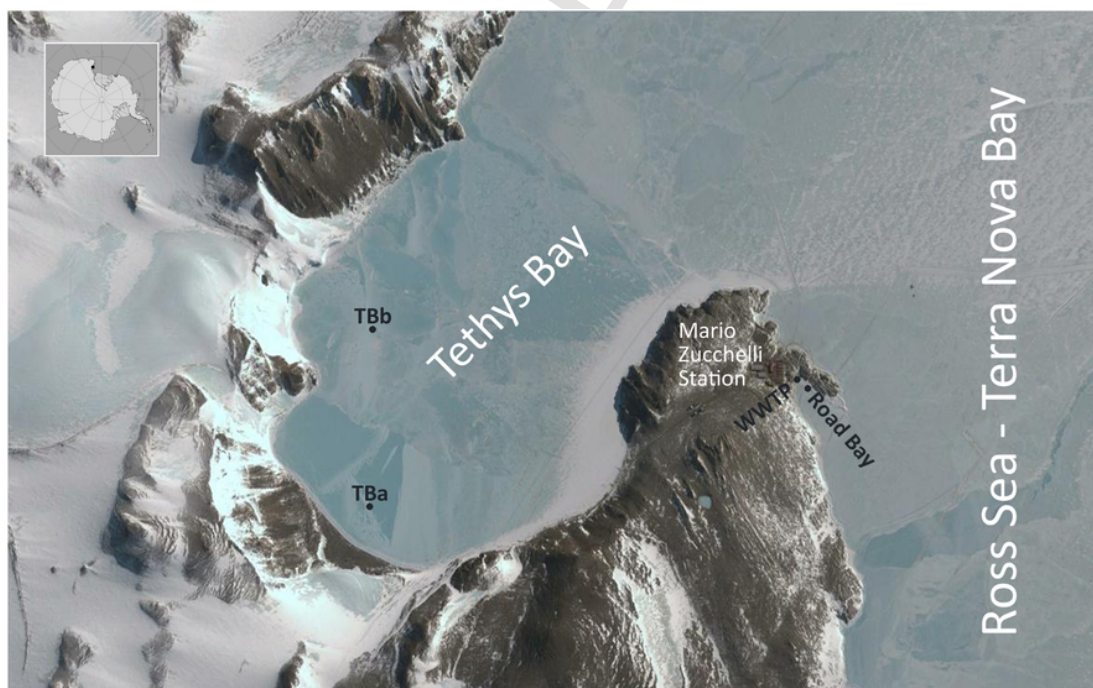


Fig. 1. Sampling locations of coastal surface seawater in Terra Nova Bay, Antarctica.

### 3. Results and discussion

FMs were detected in surface seawater with the sum of the concentrations ranging from below detection limits to  $100 \text{ ng L}^{-1}$  (Table SI3). 11 fragrances were never detected in any sample (Amberketal, Bourgeonal, Dupical, Isobutavan, Mefranal, Myraldene, Oranger Crystals, Pelargene, Peonile, Tridecene-2-Nitrile, Ultravani), therefore these compounds were excluded from the following discussion.

FMs collected at TBa show an increasing trend from the early samplings to mid-December, with concentrations starting from few  $\text{ng L}^{-1}$  (TBa1–2) and reaching the maximum cumulative value of  $100 \text{ ng L}^{-1}$  in the sample TBa7 (Fig. 2). Concentrations in samples TBa3 to 6 were at intermediate levels ( $18\text{--}64 \text{ ng L}^{-1}$ ). After one month, once the snow cover and most of the pack ice melted, the FMs resulted considerably lower in sample TBa8 ( $5 \text{ ng L}^{-1}$ ), the values of which were similar to the samples early in the season. The samples collected at TBb in early November (11/08–15/14) resulted  $< \text{MDL}$  in both samplings and no further samples were available from this site. As expected, the highest concentrations were found in the sample WW from the treated discharge of the base ( $156 \text{ ng L}^{-1}$ ; Table SI3), while the facing sample of surface seawater RB resulted considerably lower (RB1:  $5.3 \text{ ng L}^{-1}$ ; RB2:  $17 \text{ ng L}^{-1}$ ), probably reflecting dilution of the drainage of the treatment plant.

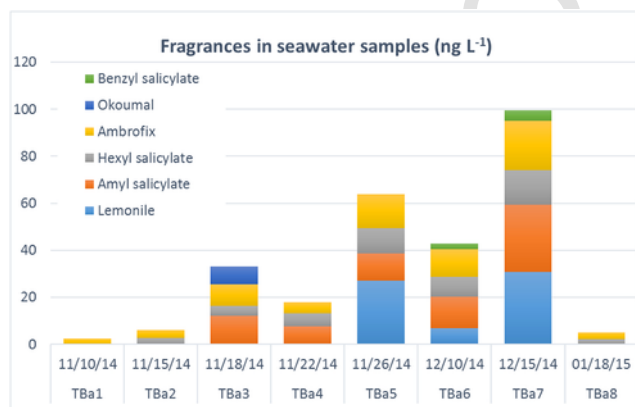
Amyl and Hexyl Salicylate were among the most frequently detected compounds (respectively  $4.2\text{--}29$  and  $2.1\text{--}28 \text{ ng L}^{-1}$ ), with trends similar to Ambroxif ( $2.5\text{--}54 \text{ ng L}^{-1}$ ). These substances were also widespread in the Venice Lagoon (Vecchiato et al., 2016). In contrast, Benzyl Salicylate was detected only in WW and in the samples from mid-December ( $2.2\text{--}4.5 \text{ ng L}^{-1}$ ) with concentrations significantly lower than Amyl and Hexyl Salicylate. Lemonile showed a similar behavior, being detected in TBa5 to 7, RB2 and WW ( $6.9\text{--}38 \text{ ng L}^{-1}$ ) but with levels higher than Benzyl Salicylate. Okoumal was found only in WW ( $5.0 \text{ ng L}^{-1}$ ) and occasionally in TBa3 ( $7.7 \text{ ng L}^{-1}$ ).

The concentrations of FMs reported here are comparable to different PCPs analyzed in other Antarctic areas: concentrations of up to  $88 \text{ ng L}^{-1}$  of UV-filters, preservatives, antimicrobials and Bisphenol A were detected in the coastal seawater near McMurdo and Scott bases (Emnet et al., 2015). Similar levels of several PCPs were reported in streams and ponds of the Northern Antarctic Peninsula (total concentrations ranging from  $145$  to  $370 \text{ ng L}^{-1}$ ), but with the highest proportions corresponding to organophosphate flame retardants and alkylphenols (Esteban et al., 2016). Comparing with musk

fragrances in other remote areas, dissolved Galaxolide (HHCB) and Tonalide (AHTN) concentrations were significantly lower in seawater collected from the Arctic and the North Sea with median values respectively of  $59$  and  $23 \text{ pg L}^{-1}$  (Xie et al., 2007). The same compounds resulted higher in Alpine snow samples (mean values HHCB =  $3.14 \text{ ng L}^{-1}$ ; AHTN =  $3.88 \text{ ng L}^{-1}$ ) (Villa et al., 2014) and more similar to the levels of FMs here presented.

FM concentrations in samples WW and RB1 and 2 are much lower than urban surface waters receiving wastewater discharges (Vecchiato et al., 2016), therefore the emission of FMs from MZS is considerably lower than those found in polluted environments. Only one test sample was collected from the WWTP and it may not be representative of the mean and overall emissions from this plant, but demonstrates that the base is a source of FMs into the Antarctic environment. The concentrations reported here for Hexyl and Benzyl Salicylate are respectively comparable and lower to the final effluent emitted from treatment plants ( $15 \pm 4$  and  $117 \pm 23 \text{ ng L}^{-1}$ ) (Simonich et al., 2000). Research stations are sources of PCPs to the Antarctic environment: UV-filters and Bisphenol A have been detected at concentrations ranging between tens to hundreds of  $\text{ng L}^{-1}$  in the WWTPs discharges of McMurdo and Scott bases (Emnet et al., 2015). Focusing on surface waters, the levels of Benzyl Salicylate in the samples TBa6 and TBa7 ( $2.2\text{--}4.5 \text{ ng L}^{-1}$ ) are the lowest among those reported in the literature: in Italy ( $< 10.7\text{--}2400 \text{ ng L}^{-1}$ ) (Vecchiato et al., 2016), Spain ( $\text{nd}\text{--}79 \text{ ng L}^{-1}$ ) (Negreria et al., 2010), ( $10\text{--}59 \text{ ng L}^{-1}$ ) (Vila et al., 2016) and Japan ( $\text{nd}\text{--}197 \text{ ng L}^{-1}$ ) (Kameda et al., 2011). One of the most concerning issues is the possibility of adverse effects to Antarctic wildlife due to the presence of emerging pollutants. Traces of PCPs and FMs with endocrine disrupting properties were detected in the Antarctic biota (Emnet et al., 2015; Sanchís et al., 2015; Schiavone et al., 2009). The values of  $\text{LogP}_{\text{ow}}$  of the six FMs detected in this study are in the range of  $3.9\text{--}6.0$  (Table SI1). Although at present there are no data available in the literature about the hypothesis of the bioaccumulation of the selected fragrances, is known that Benzyl Salicylate exhibits allergenic and oestrogenic effects in humans (Charles and Darbre, 2009; Zhang et al., 2012). However the detected values of Salicylates here are far below the acute ecotoxicity levels to *Daphnia* and other species ( $0.7\text{--}10 \text{ mg L}^{-1}$ ) (Belsito et al., 2007).

Different hypotheses could explain the trends and levels of FMs in the seawater reported above: the first one is that FMs in Tethys Bay are directly influenced by the discharges of MZS and the variability of emissions and distribution could eventually explain the differences in concentration. Coastal surface currents are strongly influenced by local winds, resulting in variability in velocity and direction (Stocchino, 1990). However, the trend detected at TBa appears linked to the seasonal melting of sea ice and of its snow cover, and is consistent with a possible release of FMs into the surface seawater: FMs result below the MDL or at few  $\text{ng L}^{-1}$  in the early melting period, increasing up to  $100 \text{ ng L}^{-1}$  during the season. Concentrations drop again in the sample TBa8. Sea ice was also expected to act as a sink and source of PCPs in Erebus bay (Emnet et al., 2015). The melting of snow and ice is known to be a source of Persistent Organic Pollutants (POPs) in the Antarctic marine environment (Dickhut et al., 2012; Geisz et al., 2008; Vecchiato et al., 2015b). This process was highlighted also for per- and polyfluoroalkyl substances (Cai et al., 2012) and volatile methyl siloxanes (VMS) (Sanchís et al., 2015), and is consistent with snow depositional inputs. The same behavior was observed in the area of Terra Nova Bay for polychlorinated biphenyls (PCBs) (Fuoco et al., 1996) and polycyclic aromatic hydrocarbons (PAHs) (Cincinelli et al., 2008), due to the release throughout the melting of pack ice of particle-bound contaminants accumulated



**Fig. 2.** Concentrations of FMs in seawater samples ( $\text{ng L}^{-1}$ ) from the site TBa. Refer to Table SI3 for other samples and details.

from atmospheric deposition during its formation. This latter hypothesis includes the role of the atmospheric transport, which cannot be excluded for these semi-volatile fragrances (vapour pressures are reported in Table S11), which were initially selected because of their stability (Vecchiato et al., 2016). Moreover, the conditions of the Antarctic environment are supposed to reduce the degradation of PCPs (Emnet et al., 2015). The long-range atmospheric transport of pollutants is well documented in this area (Piazza et al., 2013), however also a short-range transport of the FMIs evaporated from the household and PCPs used in the station is possible.

Only 6 of 17 fragrances were detected in the seawater of Terra Nova Bay, and three of these were Salicylates, that were also the most widespread FMIs also in the urban canals of Venice (Vecchiato et al., 2016). Conversely Peonile and Oranger Crystals, abundant in urban surface waters, were not found in the Antarctic samples and this could highlight possible differences in their transport and degradation processes. Another factor to be taken into account is the global production and usage, since Salicylates are High Production Volume (HPV) chemicals (Belsito et al., 2007) with an annual consumption well over 5000 tons (Gaudin, 2014). The environmental concentrations of these substances could reflect this widespread usage.

#### 4. Conclusions

This exploratory study represents the first detection of the selected FMIs in the Antarctic environment: FMIs were found to vary in the surface seawater of Tethys Bay during the seasonal melting of the sea ice, reaching total concentrations up to  $100 \text{ ng L}^{-1}$ . The direct emission from MZS research station was documented ( $156 \text{ ng L}^{-1}$ ), however atmospheric transport processes are also possible, involving areas not directly affected by the local human presence. Beyond understanding the sources, the main open question for future studies is to identify the unknown environmental fate of these substances, focusing also on their presence in the atmospheric compartment to identify possible transport processes. This is mandatory for the protection of the fragile Antarctic marine ecosystem.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.scitotenv.2017.03.197>.

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