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Organochlorine pesticides and PCBs in perch *Perca fluviatilis* from the Odra/Oder river estuary, Baltic Sea

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Abstract

DDTs (*p,p′*-DDT, *p,p′*-DDD, *p,p′*-DDE and *p,p′*-DDMU), HCHs (isomers α, β and γ), hexachlorobenzene and polychlorinated biphenyls (PCBs IUPAC Nos. 28, 52, 101, 151, 118, 153, 138 and 180) were quantified in perch sampled at three sites in the Odra/Oder river estuary in the south-western part of the Baltic Sea, in 1996–1997, to evaluate the status of contamination and possible spatial and temporal trends. All samples of muscle tissue examined contained detectable amounts of both organochlorine pesticides and PCBs, and concentrations were lower (or within the range of) those noted in perch elsewhere in the Baltic Sea in early 1990s. Some seasonal variations of DDT and PCB concentrations but not of HCHs or HCB were noted. Multivariate analysis of data resulted in distinct groups of organochlorines, related to bioaccumulation and biotransformation features of the particular compounds.

Keywords: Fish; Seafood; Food; Residues; Contaminants; Pesticides; Monitoring; POPs

1. Introduction

Organochlorine pesticides, such as DDT, HCH and HCB, as well as many intentionally and unintentionally produced chemicals, such as polychlorinated biphenyls (PCBs), polychlorinated naphthalenes (PCNs), polychlorinated terphenyls (PCTs), polybrominated diphenyl ethers (PBDEs), some polyfluorinated compounds, polychlorinated dibenzo-*p*-dioxins (PCDDs) or polychlorinated dibenzofurans (PCDFs) are nowadays categorised as members of the group of ubiquitous, persistent, lipophilic, bioaccumulative and toxic or highly toxic microcontaminants in the global environment (Erickson, 1997; Falandysz, 1998, 1999, 2004; Kannan et al., 2003a; Kannan et al., 2003b; Yamashita, Taniyasu, Hanari, Horii, & Falandysz, 2003). Chlorobiphenyls, which have been manufactured since 1929, became recognised as new synthetic compounds contaminating the environment in the 1960s (Jensen, 1966).

Seafood usually contains residues of PCBs, as well as other environmentally persistent organohalogenated compounds, and is often considered to be a major source of intake of those contaminants for humans (Falandysz, 2004). Marine biota are frequently subjects of numerous studies on the effects of man-derived organohalogenated contaminants. Those organisms also serve as effective indicators in monitoring studies of the state of pollution of the aquatic environment with persistent organohalogenated compounds. To better understand the status of PCBs, DDTs, HCB and HCHs, contamination of food resources from the southern part of the Baltic Sea, the present study examines residue concentrations and spatial variations of those chemicals actually found in perch, *Perca fluviatilis*, from the estuary of the Odra/Oder river in Poland.

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2. Materials and methods

2.1. Sample collection

Perch (*P. fluviatilis*) were caught on several occasions, using gill nets, at a site near the Świna river outlet (I), as well as during a single attempt at the site near the Dzwina river outlet (II) in the coastal zone of the Pomeranian bay, and also near the Odra/Oder river outlet (III) in the Szczecin lagoon (Fig. 1) in 1996–1997.

2.2. Study area

The Odra/Oder river estuary – multipart system composed of lagoons, lakes and river branches – is the biggest estuary of the entire Baltic Sea (Grelowski, Pastuszak, Sitek, & Witek, 2000). The Oder river provides the fifth-largest river runoff discharge of the southwestern part of the Baltic – freshwater discharge amounts to about 18 km^3/year from a drainage area of 119,000 km^2. The Odra/Oder river waters are transported throughout the Szczecin lagoon and three rivers, Peenestrom (10%), Świna (80%), and Dźwina (10%), into the Pomeranian bay. The Pomeranian bay and Szczecin lagoon are mixing zones between waters of the Baltic proper and the Odra river, which carries waters from a densely populated area, of intensive agriculture and industrialised catchments, in Poland, Germany and the Czech Republic (Falandysz, Trzosińska, Szefer, Warzocha, & Draganik, 2000). The concentration of polycyclic hydrocarbons (PAHs) in the Odra river was estimated as 1870 mg l^{-1}, and the annual load of those compounds, discharged by the Odra, was calculated as about 0.79 ton/year (Emeis et al., 2002). In summer, 1997, the largest Odra river flood of the past century occurred during two periods of remarkably heavy rainfall, in July 1997 (Siegel & Gerth, 2000).

The Szczecin lagoon has an area of about 687 km^2, a volume of about 3.4 km^3, a mean salinity of about 2 psu and a residence time of water masses of about 2 months. In the Szczecin lagoon, salinity distribution is relatively homogeneous and gradients occur mostly within the narrow outlets, which connect the lagoon with the open Baltic Sea. The sediments consist primarily of silty material (6.3–63 μm) and are rather homogeneously distributed. The lagoon is very shallow, with a mean depth of 5 m, and thus the water column is more or less permanently mixed by wind. Even the sediments are directly predisposed by wind-mixing, down to a 10 cm sediment depth (Humborg, Fennel, Pastuszak, & Fennel, 2000).

The Pomeranian bay has area of approximately 7000 km^2, with depth of about 14 m and salinity in the range 6–8 psu (Falandysz et al., 2000). The Pomeranian bay is a very active environment and a nondepositional area with sandy sediments; the material formed in its water column, from erosion of strata at the seafloor and cliffs, and delivered by rivers, is transported, near the seafloor, to the depositional areas of the Arkona, Bornholm and Gdańsk basins (Emeis et al., 2002; Miltner & Emeis, 2000).

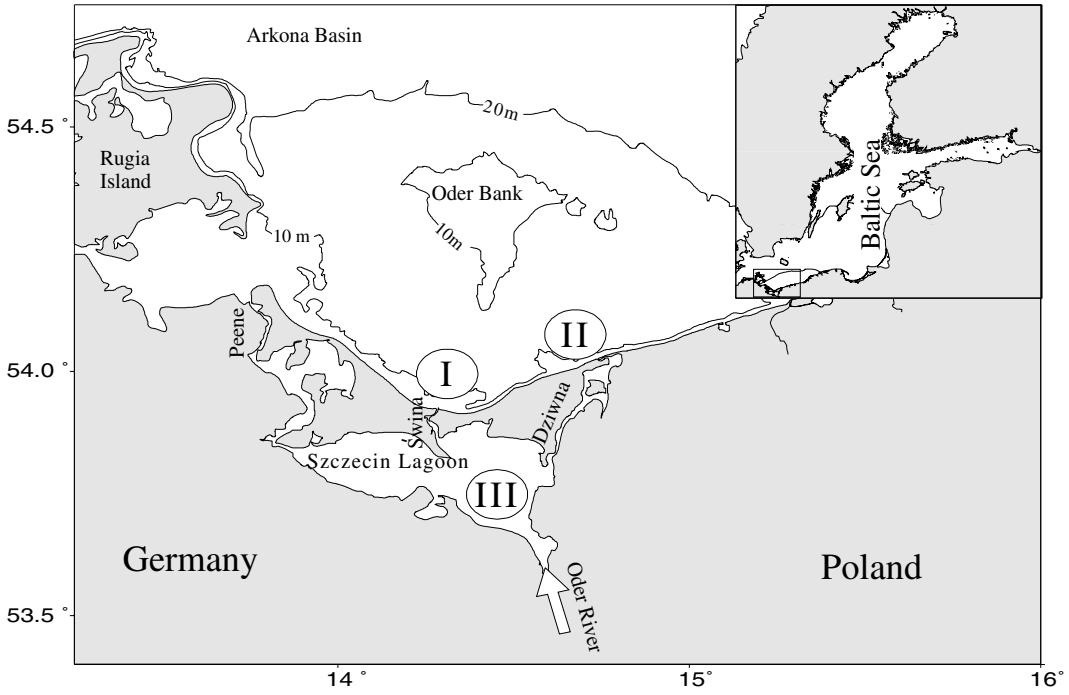

Fig. 1. Localisation of the sampling sites (I–III).

2.3. Analytical methods

Fish, immediately after capture, were packed with ice and brought back to the laboratory. Each muscle tissue sample taken was homogenised, deep-frozen (petri dishes), lyophilised, fine-ground and finally extracted with *n*-hexane in the Soxtec HT6 apparatus. The solvent was carefully evaporated and an aliquot of lipid (ca. 0.5 g of oil) was dissolved in *n*-hexane and then treated with a 10 ml mixture of concentrated sulphuric acid and 30% fuming sulphuric acid (1:1) in Teflon stopped glass tubes and periodically shaken (at 30-min intervals) during a 3-h cycle. After 3 h, the content of the tube was centrifuged, deep frozen (ca. −50 °C) and the upper *n*-hexane layer was collected. Analysis and detection were accomplished using a high resolution capillary column (Rtx-5; 60 m length) gas chromatograph (GC) with electron capture detector (ECD). A mixed standard of DDT and its analogues (*p*,*p*′-DDT, *p*,*p*′-DDD, *p*,*p*′-DDE and *p*,*p*′-DDMU), isomers of α, β and γ hexachlorocyclohexane as well as of selected chlorobiphenyl congeners (PCB Nos. 28, 52, 101, 151, 118, 153, 138 and 180) were used in quantification.

2.4. Analytical quality assurance/analytical quality control

The analytical method used was properly validated and the analytical laboratory involved in 1999 successfully passed the accreditation procedure (Accreditation certificate no. AB 017). Certified standard reference materials, such as NBS 5881, Lyophilised Fish Tissue (MA-A3/OC and MA-B-3/OC), IAEA 142/OC and Cod Liver Oil SRM 1588 were examined. In parallel, from 1995, we participated in annual intercalibration trials, led by the Państwowy Zakład Higieny (PZH) in Warsaw and, periodically, in trials organised by the Quasimeme Laboratory and the results obtained were satisfactory (Table 1).

Table 1
The results of analysis of certified reference material "Cod Liver Oil SRM 1588"

Compound	Concentration (ng/g)	
	Certified value	Found
α-HCH	73.8 ± 15.0	85.3 ± 3.4
γ-HCH	16.8 ± 4.5	24.9 ± 1.7
HCB	127 ± 20	158 ± 5.0
p,*p*′-DDE	641 ± 67	651 ± 11
p,*p*′-DDD	224 ± 6	254 ± 11
p,*p*′-DDT	624 ± 25	524 ± 12
PCB No. 28	30.6 ± 8.0	28.3 ± 0.5
PCB No. 52	93.2 ± 3.7	83.3 ± 2.3
PCB No. 101	134 ± 17	127 ± 4.3
PCB No. 151	45.3 ± 2.5	54.8 ± 2.1
PCB No. 118	172 ± 35.0	176 ± 3.8
PCB No. 153	226 ± 19.0	274 ± 7.7
PCB No. 138	266 ± 22.0	264 ± 9.1
PCB No. 180	92.2 ± 7.5	105 ± 5.2

3. Results and discussion

3.1. General

The results of the analyses, including arithmetic mean, standard deviation and range of the concentrations normalised to lipid weight basis, are presented in Table 2.

Both the residues of PCBs, as well as of DDT and its metabolites, HCH isomers and HCB, could be found in perch sampled in the Odra/Oder river estuary in 1996–1997 but the concentrations were generally much lower than those of various species of fish sampled in the southern part of the Baltic Sea in 1980–1983 (Falandysz, 1982, 1984a, 1984b, 1985a, 1985b, 1986a, 1986b). The body weight and body length of perch, from each site and period examined, varied somewhat, while variation of PCB, DDT, HCH and HCB concentrations, as well as of lipid contents, were much less marked (Table 2). Most of the samples examined originated from site I, situated near the outlet of Świna river, which carries waters from the city of Szczecin and the town of Świnoujście agglomerations, as well as from the Szczecin shipyard, considered as main sources of pollution to the Szczecin lagoon (Gonet & Cieślewicz, 1998). Preliminary data indicated some seasonal variations of PCB and DDT contents of perch muscle tissue, while the values noted for HCHs and HCB were more or less similar (Table 2).

It has to be taken into consideration that the fish samples were collected before and after the flood of summer, 1997. Heavy rains in the southern part of Poland resulted in a rapid rise in the river water flow. This was especially dangerous in the catchment area of the Odra, which overflowed its banks in many places, covering large land areas, over 500 000 ha, and numerous towns and villages with water. Between July 21 and August 20 1997, a sixfold greater volume of water was carried by the Odra river than in the relevant period of 1996; this was 70% greater than the long-term maximum discharges for the Odra. This phenomenon was accompanied by a drastic decrease of oxygen content in the river water, carrying simultaneously large quantities of organic matter and potentially, also, various pollutants (Falandysz et al., 2000).

3.2. PCBs

PCB congeners (Nos. 28, 52, 101, 118, 138, 151, 153 and 180) were selected form the group of 'standard indicative' congeners recommended for the purpose of quantification, as potential target analytes, which are

Table 2
Polychlorinated biphenyl, DDT, HCH and HCB contents (ng/g lipid) in perch from Odra river estuary

Site	Date of samples collection	Sample No.	Body weight (g)	Lipid (%)	PCBs	DDTs	HCHs	HCB
I	28.5.1996	29	66–545	0.51 ± 0.46	610 ± 410	680 ± 460	44 ± 31	12 ± 6.4
				(0.24–2.85)	(21–2100)	(130–2400)	(1–140)	(0.10–36)
I	23.1.1997	21	157–740	1.28 ± 1.51	370 ± 370	410 ± 380	37 ± 61	7.8 ± 10
				(0.39–4.7)	(36–1800)	(34–1700)	(3–280)	(0.05–52)
I	27.2.1997	22	58–835	0.57 ± 0.13	470 ± 270	540 ± 240	18 ± 14	15 ± 13
				(0.30–0.76)	(340–1100)	(150–1000)	(3–59)	(0.90–66)
I	25.4.1997	19	25–450	0.45 ± 0.09	1500 ± 1300	1000 ± 880	42 ± 43	8.6 ± 6.6
				(0.32–0.62)	(430–5000)	(330–4000)	(16–170)	(0.7–31)
I	25.8.1997	20	51–514	0.49 ± 0.09	630 ± 240	370 ± 97	39 ± 15	15 ± 10
				(0.42–0.80)	(380–1400)	(270–610)	(15–78)	(3.1–58)
I	22.10.1997	19	35–245	0.50 ± 0.09	1100 ± 790	690 ± 520	40 ± 38	17 ± 14
				(0.36–0.78)	(620–4400)	(330–2800)	(14–190)	(6.7–92)
II	22.8.1997	10	104–244	0.44 ± 0.02	650 ± 100	370 ± 23	44 ± 45	8.0 ± 3.7
				(0.42–0.49)	(550–910)	(34–410)	(9–170)	(1.1–17)
III	22.8.1997	7	53–167	0.44 ± 0.05	470 ± 98	540 ± 260	47 ± 11	10 ± 4.6
				(0.38–0.50)	(310–570)	(280–890)	(32–65)	(5–22)

frequently the most abundant in various environmental matrices and cover a wide degree of chlorination (Falandysz, Strandberg, Strandberg, & Rappe, 1999; USEPA, 1995). The data on the total PCB contents of perch fillets are summarised in Table 2.

There are only few data available on residue concentrations of POPs in perch from the Polish coast of the Baltic Sea (Falandysz, Wyrzykowska, Puzyn, Strandberg, & Rappe, 2002; Falandysz, Strandberg, Puzyn, Gucia, & Rappe, 2001; Falandysz et al., 1996). PCB (68 congeners of tri- through decachlorobiphenyl homologue groups) contents of lipids, extracted from a homogenate of the whole body perch caught at the several sites from the Swedish coast of the Baltic Sea (Bothnian bay and Bothnian sea) in 1991, varied between 1000 and 5400 ng/g, while 2900 ng/g were noted for perch caught from the Polish coast (Gulf of Gdańsk) of the Baltic Sea in 1992 (Falandysz et al., 2002; Strandberg et al., 1998). Perch caught from the Swedish waters in 1992–1993 contained total PCBs, in concentrations from 1200 to 2200 ng/g lipid, while in perch caught in coastal waters of Latvia were from 730 to 1400 (410–3600) and, in specimens from inland waters, were from 180 to 2400 ng/g lipid weight (Olsson, Vitinsh, Plikshs, & Bergman, 1999; Strandberg et al., 1998; Valters, Olsson, Asplund, & Bergman, 1999). The mean residue concentration of PCBs quantified in muscle tissue of perch noted in this study are between 370 and 1100 ng/g lipid, which is substantially lower than that observed by the authors cited above; however, only selected, but usually dominating congeners, were quantified in the present study.

3.3. DDT and its metabolites (DDTs)

Because of their wide distribution in air, water, soil and food, *p,p′*-DDT and its metabolites remain a human health concern and have been quantified in the present study. In the case of DDTs, the mean concentrations varied between 370 ± 23 and 1000 ± 880 ng/g lipid, which is much lower than the value of 1400 ng/g noted for perch caught in the Gulf of Gdańsk in 1992 (Falandysz et al., 1999). In perch from the Bothnian bay and Bothnian sea in 1992–1993, they were 225 and 210–880 ng/g lipid, respectively (Strandberg et al., 1998). Contamination, with DDT and its metabolites, of perch from the freshwaters in Norway was in the range 400–2300 ng/g lipid weight for DDT, between 370 and 2960 ng/g for DDD and 760–5100 ng/g for DDE, with "sum-DDT" in the range 1200–1600 ng/g lipid (Brevik, Grande, Knutzen, Polder, & Skaare, 1996). The concentrations of DDE in pooled perch samples from the rural locations (lakes and rivers) in Latvia ranged from 60 to 460 ng/g lipids, for DDD from 3.1 to 210 ng/g, and when calculated as DDTs, from 140 to 280 ng/g (Valters et al., 1999).

3.4. HCH isomers

γ-HCH was the dominant HCH isomer quantified in perch, followed by the isomers β and α, which seems to confirm data from the reports showing a substantial decreasing trend of the European usage of technical α-HCH in recent decades, compared to a much slower decrease of γ-HCH usage (Breivik, Pacyna, & Münch, 1999). No seasonal or site-specific differences could be observed in HCH contents of perch muscle tissue (Table 1), and an overall concentration was between 18 ± 14 and 44 ± 31 ng/g lipids. As with DDTs, the concentrations of HCHs noted in perch, in 1996–1997, were much lower than in various species of fish netted in the southern part of the Baltic Sea in 1980–1983 or in Baltic perch caught in 1991–1992 (Falandysz, 1982, 1984a, 1984b, 1985a, 1985b, 1986a, 1986b). Similar trends in concentration patterns of HCHs, as in the Polish coastal zone of the Baltic Sea, were observed in the Latvian waters, with no obvious differences regarding the sampling sites studied. This has been suggested as a indication that the main source of these compounds is an aerial deposition, due to long-range atmospheric transport (Valters et al., 1999).

3.5. HCB

The results of this study have not shown seasonal or temporal trends in the concentrations of hexachlorobenzene in perch. Concentrations were similar to or smaller than those observed in previous studies; e.g., the concentrations of HCB in Baltic samples of perch caught in Swedish waters in 1992–1993 were, on average, 0.73 ng/g fresh weight (Atuma, Linder, Andersson, & Larsson, 1996). In Latvian waters, HCB concentrations in perch were in a similar range (6–27 ng/g) and, also, no substantial differences, according to the place of sample collection were observed (Valters et al., 1999).

3.6. Principal component analysis

A correlation matrix was computed by the principal component method. The correlation matrix shows that a certain degree of association exists between some organochlorines. The number of components was chosen with the Kaiser criterion – only factors with eigenvalues greater than 1 were retained. A factor matrix was obtained after Varimax rotation. The PCA model of data matrix explained 75% of the total variation by the first four principal components.

Configuration of cluster intercorrelations (Figs. 2 and 3) could be explained by differences in bioaccumulation and biotransformation features of certain groups of organochlorines in perch and furthermore, by differences due to region, season, sex and the weight and length of the fish and also lipid contents. Taking into consideration the separation due to age of the fish, we should bear in mind the hypothesis that age of the fish influences, not only the body burden of pollutants by the longer exposure factor, but also by the factor of migratory habits. Different age classes could be more or less migratory, and thus more or less suitable for trend biomonitoring studies (Atuma et al., 1996).

Interestingly, HCHs and HCBs (which show no significant temporal trends in their concentrations) cluster together. It is possible that long-range atmospheric

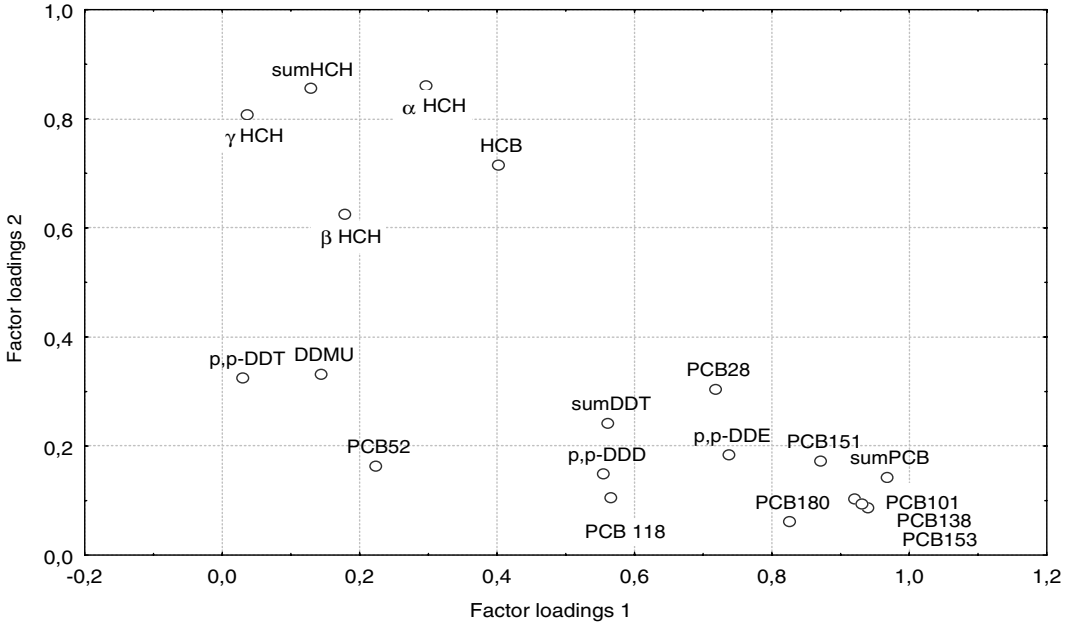

Fig. 2. PCA variable loading plot of organochlorines in perch from the Odra/Oder river estuary.

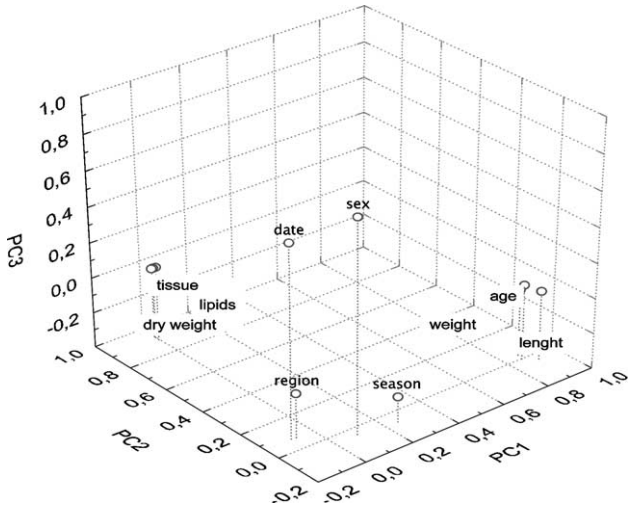

Fig. 3. A PCA object score plot of PCB concentrations in perch from the Odra/Oder river estuary.

transport and subsequent deposition on the sea surface is a main cause of those pollutants in the investigated area. The concentrations of DDTs and PCBs, both with rather low scores in PC2, showed temporal trends, probably due to the flood in 1997. Separations between PCBs could be explained by their chlorination patterns and, furthermore their persistency resulted from substitution pattern. For example, most persistent congeners are thought not to be metabolised by fish (e.g., Nos. 153, 138 and 180) (Falandysz et al., 2002), if they have only *ortho–meta* vicinal H positions and no *meta–para* H vicinal positions and less persistent congeners (e.g., Nos. 101 and 151), possessing *meta–para* vicinal H atoms cluster together. Apparently, less chlorinated chlorobiphenyls (Nos. 28 and 52) are separated from more chlorinated ones.

The concentrations of all chlorinated compounds studied in perch from the Odra estuary seem to have decreased compared to earlier studies in the southern part of the Baltic Sea.

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