Chlorinated Biphenyls and $p,p'$-DDE in Mediterranean Surface Waters*

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Individual chlorobiphenyls (CBs) and $p,p'$-DDE were determined in solution and suspension in surface transects of the open Mediterranean Sea in 1987. Large volumes of sea water (1400–1700 dm$^3$) were analyzed. Concentrations of CBs were low in solution (<5 pg/dm$^3$) and in suspension (<60 pg/dm$^3$). Values for their sum (ECB) were 1.7–43.9 and 190–615 pg/dm$^3$, respectively. Compositions of the CB mixtures in solution as well as those in suspension were very similar in the various transects, but considerable differences were present between solution and suspension in each transect. It was attempted to interpret these differences in terms of molecular characteristics (octanol-water distribution coefficients, $K_{ow}$). The resulting plots of log $K_{ow}$ vs. the apparent distribution coefficient (log $K_d$) gave evidence for the uptake of CBs by suspended particles, followed by sedimentation out of the surface layer. Concentrations in solution were much lower (1–2 orders of magnitude) than those reported before. This may result from improvements in the sampling and analytical techniques. No clear evidence for significant local sources of these compounds was detected in the surface waters, as the levels in solution were very similar to the very low concentrations in the adjacent open Atlantic.

* Dedicated to Marko Branica on the occasion of his 65th birthday.
INTRODUCTION

Lipophilic man-made organochlorines have been considered a serious threat to the long-term health of the marine environment for many years. The main reasons are their strong accumulation in lipid tissues of marine biota as well as the high toxicity for marine organisms and the slow degradation of several members of this group. Much effort has been spent in studies on the distribution of these compounds on a worldwide scale, especially in biota. The Mediterranean Sea does not represent an exception in this aspect. There is also an interest in this class of chemicals from a more fundamental scientific point of view. This interest has been steadily growing in recent years, because methods have become available for the accurate determination of individual compounds in various environmental compartments, including sea water. These levels can now be correlated with known molecular properties. In particular chlorobiphenyls, representing a wide range of water solubilities, vapour pressures, etc., can be used as model compounds. Their distributions between water and particles, or between various tissues of an organism can be used to develop distribution models which can in turn be used to forecast the behaviour of other, less well studied compounds.

Reliable data on dissolved and particulate suspended organochlorines in sea water is scarce. This is mainly due to the inadequate sampling and analytical techniques that have been most commonly used in the past. Moreover in many older studies the technical polychlorobiphenyl mixtures (PCBs) were used for quantification. This results in data that have relatively low scientific significance.

We have spent considerable time to develop a reliable method to determine CBs in sea water. One of the crucial steps to reach this goal was the sampling and analysis of large volumes of water, at least 500–1000 dm$^3$. For surface water, this can be realized with a sub-surface pumping system mounted at the bow of the ship. The Mediterranean Sea was selected as our initial test area for large volume sampling, mainly because of the lively discussions on this topic during several of the famous »International Conferences on the Chemistry of the Mediterranean«, organized and chaired by Marko Branica. Since this first cruise in 1987, we have checked the method with other methods. For instance, in situ pumps can be applied in surface as well as in deep water. Their use has given us confidence that the present data for surface waters of the Mediterranean are reliable. The present report on the levels of chlorobiphenyls in water and suspended particles of the Mediterranean Sea allows a comparison of this area with the North Sea, Baltic Sea and open Atlantic.

Samples were taken during a cruise of the Physical Oceanography of the Eastern Mediterranean (POEM) programme, aimed at improving the data
base for a comprehensive description of the general circulation of the Eastern Mediterranean. In addition to measurements of temperature, salinity and chlorofluoromethane which were made by others, our group was responsible for the measurements of dissolved oxygen and nutrients (phosphate, silicate) in surface water and in vertical profiles of organochlorines in surface transects.

EXPERIMENTAL

1400–1700 dm³ sea water were obtained with the pumping system installed on board R. V. Meteor. Water was taken continuously while the ship was moving. It is taken from a by-pass for filtration and extraction by XAD–2 resin in the ship's laboratory. The filtrate and the filtered particulate material were analyzed separately. Results for hydrocarbons in the water extracts have been published by Ehrhardt and Petrick.

The levels of organochlorines in sea water are extremely low (pg/dm³ range). Contamination must be kept at a correspondingly very low level in the entire procedure, in which sampling is the most critical step. The sampling of large volumes of water helps to reduce the effect of contamination. Also, sufficient material must be collected to satisfy the sensitivity requirements of the instrumental analytical method, and this cannot be achieved with samples of only a few liters of water.

The methods used to detect and eliminate contamination, to extract water and particulates, and to perform the GC–ECD analyses have been described in detail.

Samples were taken in seven transects in the eastern part of the Mediterranean Sea in August/September 1987 (Figure 1), resulting in integrated signals over a certain range of temperature, salinity and chemical characteristics of the water bodies. Additionally, one transect, extending from the end of transect 7, covered the western Mediterranean Sea up to Strait of Gibraltar (transect 8).

As we are interested particularly in the distribution of individual CBs between solution and suspension, only those CBs are reported that were present above their detection limits (0.01 pg/dm³ for each congener) in both compartments. A further selection has been made by only including CBs that could be analyzed as single GC peaks. Some relatively strong peaks in the chromatograms (e.g. CBs–66/95) have therefore been omitted from the calculations. Thus, 27 CBs are reported. Their sum (ΣCB) represents about 85% of total CBs. Also p,p'-DDE has been analyzed (in the following text described as DDE). Filters were not weighed to avoid contamination. The amounts of each congener associated with particles are therefore presented only in terms of concentrations (pg/dm³) and not in terms of contents (pg/g particles).

RESULTS AND DISCUSSION

The distributions of temperature, salinity and dissolved oxygen, phosphate and silicate in vertical profiles along the transects were characterized by only small gradients. Phosphate and silicate concentrations were small.
Values in deep waters were: phosphate $\approx 250$ nM, silicate $\approx 11$ $\mu$M) and in surface waters: phosphate $<50$ nM and silicate 0–1 $\mu$M. The surface transects were affected by contributions from deeper water in some regions, but the inhomogeneities in the surface distributions of temperature, salinity, phosphate and silicate were only minor within each of the transects. An example is given in Figure 2 for transect 2. It can be assumed therefore that chlorobiphenyls in the transects are not affected significantly by mixing with deep saline or fresh water. There are only very small differences in the distributions of temperature, salinity and for the nutrient concentrations between the various transects.

The surface concentrations of individual CBs and DDE are presented in Table I. In order to facilitate a comparison with data that we reported for the North Sea,$^{16}$ the Baltic Sea$^5$ and the open Atlantic,$^{17}$ the sum of CBs is also given ($\Sigma$ CB).

**Concentrations of DDE**

DDE concentrations in solution (0.4–9.0 pg/dm$^3$) were lower than in suspension (7–70 pg/dm$^3$) in each transect. Transects 4 had increased DDE levels in solution (Table I).
Concentrations of Chlorobiphenyls

As the compositions of the CB mixtures in solution in transects 1–8 were very similar among them (the same applies to the suspended matter), ΣCB values can be used to compare regions and to identify gross regional concentration differences.

Figure 2. Vertical distribution of temperature, salinity, dissolved phosphate and dissolved silicate in the section covered by transect 2 (Figure 1). Bottom topography is represented schematically.
<table>
<thead>
<tr>
<th>CB number</th>
<th>Solution - Sampling transect number</th>
<th>Suspension - Sampling transect number</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 2 3 4 5 6 7 8</td>
<td>1 2 3 4 5 6 7 8</td>
</tr>
<tr>
<td>18</td>
<td>0.9 0.6 0.6 0.6 0.6 0.3 0.6</td>
<td>18.0 7.0 12.0 3.0 10.0 26.0 12.0 5.0</td>
</tr>
<tr>
<td>28</td>
<td>1.0 0.2 0.6 4.0 0.8 0.3 0.2 0.7</td>
<td>10.0 9.0 9.0 3.0 5.0 16.0 9.0 4.0</td>
</tr>
<tr>
<td>31</td>
<td>0.9 0.1 0.6 4.0 0.8 0.3 0.2 0.6</td>
<td>11.0 9.0 11.0 4.0 6.0 19.0 10.0 5.0</td>
</tr>
<tr>
<td>40</td>
<td>0.2 0.0 0.1 0.8 0.1 0.0 0.1 0.1</td>
<td>12.0 11.0 14.0 15.0 7.0 17.0 14.0 15.0</td>
</tr>
<tr>
<td>44</td>
<td>0.5 0.1 0.5 2.5 0.2 0.2 0.4</td>
<td>16.0 22.0 37.0 17.0 12.0 40.0 21.0 15.0</td>
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<tr>
<td>49</td>
<td>0.6 0.0 0.5 4.1 0.5 0.2 0.2 0.4</td>
<td>11.0 8.0 19.0 8.0 5.0 26.0 15.0 8.0</td>
</tr>
<tr>
<td>52</td>
<td>0.7 0.1 0.6 3.1 0.7 0.3 0.4 0.6</td>
<td>17.0 25.0 60.0 26.0 13.0 59.0 25.0 19.0</td>
</tr>
<tr>
<td>70</td>
<td>0.3 0.0 0.3 1.9 0.3 0.2 0.0 0.1</td>
<td>8.0 9.0 20.0 11.0 6.0 45.0 11.0 8.0</td>
</tr>
<tr>
<td>74</td>
<td>0.6 0.1 0.2 1.0 0.4 0.2 0.1 0.2</td>
<td>5.0 4.0 7.0 4.0 3.0 10.0 5.0 2.0</td>
</tr>
<tr>
<td>84</td>
<td>0.2 0.0 0.2 0.9 0.1 0.1 0.1 0.1</td>
<td>5.0 9.0 15.0 10.0 5.0 25.0 8.0 5.0</td>
</tr>
<tr>
<td>92</td>
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<td>7.0 7.0 8.0 5.0 4.0 25.0 6.0 2.0</td>
</tr>
<tr>
<td>99</td>
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<td>3.0 5.0 11.0 6.0 3.0 12.0 5.0 3.0</td>
</tr>
<tr>
<td>101</td>
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<td>13.0 16.0 33.0 18.0 9.0 41.0 15.0 11.0</td>
</tr>
<tr>
<td>110</td>
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<td>12.0 23.0 29.0 19.0 15.0 53.0 27.0 11.0</td>
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<tr>
<td>118</td>
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</tr>
<tr>
<td>128</td>
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<td>9.0 7.0 6.0 5.0 6.0 12.0 11.0 5.0</td>
</tr>
<tr>
<td>138</td>
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<td>50.0 36.0 31.0 20.0 27.0 46.0 33.0 18.0</td>
</tr>
<tr>
<td>146</td>
<td>0.1 0.1 0.1 2.0 0.1 0.0 0.0 0.1</td>
<td>4.0 3.0 4.0 2.0 3.0 4.0 10.0 1.0</td>
</tr>
<tr>
<td>149</td>
<td>0.2 0.1 0.1 1.0 0.2 0.1 0.1 0.1</td>
<td>15.0 12.0 15.0 8.0 10.0 18.0 12.0 6.0</td>
</tr>
<tr>
<td>153</td>
<td>0.2 0.1 0.1 1.2 0.3 0.1 0.1 0.1</td>
<td>42.0 26.0 26.0 14.0 21.0 32.0 26.0 15.0</td>
</tr>
<tr>
<td>170</td>
<td>0.1 0.1 0.1 0.0 0.2 0.0 0.0 0.0</td>
<td>16.0 12.0 7.0 6.0 10.0 11.0 13.0 4.0</td>
</tr>
<tr>
<td>174</td>
<td>0.1 0.0 0.1 0.3 0.1 0.0 0.0 0.0</td>
<td>16.0 11.0 7.0 5.0 8.0 12.0 9.0 4.0</td>
</tr>
<tr>
<td>177</td>
<td>0.0 0.0 0.0 0.2 0.1 0.0 0.0 0.0</td>
<td>8.0 6.0 3.0 3.0 5.0 6.0 5.0 3.0</td>
</tr>
<tr>
<td>180</td>
<td>0.1 0.1 0.1 0.0 0.3 0.1 0.0 0.1</td>
<td>31.0 23.0 14.0 11.0 18.0 23.0 24.0 8.0</td>
</tr>
<tr>
<td>183</td>
<td>0.0 0.0 0.0 0.3 0.1 0.0 0.0 0.1</td>
<td>6.0 7.0 3.0 3.0 5.0 7.0 5.0 4.0</td>
</tr>
<tr>
<td>187</td>
<td>0.1 0.1 0.0 0.4 0.1 0.1 0.0 0.0</td>
<td>7.0 9.0 6.0 4.0 7.0 12.0 9.0 4.0</td>
</tr>
<tr>
<td>199</td>
<td>0.0 0.0 0.0 &lt; &lt; &lt; &lt; &lt; &lt; &lt; &lt;</td>
<td>4.0 3.0 1.0 1.0 2.0 3.0 1.0</td>
</tr>
</tbody>
</table>

| Sum pg/dm³ | 8.0 1.7 6.1 43.9 8.8 2.9 2.9 5.6 364.0 327.0 419.0 238.0 231.0 615.0 351.0 190.0 |
| Sum fmol/dm³| 13.9 3.2 11.0 79.1 16.1 5.4 5.6 10.0 730.0 657.0 853.0 478.0 463.0 1213.0 693.0 371.0 |
| DDE pg/dm³  | 2.0 0.6 1.3 9.0 2.4 0.5 0.4 1.0 7.0 20.1 15.1 9.0 13.9 69.9 37.2 7.1 |

<: below detection limit. 0.0: between 0.01 and 0.05.
Concentrations of ΣCB in solution ranged between 1.7 and 8.8 pg/dm$^3$ (except in transect 4 with 43.9 pg/dm$^3$, this may indicate a local source). These values reflect the very low values in adjacent Atlantic ocean water (measured during the same cruise) and in transect 8. There is thus no evidence for strong local inputs into these surface waters.

Values of individual CBs were below 1 pg/dm$^3$, except in transect 4 (0.0–4.6 pg/dm$^3$).

The presently reported ΣCB concentrations in solution (1.7–8.8 pg/dm$^3$) are considerably (at least two orders of magnitude) lower than those reported by Burns and Villeneuve$^{18,19}$ and Elder and Villeneuve.$^{20}$ This probably reflects the reduction of sample contamination by the analysis of large water volumes, the extreme care in sampling$^{10}$ and the decrease of PCB production and input into the environment. We have not been able to observe significant changes in CB concentrations in the North Sea and Baltic Sea during recent years. The values reported here can therefore also be considered as a reliable estimate of present concentrations in the Mediterranean Sea.

The concentrations of ΣCB in suspension ranged between 190 and 615 pg/dm$^3$, those of individual CBs in suspension were between 1 and 60 pg/dm$^3$.

The concentrations of ΣCB in suspension were much larger than in solution. The particulate forms represent nearly 100% of the total concentrations in solution and suspension for each transect. Burns and Villeneuve$^{18}$ reported values between 37 and 61%. Our very low concentrations in solution account for these differences in the contribution of the particulate forms to the total concentrations in a water body.

The presently reported concentrations in solution and suspension are of the same order of magnitude as those found in the North Sea, the Baltic Sea and some regions in the North Atlantic, but they are still considerably higher than those in a remote area like the Arctic Ocean (unpublished results).

Compositions of the CB Mixtures

The comparison of compositions of CB mixtures can be carried out conveniently by a transformation of concentrations into relative concentrations. We have chosen here the representation of mol percent contributions of individual CBs to their sum (Figure 3).

The compositions in solution were very similar among the transects. Tri- and tetra-CBs (CB–18, −28, −31, −44, −49, −52, −70, −74) generally occurred with the highest contributions in each transect (4–14 mol%). The average contributions of penta-, hexa- and hepta- CBs were about 2 mol%.

The compositions in suspension were also very similar in the various transects. Contributions were generally in the range 1–5 mol%, but some
Figure 3. Contributions of individual chlorobiphenyls to their sum in mol% in transects 1-8 in solution (top) and in suspension (bottom). CBs (IUPAC numbers) are identified according to Schulz et al. (1989).
values were up to 15 mol%. The CBs with the highest contributions were CBs 
-101, -110, -138, -153 (n_{Cl} = 5-7), -52 and -44 (n_{Cl} = 4) and -180 (n_{Cl} = 7).

The compositions of the CB mixtures differed between solution and sus-
pension. In the following, an attempt is made to relate the differences to mo-
olecular characteristics of the individual constituents.

The ratio of the concentration of CB-X in suspension and in solution can 
be defined as an apparent distribution coefficient (K_d). Its value can be 
calculated from the concentrations (pg/dm^3) without knowledge of the sus-
pended matter concentrations (SPM). A linear relation is expected between 
log K_{ow} and log K_d if equilibrium exists between the two compartments 
water and suspended particulates (represented by a linear sorption iso-
therm).

The plots (Figure 4) do show a positive relation for most transects, but 
the relations are far from being linear (the ideal case). The slopes of the cal-
culated regression lines were between 0.03 (transect 2) and 0.71 (transect 
1). As we illustrate below the deviations from the ideal case may be inter-
preted as a result of biological activities in the water column.

The general increase of K_d with increasing K_{ow} suggests that partition-
ing of CBs between solution and SPM is determined primarily by water 
solubility (as reflected by K_{ow}) and particle availability. Within the series of 
CBs, decreasing water solubility favours a higher K_d. The concentration ra-
tios »CBs in suspension/CBs in solution« (both expressed as mass per unit 
volume) are roughly constant for all CBs within a transect, independent 
from actual concentrations.

In our recent study on CBs in the Baltic Sea, we found a strong sea-
sonal variation of both absolute and relative concentrations in solution and 
suspension. During winter time with low biological activity, concentrations 
in solution were relatively high. Dissolved concentrations have extremely 
decreased in the summer situation, after the plankton-bloom. The opposite 
was found for CBs on particulate matter, with highest values during spring-
bloom in April. This was attributed to the uptake of CBs from solution into 
phytoplankton during bloom, followed by accumulation in zooplankton and 
excretion in feces. The latter may sink out of the euphotic zone rather rap-
idly. CBs are removed by this mechanism from the surface layer into deeper 
water where they are partly remobilized but for the larger part deposited 
into the sediments of the Baltic Sea.

The same mechanism may apply to the Mediterranean. Table II 
summarizes the concentration range of CBs in the Mediterranean in July/
August as found in the present work, in the Baltic Sea in two different sea-
sons (the winter situations in 1988 and 1989 and after-bloom period in 
spring 1989) and in the North Sea (winter and summer). The situation in 
the Mediterranean Sea in late summer, with low concentrations of ΣCB in
solution (2–8 pg/dm$^3$) and high values in suspension (190–615 pg/dm$^3$) are comparable with the after-bloom (spring) situation in the Baltic.

It is interesting to compare the present results with some CB contents determined by Burns and Villeneuve.$^{18}$ These authors reported 110–490 ng PCB/g for copepods (obtained with nets) and 950–1350 ng/g for feces produced by these organisms. We have not measured SPM concentrations, so we can only approximate the contents of CBs in our particulate matter samples by using average SPM concentrations.$^{25}$ Using the mean value of the latter authors (1 mg/dm$^3$), present ΣCB values would be in the range 190–615 ng ΣCB/g SPM. This turns out to be of the same magnitude as the values mentioned above for copepods and feces.

Burns et al.$^3$ reported fluxes of PCB (as Aroclor 1254 equivalents) from the surface layer as determined with sediment traps. They found a signifi-

![Figure 4. Log $K_d$ – log $K_{ow}$ plots for 27 CBs (Table I) in the various transects. $K_{ow}$ values were taken from Hawker and Connell$^{26}$ and apparent distribution coefficients were calculated from molar concentrations in suspension and solution according to Duinker.$^7$]
TABLE II

<table>
<thead>
<tr>
<th></th>
<th>Medit. Sea</th>
<th>Baltic Sea</th>
<th>North Sea</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>summer</td>
<td>winter</td>
<td>spring</td>
</tr>
<tr>
<td>solution</td>
<td>2–44</td>
<td>12–71</td>
<td>2–14</td>
</tr>
<tr>
<td>suspension</td>
<td>190–615</td>
<td>2–26</td>
<td>62–2860</td>
</tr>
<tr>
<td>nm= not measured</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

cant seasonal variability in the period 1979–1982. The highest contents of PCB occurred in June/July 1979, May/August 1980 and in May 1981, with values 10–600 ng PCB/g trap material (values for DDE were appreciably lower: 100 pg/g). Fowler et al.\textsuperscript{22} also established large temporal as well as depth variations in the contents of PCB and individual congeners in large particles collected with traps in the western Mediterranean in 1983–1986.

The range of PCB was 20–1500 ng/g (in the surface layer 100–500); the range of the dominant congeners CB-52 and -101 in the surface layer was 10–100 ng/g). In the present data, CB-52 is also one of the strongest peaks in SPM, CB-101 is much lower. This may be due to the inherent differences between suspended matter and trap material.

The qualitative agreement between these data and the presently reported data should not be overestimated, as the methods to calculate CB contents are principally different (most older data was not based on individual congeners and also regional and temporal variations are considerable).

The log $K_{ow}$ – log $K_d$ plots for the Baltic data showed clear positive slopes for transects where the spring bloom had not started yet and no clear or even negative relations in those regions where the spring bloom had started, or had been going on for some time already. The present situation in the Mediterranean is different, as we have found positive slopes in nearly all transects where production is expected to have been going on for some time.

The partitioning between solution and suspension may have been closer to equilibrium in the oligotrophic Mediterranean than in the eutrophic Baltic Sea. For instance, most of the biological material may have left the surface water before August. This was found in sediment trap studies.\textsuperscript{18} Other factors may also play a role. For instance, the effect of colloidal material has not been solved yet, dissolved concentrations or $K_{ow}$ values may not be accurate enough, octanol-water distribution coefficients may be inadequate to
represent the partitioning of lipophilic compounds between water and natural particles, or the composition of the particles plays a dominant role (e.g., $C_{org}$ content). We are presently carrying out studies to resolve some of the uncertainties.

REFERENCES

SAŽETAK

Klorirani bifenili i $p,p'$-DDE u površinskim vodama Sredozemnog mora

Detlef E. Schulz-Bull, Gert Petrick, Hergen Johannsen i Jan C. Duinker

Pojedinačni klorobifenili (CB) i $p,p'$-DDE izmjereni su u otopini i suspensiji duž transektata u otvorenim vodama Sredozemnog mora 1987. godine. Analizirani su veliki volumeni morske vode (1400–1700 dm³).

Masene koncentracije pojedinih CB bile su niske u otopini (<5 pg/dm³) i u suspensiji (<60 pg/dm³). Ukupne vrijednosti ($\Sigma$CB) iznosile su 1,7–43,9 odnosno 190–615 pg/dm³. Sastav smjesa CB u otopini i suspensiji bio je vrlo sličan na različitim transektima, ali su bile prisutne značajne razlike između otopina i suspensija istog transektia. Te su razlike interpretirane razmatranjem molekulskih karakteristika (koeficijenta raspodjele oktanol-voda, $K_{ow}$). Logaritamski prikazi ovisnosti $K_{ow}$ i prividnog koeficijenta raspodjele ($K_{d}^*$) upućuju na vezanje CB na suspendirane čestice koje prethodi sedimentaciji iz površinskog sloja.

Koncentracije u otopini znatno su niže (1–2 reda veličine) od ranije objavljenih, što se može pripisati postignutim poboljšanjima tehnika uzorkovanja i analize.

Nema jasnog dokaza znatnijih lokalnih izvora tih spojeva u površinskim vodama, budući da su razine u otopini bile uspoređive s vrlo niskim koncentracijama u obližnjim otvorenim vodama Atlantskog oceana.