

# $\gamma$ Irradiation Induced Formation of PCB-Solvent Adducts in Aliphatic Solvents

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$\gamma$  irradiation induced formation of PCB-solvent adducts was investigated as a model for PCB residues in irradiated food. Formation of cyclohexyl adducts of PCBs was found to be significant when pure PCB congeners and Aroclor mixture were irradiated in cyclohexane and cyclohexene. Reaction pathways were investigated, and the effects of oxygen and electron scavenger were studied.

## INTRODUCTION

The radiation chemistry of the most important food components is well documented. However, the effects of  $\gamma$  irradiation on food xenobiotics such as the organochlorinated residues have received considerably less attention. Ceurvel and co-workers (Ceurvel et al., 1974) have observed up to 60% degradation of many chlorinated pesticides in the 0.5-2.5-Mrad dose range in organic solvent. Cichy and co-workers (Cichy et al., 1979) also found a 40% degradation of PCBs in fish fillets irradiated at 1 Mrad, the maximum dose recommended for food irradiation by the Food and Agriculture Organization (FAO) of the United Nations.

Sawai and co-workers (Sawai et al., 1974) have established that upon  $\gamma$  irradiation, PCB degradation proceeds by successive dechlorination of the biphenyl nucleus. This group also observed the formation of addition products between chlorinated aromatics and the solvent upon  $\gamma$  irradiation in 2-propanol (Shimokawa and Sawai, 1977). Buser (1985) even used  $\gamma$  irradiation in dimethyl disulfide to generate mixtures of PCB methyl thioether as standards for the chromatographic analysis of these PCB metabolites.

In a food irradiation perspective, if PCB adduct formation is to be expected, it is likely to be with nonpolar aliphatic molecules because PCBs are essentially concentrated in the lipids of living organisms. This work deals with the  $\gamma$  radiation induced formation of aliphatic solvent adducts of PCBs as a model for food irradiation.

## EXPERIMENTAL PROCEDURES

**Gas Chromatography.** A Varian 3500 gas chromatograph equipped with a fused silica DB-5 column (30 m  $\times$  0.3 mm i.d.) with helium as carrier gas was used. The injections were performed with an on-column injector at 160 °C. The column temperature program was initially set at 70 °C up to 160 °C at 20 °C/min, then to 260 °C at 5 °C/min, and finally to 320 °C at 20 °C/min.

**Mass Spectrometer.** The detector was a Finnigan ITD 800 interfaced to a IBM XT computer. The scanning mass range was from 218 to 508 amu.

**Chemicals.** PCB congeners were purchased from Analab (CT) and were over 99% pure as checked by GC-MS. Cyclohexane was spectrograde, and cyclohexene was distilled under nitrogen prior to use.

**Sample Preparation and Irradiation.** Solvents were deoxygenated by bubbling nitrogen for 15 min. To the PCB congener (50  $\mu$ g) in a 1.5-mL vial was added 0.5 mL of the solvent, and the vial was closed with a screw cap under nitrogen. All samples were prepared in triplicate. Irradiation was performed at 25 °C with a Gammacell 220 with a <sup>60</sup>Co source delivering 1.93 krad/

min for a total dose of 2 Mrad. Decachlorobiphenyl was then added (12.5  $\mu$ g) as internal standard.

## RESULTS AND DISCUSSION

Cyclohexane and cyclohexene were selected as solvents to account for the various unsaturation levels of the lipids found in food. Saturated and unsaturated fatty acids are differently affected by  $\gamma$  irradiation, the latter undergoing dimerization to a considerable extent in comparison with the former (Howton and Wu, 1967). Six PCB congeners bearing from four to eight chlorine substituents were selected. They are numbered according to the Ballschmitter nomenclature (Ballschmitter and Zell, 1980). The congeners listed in Table I are present in considerable amounts in commercial Aroclor mixtures and in food products (Albro et al., 1981; Mes et al., 1989).

The mechanism of  $\gamma$  irradiation induced dechlorination of PCBs is depicted in Figure 1 according to Sawai et al. (1974). Upon  $\gamma$  irradiation, solvated electrons generated from the solvent react through dissociative attachment with PCBs (1a) to produce the dechlorinated PCB radical 1b and a chloride anion. The radical then scavenges a hydrogen from the solvent to give the dechlorinated product 1c. Because oxygen has a high affinity for solvated electrons (Anbar et al., 1973), it efficiently scavenges them and inhibits PCB dechlorination (Singh et al., 1985). To see if such a phenomenon could also affect solvent adduct formation, irradiation was performed with and without exclusion of dissolved oxygen.

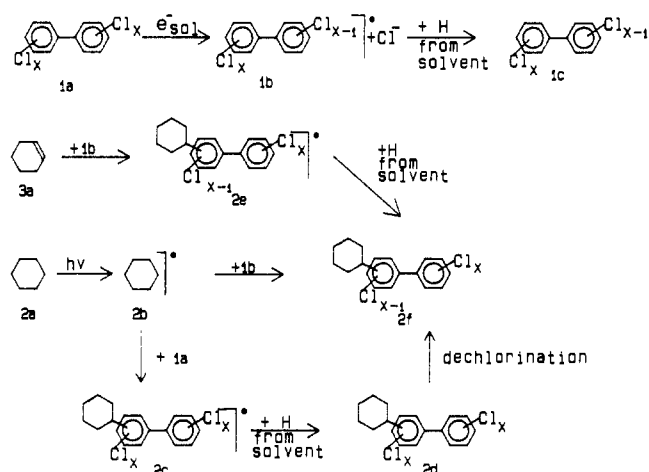
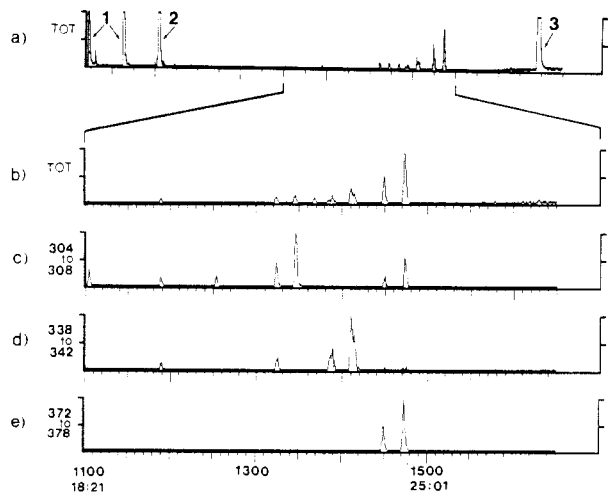
**Irradiation in Cyclohexane.** Figure 2 shows a chromatogram of a 2,2',5,5'-tetrachlorobiphenyl (congener 52) solution in cyclohexane after 2-Mrad irradiation. A series of peaks of dechlorinated congeners appears at a shorter retention time than the starting material. At longer retention times, another series of small peaks appears, and their mass chromatograms indicate that they are monocyclohexyl or even dicyclohexyl adducts of the starting material or its mono-, di-, and tridechlorinated degradation products. The mass fragmentogram of these compounds shows the molecular ion cluster and a favored loss of a 56 amu fragment which corresponds to the loss of C<sub>4</sub>H<sub>8</sub> from the cyclohexyl moiety (Figure 3). The sum of the area of the peaks of identical molecular weight is listed in Table I along with the number of different peaks observed. Individually these peaks represent less than 1% of the starting material but, as a whole, cyclohexyl adducts account, in some cases, for 10% of the starting material (Table III).

In deoxygenated cyclohexane the destruction of the starting material is high and mono-, di-, and tridechlorinated products are obtained in high yields. Cyclohexyl

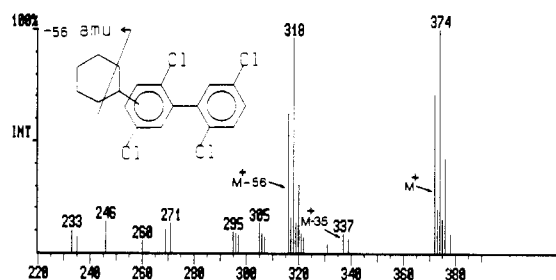
**Table I. Relative Amounts of Degradation Products Obtained upon 2-Mrad Irradiation of PCB Congeners in Cyclohexane<sup>a</sup>**

| starting material  | -1Cl         | -2Cl          | -3Cl            | % dest <sup>b</sup> | -Cl + C <sub>6</sub> H <sub>11</sub> | -2Cl + C <sub>6</sub> H <sub>11</sub> | -3Cl + C <sub>6</sub> H <sub>11</sub> | +C <sub>6</sub> H <sub>11</sub> | +2x C <sub>6</sub> H <sub>11</sub> |
|--------------------|--------------|---------------|-----------------|---------------------|--------------------------------------|---------------------------------------|---------------------------------------|---------------------------------|------------------------------------|
| 52/N <sub>2</sub>  | 20.8(2)[1.8] | 5.5 (2)[0.6]  | ND <sup>c</sup> | 47.3[3.1]           | 2.1 (4) [0.3]                        | 1.0 (4) [0.5]                         | ND                                    | 3.9 (2) [0.3]                   | 1.0 (1) [0.0]                      |
| 52/O <sub>2</sub>  | 10.5(2)[0.6] | 0.6 (2)[0.1]  | ND              | 19.2[4.3]           | 0.2 (2) [0.0]                        | 0.0 (2)                               | ND                                    | 0.2 (2) [0.0]                   | ND                                 |
| 118/N <sub>2</sub> | 33.8(3)[1.1] | 6.2 (4)[0.3]  | 0.7 (2) [0.1]   | 71.6[0.3]           | 1.6 (4) [0.2]                        | 0.9 (4) [0.1]                         | 0.1 (1) [0.0]                         | 0.8 (2) [0.1]                   | ND                                 |
| 118/O <sub>2</sub> | 21.9(3)[1.0] | 0.8 (3)[0.0]  | ND              | 27.0[1.9]           | 0.4 (2) [0.0]                        | 0.0 (1)                               | ND                                    | 0.0 (1)                         | ND                                 |
| 138/N <sub>2</sub> | 30.5(6)[0.7] | 14.8 (6)[2.5] | 1.0 (2) [0.1]   | 70.3[2.2]           | 1.6 (6) [0.0]                        | 0.5 (2) [0.0]                         | 0.1 (1) [0.0]                         | 1.2 (2) [0.1]                   | ND                                 |
| 138/O <sub>2</sub> | 31.8(6)[0.3] | 3.9 (6)[0.8]  | 0.1 (2) [0.0]   | 35.5[0.4]           | 0.2 (1) [0.1]                        | ND                                    | ND                                    | ND                              | ND                                 |
| 153/N <sub>2</sub> | 22.3(3)[0.9] | 7.9 (5)[0.2]  | 0.9 (4) [0.1]   | 72.1[1.5]           | 3.0 (6) [0.3]                        | 0.7 (5) [0.1]                         | 0.0 (1)                               | 1.5 (1) [0.0]                   | ND                                 |
| 153/O <sub>2</sub> | 14.1(3)[0.3] | 1.3 (4)[0.1]  | ND              | 22.6[2.6]           | 0.2 (2) [0.0]                        | ND                                    | ND                                    | ND                              | ND                                 |
| 183/N <sub>2</sub> | 23.4(6)[0.5] | 12.4 (9)[0.4] | 1.2 (9) [0.1]   | 85.3[1.0]           | 4.0 (7) [0.3]                        | 2.2 (11) [0.2]                        | 0.2 (4) [0.0]                         | 1.7 (2) [0.2]                   | ND                                 |
| 183/O <sub>2</sub> | 30.0(6)[0.4] | 3.6 (8)[0.0]  | 0.1 (2) [0.0]   | 36.9[2.3]           | 0.2 (1) [0.0]                        | 0.1 (2) [0.0]                         | ND                                    | ND                              | ND                                 |
| 194/N <sub>2</sub> | 44.1(4)[2.0] | 22.7(12)[0.7] | 5.1 (9) [0.4]   | 70.1[1.2]           | 1.1 (1) [0.2]                        | 0.9 (4) [0.1]                         | 0.3 (3) [0.0]                         | ND                              | ND                                 |
| 194/O <sub>2</sub> | 25.2(4)[1.8] | 2.6(12)[1.3]  | 0.2 (8) [0.1]   | 36.9[2.3]           | ND                                   | ND                                    | ND                                    | ND                              | ND                                 |

<sup>a</sup> Sum of the surface area of relevant peaks relative to the surface area of the unirradiated starting material (in percent). Number in parentheses represents the number of different peaks. Number in brackets is the standard deviation. <sup>b</sup> Percent destruction of the starting material. <sup>c</sup> Not detected.

**Figure 1. Radiation-induced degradation and solvent adduct formation of PCBs.****Figure 2. Mass chromatograms of 2,2',5,5'-tetrachlorobiphenyl (congener 52) after 2-Mrad irradiation in deoxygenated cyclohexane: (a) dechlorinated PCB congeners (1), starting material (2), internal standard (3); (b) enlarged portion of previous chromatogram showing the cyclohexyl adducts; (c) dichlorinated cyclohexyl adducts; (d) trichlorinated cyclohexyl adducts; (e) tetrachlorinated cyclohexyl adducts.**

adducts of mono-, di-, and tridechlorinated isomers are also observed, and their relative abundance follows the same trend as the dechlorinated congeners, monodechlorinated isomers being more abundant than the didechlorinated and tridechlorinated isomers. Reaction of the de-

**Figure 3. Mass fragmentogram of cyclohexyl adducts of 2,2',5,5'-tetrachlorobiphenyl (congener 52).**

chlorinated PCB radical **1b** (Figure 1) with cyclohexyl radical **2b** formed from the solvent upon radiolysis could account for the formation of the adduct **2f**. Interestingly however, mono- and, in one instance, dicyclohexyl adducts of the nondechlorinated starting material can also be observed. Formation of these adducts has to be explained by the electrophilic attack of the cyclohexyl radical **2b** onto the starting material **1a** or its monocyclohexyl adducts. The cyclohexyl radical adducts **2c** then pick up a hydrogen from the solvent to give the adducts **2d**, which are likely to undergo subsequent dechlorination to **2f**.

The electrophilic addition of radicals to PCB has been described by Atkinson (1987). The author found that the reactivity of PCBs toward the hydroxy radical increased with a decrease in the number of chlorine substituents. In Table I the lesser substituted congener 52 generates the largest amounts of nondechlorinated cyclohexyl adducts. It is also the sole congener to produce dicyclohexyl adducts. These two observations along with Atkinson's results support the proposed electrophilic substitution mechanism because a decrease in electron-withdrawing substituents will increase the electron-rich character of the aromatic ring and favor the production of adducts generated by electrophilic free-radical attack. To further support this proposed mechanism, irradiation of congener 52 in cyclohexane was repeated with increasing concentrations of a solvated electron scavenger such as nitroethane. The proposed dechlorinative adduct formation mechanism, which is initiated by dissociative attachment of electrons, should be inhibited by electron scavengers while the nondechlorinated adduct formation pathway should not be affected. Figure 4 shows that indeed, compared to the solution without scavengers, dechlorinated cyclohexyl adduct formation is inhibited in a similar way as is the dechlorination of the starting material, while

**Table II. Relative Amounts of Degradation Products Obtained upon 2-Mrad Irradiation of PCB Congeners in Cyclohexene<sup>a</sup>**

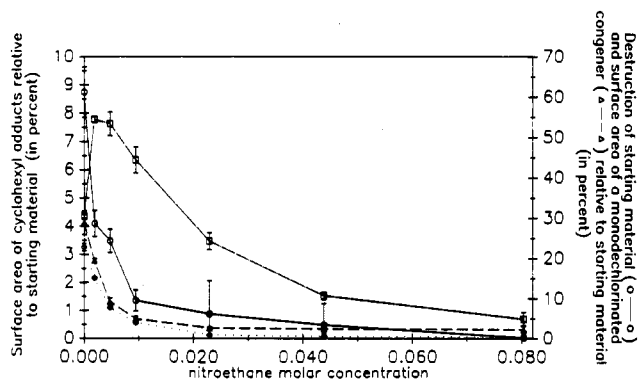
| starting material  | -1Cl         | -2Cl          | -3Cl            | % dest <sup>b</sup> | -Cl + C <sub>6</sub> H <sub>11</sub> | -2Cl + C <sub>6</sub> H <sub>11</sub> | -3Cl + C <sub>6</sub> H <sub>11</sub> | +C <sub>6</sub> H <sub>11</sub> | +2x C <sub>6</sub> H <sub>11</sub> |
|--------------------|--------------|---------------|-----------------|---------------------|--------------------------------------|---------------------------------------|---------------------------------------|---------------------------------|------------------------------------|
| 52/N <sub>2</sub>  | 27.7(2)[0.2] | 7.3 (2)[0.0]  | ND <sup>c</sup> | 49.4[1.0]           | 0.1 (1) [0.0]                        | 0.1 (1) [0.0]                         | ND                                    | ND                              | ND                                 |
| 52/O <sub>2</sub>  | 8.8(2)[0.4]  | 0.3 (1)[0.0]  | ND              | 12.4[1.6]           | ND                                   | ND                                    | ND                                    | ND                              | ND                                 |
| 118/N <sub>2</sub> | 43.3(3)[2.0] | 4.1 (2)[0.6]  | 0.3 (2) [0.1]   | 57.8[3.0]           | 3.0 (2) [0.1]                        | 2.0 (2) [0.3]                         | 0.3 (1) [0.1]                         | ND                              | ND                                 |
| 118/O <sub>2</sub> | 16.4(3)[0.7] | 0.2 (1)[0.0]  | ND              | 19.2[0.9]           | 2.0 (2) [0.2]                        | 0.1 (1) [0.0]                         | ND                                    | ND                              | ND                                 |
| 138/N <sub>2</sub> | 33.5(6)[2.2] | 15.7 (4)[0.4] | 1.7 (2) [0.4]   | 67.4[1.5]           | 2.3 (3) [0.2]                        | 0.8 (2) [0.1]                         | 0.4 (1) [0.1]                         | ND                              | ND                                 |
| 138/O <sub>2</sub> | 23.9(6)[0.3] | 1.9 (4)[0.0]  | ND              | 25.6[1.4]           | 0.5 (1) [0.1]                        | 0.1 (1) [0.0]                         | ND                                    | ND                              | ND                                 |
| 153/N <sub>2</sub> | 22.1(3)[0.5] | 5.1 (5)[0.3]  | 0.2 (4) [0.1]   | 55.7[0.8]           | 7.7 (2) [0.1]                        | 2.5 (5) [0.1]                         | 0.1 (1) [0.0]                         | ND                              | ND                                 |
| 153/O <sub>2</sub> | 11.5(3)[0.6] | 0.3 (4)[0.0]  | ND              | 24.0[2.2]           | 4.0 (2) [0.2]                        | 0.2 (2) [0.0]                         | ND                                    | ND                              | ND                                 |
| 183/N <sub>2</sub> | 40.0(6)[1.0] | 12.0 (7)[2.8] | 0.36 (1) [0.1]  | 66.7[1.1]           | 4.9 (2) [0.2]                        | 5.3 (5) [0.6]                         | 0.3 (1) [0.1]                         | ND                              | ND                                 |
| 183/O <sub>2</sub> | 27.5(6)[1.1] | 1.8 (4)[0.1]  | ND              | 28.8[2.8]           | 2.2 (2) [0.3]                        | 0.6 (2) [0.1]                         | ND                                    | ND                              | ND                                 |
| 194/N <sub>2</sub> | 39.1(4)[2.6] | 27.2(12)[0.6] | 4.6 (8) [0.3]   | 70.3[1.2]           | 1.3 (1) [0.1]                        | 0.4 (1) [0.0]                         | 0.5 (2) [0.0]                         | ND                              | ND                                 |
| 194/O <sub>2</sub> | 32.6(4)[1.2] | 3.9 (8)[0.4]  | 0.1 (1) [0.0]   | 24.8[2.9]           | 1.1 (1) [0.2]                        | ND                                    | ND                                    | ND                              | ND                                 |

<sup>a</sup> See Table I footnotes.

**Table III. Total Amounts of PCB Cyclohexyl Adducts upon 2-Mrad Irradiation<sup>a</sup>**

| starting material | in cyclohexane  |                 | in cyclohexene  |                 |
|-------------------|-----------------|-----------------|-----------------|-----------------|
|                   | /N <sub>2</sub> | /O <sub>2</sub> | /N <sub>2</sub> | /O <sub>2</sub> |
| 52                | 7.9 [0.6]       | 0.5 [0.0]       | 0.3 [0.0]       | ND <sup>b</sup> |
| 118               | 3.3 [0.4]       | 0.5 [0.0]       | 5.3 [0.4]       | 2.1 [0.2]       |
| 138               | 3.5 [0.2]       | 0.2 [0.1]       | 3.5 [0.1]       | 0.6 [0.1]       |
| 153               | 5.2 [0.3]       | 0.2 [0.0]       | 10.3 [0.2]      | 4.2 [0.1]       |
| 183               | 8.0 [0.7]       | 0.3 [0.0]       | 10.5 [0.8]      | 2.9 [0.3]       |
| 194               | 2.4 [0.3]       | 0.3 [0.0]       | 2.2 [0.1]       | 1.1 [0.2]       |

<sup>a</sup> Sum of the relative surface area of relevant peaks from Tables I and II. Number in brackets is the standard deviation. <sup>b</sup> Not detected.



**Figure 4.** Effect of increasing concentration of nitroethane on congener 52 dechlorination (○) and on the production of a dechlorinated congener (△) and monodechlorinated (◇) and non-dechlorinated (□) cyclohexyl adducts production upon 2-Mrad irradiation in deoxygenated cyclohexane. Left y axis is the peak area of adducts relative to the area of the starting material (in percent). Right y axis is the percentage destruction of the starting material and the peak area of a monodechlorinated congener relative to the starting material (in percent).

the nondechlorinated adducts actually increased for a certain concentration range of nitroethane. This increase is due to dechlorination inhibition of the nondechlorinated adducts (2d-f), which indicates that this pathway is also involved in the formation of the dechlorinated cyclohexyl adducts.

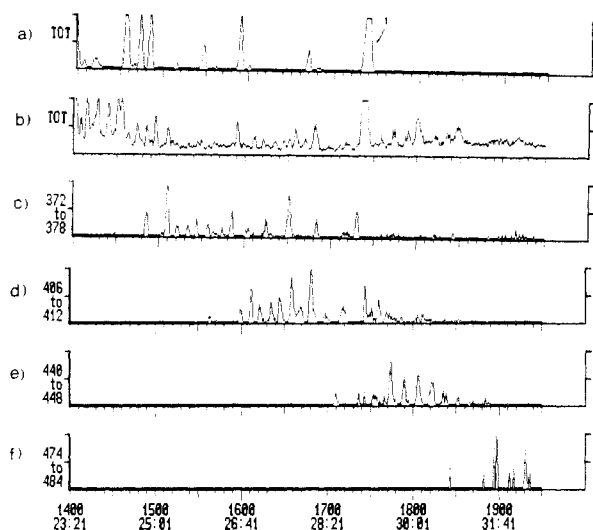
When irradiation was performed in cyclohexane in the presence of dissolved oxygen, the degradation of the starting material decreased by a factor of 2-2.5 and adduct formation by a factor of 7-16 (Table III). Scavenging of solvated electrons by oxygen explains the former observation, and the affinity of oxygen for aliphatic radicals such as the cyclohexyl radical can explain the additional inhibition of adduct formation.

**Irradiation in Cyclohexene.** The chromatograms of

the deoxygenated cyclohexene solutions after irradiation show a relatively small number of peaks having retention times identical with those observed in deoxygenated cyclohexane. The mass spectra of these peaks are identical with those observed in the saturated solvent, and no cyclohexene adducts can be detected by mass spectrometry. The degradation extent of the starting material is somewhat less than in deoxygenated cyclohexane, and no mono- or dicyclohexyl adducts of the nondechlorinated starting material could be observed (Table II). These data suggest that in cyclohexene the sole free-radical species involved in adduct formation is the dechlorinated PCB radical 1b which adds to the double bond of cyclohexene to form the cyclohexyl PCB radical 2e which ultimately scavenges a hydrogen atom from the solvent to give the cyclohexyl adduct 2f. Contrary to the observations in cyclohexane, adduct formation is minimal with congener 52 compared to that with the other more substituted congeners (Table III). This supports the idea that the PCB radical intermediate 1b is the electrophilic species because a decrease in the number of electron-withdrawing substituents should decrease the electrophilic character of such a radical and consequently decrease adduct formation.

When oxygen is present, the degradation of the starting material and adduct formation are reduced by similar factors, in contrast with the cyclohexane experiment (Table III). This further supports the proposed decomposition pathway in which the only radical species involved is the PCB radical 1b, in contrast to the reactions in cyclohexane in which the cyclohexyl radical 2b is also involved and susceptible to scavenging by oxygen.

**Irradiation of Aroclor Mixtures.** Because PCB residues are present in food as complex mixtures, Aroclor 1260, a commercial PCB mixture that contains an average of six chlorine substituents per biphenyl, was irradiated in deoxygenated solutions of the two solvents. Figure 5 shows the terminal end of the chromatogram of Aroclor 1260 along with the total and mass chromatogram of the sample after irradiation in cyclohexane. The sum of the relative peak area of the molecular ions of the various chlorinated cyclohexyl adducts is presented in Table IV. In both solvents the most abundant cyclohexyl adducts are those containing five chlorines, which reflects the importance of the dechlorinative adduct formation pathway observed in the two solvents, considering that, on average, hexachlorinated congeners are predominant in Aroclor 1260. In cyclohexane, where adduct formation also occurs without dechlorination, the relative amount of hexachlorinated adducts is larger than in cyclohexene.



**Figure 5.** Mass chromatograms of the latest eluting PCB congeners of Aroclor 1260: (a) before irradiation, internal standard (1); (b) after 2-Mrad irradiation in deoxygenated cyclohexane; (c) tetrachlorocyclohexyl adducts; (d) pentachlorocyclohexyl adducts; (e) hexachlorocyclohexyl adducts; (f) heptachlorocyclohexyl adducts.

**Table IV. Relative Amounts of PCB Cyclohexyl Adducts upon 2-Mrad Irradiation of Aroclor 1260 in Deoxygenated Solvent<sup>a</sup>**

| adducts                              | cyclohexane <sup>b</sup> | cyclohexene <sup>c</sup> |
|--------------------------------------|--------------------------|--------------------------|
| 3Cl + C <sub>6</sub> H <sub>11</sub> | 0.05 [0.00] <sup>d</sup> | ND <sup>e</sup>          |
| 4Cl + C <sub>6</sub> H <sub>11</sub> | 0.25 [0.02]              | 0.33 [0.01]              |
| 5Cl + C <sub>6</sub> H <sub>11</sub> | 0.44 [0.00]              | 0.50 [0.07]              |
| 6Cl + C <sub>6</sub> H <sub>11</sub> | 0.27 [0.02]              | 0.17 [0.01]              |

<sup>a</sup> Calculated by using the sum of surface area of the various molecular ion clusters. <sup>b</sup> Duplicate samples. <sup>c</sup> Triplicate samples. <sup>d</sup> Number in brackets is the standard deviation. <sup>e</sup> Not detected.

## CONCLUSIONS

The results presented in this study demonstrate that at the radiation dose used PCBs react to a considerable extent with saturated and unsaturated aliphatic molecules. Adduct formation is maximum in deoxygenated solvent, and the inhibition by oxygen is reduced in the presence of unsaturated solvent molecules.

To see to what extent these results can be applied to food irradiation, other organic matrices such as fatty acid

esters and triglycerides should be used. This work is currently under way in our laboratories.

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**Registry No.** O<sub>2</sub>, 7782-44-7; PCB congener 52, 35693-99-3; PCB congener 118, 31508-00-6; PCB congener 138, 35065-28-2; PCB congener 153, 35065-27-1; PCB congener 183, 52663-69-1; PCB congener 194, 35694-08-7; arochlor, 62251-11-0; cyclohexane, 110-82-7; cyclohexene, 110-83-8.