

Solvent-driven Radiolytic Transformation of Hcb in Propan-2-ol Under Gamma Irradiation

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Research Article

Keywords: Hexachlorobenzene, POPs, Environmental remediation, Detoxification

Posted Date: August 7th, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-10536593/v1>

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Additional Declarations: No competing interests reported.

Abstract

Hexachlorobenzene (HCB), a persistent and toxic organochlorine pollutant, was transformed by Co-60 γ -radiolysis in isopropanol over an absorbed dose range of 0–169.5 kGy. GC–MS analysis identified successive reductive dechlorination products, including pentachlorobenzene, 1,2,4,5-tetrachlorobenzene, and 1,2,3-trichlorobenzene, together with oxygenated and aliphatic compounds derived from isopropanol radiolysis. Product evolution indicated dose-dependent dechlorination accompanied by solvent-radical reactions. A plausible mechanism involving electron-induced C–Cl bond cleavage, hydrogen abstraction, oxidation, radical recombination, cyclization, and rearrangement was proposed. These findings provide mechanistic insight into the γ -radiolytic transformation of HCB in organic media.

1. Introduction

HCB is one of the most persistent organochlorine pollutants due to its highly chlorinated aromatic structure, exceptional chemical stability, hydrophobicity, and resistance to natural degradation processes [1, 2]. These properties enable HCB to persist in soil, sediments, and aquatic environments for extended periods, where it bioaccumulates in living organisms and biomagnifies through the food chain, posing significant risks to both ecosystems and human health [3]. Because of its carcinogenicity, long-range environmental transport, and persistence, HCB has been listed as a persistent organic pollutant under the Stockholm Convention, necessitating the development of efficient technologies for its destruction rather than simple containment or transfer [4].

Conventional remediation technologies, including adsorption, chemical oxidation, thermal treatment, and bioremediation, have been investigated for HCB removal. However, these approaches frequently suffer from drawbacks such as incomplete mineralization, high operational costs, secondary waste generation, or the formation of toxic transformation products [5]. Consequently, ionizing radiation has emerged as an attractive advanced treatment technology because it generates highly reactive transient species capable of initiating rapid dechlorination and molecular transformation without the addition of chemical reagents [6, 7, 8, 9, 10]. In organic media, γ -irradiation produces solvent-derived radicals that participate in electron-transfer, hydrogen abstraction, and radical substitution reactions, promoting the sequential cleavage of carbon–chlorine bonds and the degradation of highly chlorinated aromatic compounds [11, 12].

Previous studies on radiation-induced degradation of chlorinated organic pollutants have primarily focused on degradation efficiency, reaction kinetics, and radiation chemical yields. Although these investigations have confirmed the effectiveness of γ -radiolysis for the decomposition of chlorinated benzenes, comparatively little attention has been paid to the evolution of intermediate products during the degradation process [13]. The formation, accumulation, and subsequent disappearance of these byproducts provide essential information for understanding dechlorination pathways, solvent participation, and the overall reaction mechanism. Such knowledge is also necessary for evaluating whether the treatment process leads to environmentally safer products rather than merely transforming the parent pollutant into equally hazardous intermediates.

Among organic solvents, isopropanol represents an interesting reaction medium because its radiolysis generates reducing radical species that can effectively promote dechlorination while simultaneously participating in secondary radical reactions [14]. These processes influence both the identity and abundance of degradation products, resulting in byproduct profiles that differ from those observed in other alcohols. Therefore, investigating the temporal evolution of radiolytic products in isopropanol is important for clarifying solvent-dependent reaction pathways and improving the selectivity of radiation-based remediation processes.

In this study, the γ -radiolytic degradation of HCB in isopropanol was investigated with particular emphasis on the dynamics of degradation byproducts. GC–MS was employed to identify intermediate and final products formed throughout irradiation, and their changes with absorbed dose were systematically evaluated. Based on the observed product evolution, possible degradation pathways are proposed to provide mechanistic insight into the radiation-induced transformation of HCB in isopropanol and to support the development of more effective radiation-based technologies for the remediation of persistent chlorinated pollutants.

2. Materials and Methods

2.1. *Chemicals.* HCB (analytical grade, purity $\geq 99\%$) was used as the target compound without further purification. Isopropanol (HPLC grade) served as the reaction medium. Its selected properties are given in Table 1.

Table 1
Selected physicochemical properties of isopropanol.

Relative Polarity	Dielectric Constant	Viscosity (mPa·s, 25°C)	Dipole Moment (D)	Density (g/cm³)	Ionizing energy (eV)
0.546 [15]	19.92 [16]	2.038 [17]	1.59 [18]	0.785 [16]	10.17 [19]

Standard solutions were prepared by dissolving HCB in isopropanol to obtain a homogeneous solution with an initial concentration of 0.055 g L⁻¹. All solutions were freshly prepared before irradiation and stored in sealed glass vials protected from light.

2.2. *Gamma irradiation.* The samples were irradiated using a ⁶⁰Co γ -radiation source (MRX- γ -25) at room temperature. The absorbed dose was determined using Fricke dosimetry, while alanine dosimeters were employed for routine dose verification. Dose rate of the source was 1.74 Gy s⁻¹ and samples were irradiated over an absorbed dose range of 0–169.5 kGy, corresponding to irradiation times of up to 27 h. All experiments were performed in triplicate to ensure reproducibility.

2.3. GC–MS analysis. Radiolytic products were identified using a Shimadzu QP2010 SE GC-MS equipped with an Rxi-5ms capillary column. Electron ionization (70 eV) was operated in both full-scan and selected ion monitoring (SIM) modes. Helium was used as the carrier gas at a constant flow rate of 1.0 mL min⁻¹. A 1 µL sample was injected with a split ratio of 20:1. The oven temperature program was operated from 40 to 320°C. Chromatographic data acquisition and processing were performed using LabSolutions v5.92, and compound identification was achieved by comparison with the NIST mass spectral library together with retention time analysis.

2.4. Evaluation of byproduct dynamic. The evolution of degradation products was investigated by comparing GC–MS chromatograms obtained at different absorbed doses. Peak areas of identified compounds were integrated under identical instrumental conditions and normalized to enable comparison among irradiation stages. Particular attention was given to the sequential formation and disappearance of chlorinated benzene intermediates and solvent-derived products, allowing the temporal evolution of radiolytic byproducts to be evaluated throughout the degradation process.

2.5. Data analysis. GC–MS peak integration and spectral interpretation were performed. Product identities were accepted only when supported by satisfactory agreement with the NIST library and chromatographic behavior. The observed changes in byproduct abundance with increasing absorbed dose were used to propose degradation pathways and elucidate the reaction mechanism of γ-radiolytic transformation of HCB in isopropanol.

3. Results and discussion

3.1. GC–MS identification of radiolytic products. A representative GC–MS chromatogram of the HCB solution irradiated for 2 h is shown in Fig. 1.

The chromatogram demonstrates successful separation of the radiolytic products formed during γ-irradiation. Besides the residual HCB peak, several additional peaks corresponding to chlorinated and non-chlorinated compounds were detected. Representative mass spectra of the major chlorinated degradation products are presented in Fig. 2. The mass spectra of the chlorinated intermediates have been reported previously and are therefore not reproduced here; they are available in [14].

Table 2 lists the radiolytic products identified during the γ-irradiation of HCB in isopropanol, together with their molecular ions and some hazard information. The identified compounds include partially dechlorinated chlorobenzenes together with several oxygenated and aliphatic products formed through the radiolysis of isopropanol and subsequent radical reactions. These identified products are discussed further in terms of their dose-dependent evolution and proposed formation pathways in the following sections.

3.2 Dynamics of byproduct formation and their hazard information. Figure 3 shows the evolution of the relative GC–MS peak-area composition during γ-irradiation of HCB in isopropanol.

Table 2. Identified radiolytic products formed during the γ-irradiation of HCB in isopropanol and their reported hazard information.

No	Byproduct	CAS	Molecular ion M ⁺	Hazards identification			
				Carcinogenicity	General toxicity	Hazardous to the aquatic environment	
						Short term (Acute)	Long term (Chronic)
1	HCB	118-74-1	282	Category 1B	Carcinogenic	Category Acute 1	Category Chronic 1
2	Acetone	67-64-1	58	Not available	Skin, Eye irritation	Not available	Not available
3	n-Dodecane	112-40-3	170	Not available	Aspiration Toxicity	Insoluble in water, May persist	
4	n-Tetradecane	629-59-4	198	Not available	Aspiration Toxicity	Not available	
5	n-Pentadecane	629-62-9	212	Not available	Not available	Readily biodegradable	
6	Pentachlorobenzene	608-93-5	248	Not available	Oral	Category Acute 1	Category Chronic 1
7	2-Methyl-2,4-pentanediol	107-41-5	118	Not available	Skin, reproductive, eye	Persistence is unlikely	
8	l-Limonene	5989-54-8	136	Not available	Aspiration, Reproductive	Not available, may persist	
9	1,2,3-TCB	87-61-6	180	Not available	Acute toxic, oral	Category Acute 1	Category Chronic 1
10	TETRAHYDRO-2,2-DIMETHYL-5-(1-METHYLETHYL)-Furan	696-59-3	142	Not available	Aspiration, Eye irritation	Not available, Persistence is unlikely	
11	cis-3-Hexenyl butyrate	16491-36-4	170	Not available	Skin, Eye irritation	Not available	
12	2-Butenoic acid, propyl ester	87-56-9	128	Not available	Respiratory, Skin, Eye	Harmful to aquatic organisms, Persistence is unlikely	
13	6-HEPTEN-2-OL, 2,6-DIMETHYL-	13254-34-7	142	Not classified	Skin, Eye irritation	Not classified as hazardous	
14	1,2,4,5-TeCB	95-94-3	214	Not classified	Respiratory, Skin, Eye	Category Acute 1	Category Chronic 1
15	2,5-Dimethyl-2,5-hexanediol	110-03-2	146	Not available	Eye damage	Not available	
16	Ethyl isobutyrate	97-62-1	116	Not available	Respiratory, eye, skin	Not available	

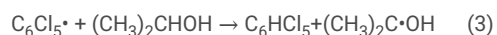
The relative abundance of HCB decreased progressively with increasing absorbed dose, accompanied by the formation of several transient chlorinated intermediates, including pentachlorobenzene and 1,2,3-trichlorobenzene, at low and intermediate doses. These intermediates subsequently diminished or disappeared with continued irradiation, indicating their further transformation. At higher absorbed doses, the product distribution shifted toward oxygenated and aliphatic compounds derived predominantly from isopropanol radiolysis together with lower-chlorinated aromatic products such as 1,2,4,5-tetrachlorobenzene. This progressive change in product composition suggests the sequential transformation of HCB through intermediate species rather than the accumulation of stable chlorinated byproducts.

3.3 Radical mechanism and radiolytic transformation of HCB in isopropanol. The products identified by GC–MS (Table 2) and their dose-dependent evolution (Fig. 3) indicate that the γ -irradiation of HCB in isopropanol proceeds through multiple concurrent radical reactions. The sequential appearance and disappearance of chlorinated aromatic intermediates, together with the formation of oxygenated and aliphatic compounds, suggest the involvement of both reductive dechlorination of HCB and solvent-derived radical chemistry. Based on these experimental observations a plausible transformation pathway is proposed in Fig. 4.

Ionizing radiation induces the radiolysis of isopropanol, generating reactive species including solvated electrons e^-_{sol} , hydrogen atoms ($H^\bullet$), and α -hydroxyisopropyl radicals $(CH_3)_2C^\bullet OH$ [14]. Among these species, the solvated electron is considered the primary reducing agent responsible for the dechlorination of HCB. Electron attachment to HCB produces a transient radical anion, which undergoes dissociative cleavage of a C–Cl bond with the release of chloride ions and the formation of a pentachlorophenyl radical:

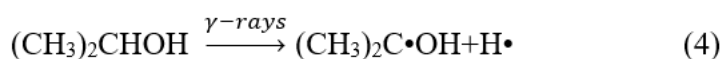


The resulting pentachlorophenyl radical subsequently abstracts a hydrogen atom from isopropanol, yielding pentachlorobenzene while simultaneously generating an α -hydroxyisopropyl radical.



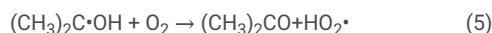
Pentachlorobenzene subsequently undergoes the same reaction sequence described in Reactions (1)–(3), leading to the formation of tetrachlorobenzene. Repetition of this reductive dechlorination pathway results in the successive formation of lower chlorinated benzenes. While 1,2,4,5-tetrachlorobenzene and 1,2,3-trichlorobenzene were identified in the present study, the subsequent formation of dichlorobenzene and monochlorobenzene has been reported in previous studies and is included in the proposed pathway for completeness.

Pathway B represents the formation of carbonyl, ester, and cyclic ether products derived primarily from the radiolysis of isopropanol.



The α -hydroxyisopropyl radical is the predominant intermediate and serves as the precursor for several oxygenated products through oxidation, radical recombination, and rearrangement reactions.

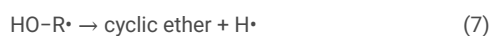
In the presence of dissolved oxygen, the α -hydroxyisopropyl radical readily undergoes oxidation to produce acetone.



The formation of acetone as the dominant oxygenated product at low absorbed doses indicates that oxidation of solvent-derived radicals is one of the primary reactions during γ -irradiation. Subsequent oxidation of carbon-centered radicals generates carbonyl- and carboxyl-containing intermediates, which can further react with alcohols present in the solution to produce ester compounds.



The detected tetrahydro-2,2-dimethyl-5-(1-methylethyl)furan is proposed to originate from the intramolecular cyclization of oxygenated radical intermediates, followed by hydrogen abstraction.



Under γ -irradiation, solvent-derived carbon-centered radicals undergo hydrogen abstraction, radical recombination, disproportionation, and rearrangement reactions, leading to the formation of a variety of oxygenated and hydrocarbon products. The identified 2-methyl-2,4-pentanediol, 6-hepten-2-ol, 2,6-dimethyl-, and 2,5-dimethyl-2,5-hexanediol are proposed to originate from the recombination and subsequent stabilization of radical intermediates. Similarly, the detected n-dodecane, n-tetradecane, n-pentadecane, and l-limonene are attributed to secondary radical recombination and rearrangement reactions involving solvent-derived intermediates. Collectively, the formation of these oxygenated, aliphatic, and terpenoid products further highlights the complex radical-mediated, non-selective transformation pathways of isopropanol under γ -irradiation.

4. Conclusion

The γ -radiolytic transformation of HCB in isopropanol was investigated using GC–MS to identify the radiolytic products and elucidate the underlying reaction pathways. The results demonstrated the progressive transformation of HCB with increasing absorbed dose, accompanied by the formation of chlorinated aromatic intermediates, oxygenated compounds, aliphatic hydrocarbons, and terpenoid products. Hazard assessment based on reported classifications indicated that several transformation products exhibit lower reported hazards than the parent compound, although some chlorinated intermediates remain environmentally relevant.

The identified products and their dose-dependent evolution support a reaction mechanism involving successive reductive dechlorination of HCB together with concurrent solvent-derived radical reactions. Electron-induced C–Cl bond cleavage followed by hydrogen abstraction accounted for the formation of lower chlorinated benzenes, while oxidation, radical recombination, cyclization, and rearrangement of isopropanol-derived intermediates explained the formation of the remaining byproducts. The proposed reaction pathways provide a plausible mechanistic interpretation consistent with the experimental observations and previously reported radiolytic reactions.

Overall, this study improves the understanding of the γ -radiolytic transformation of HCB in isopropanol and provides mechanistic insights into the degradation behavior of chlorinated aromatic pollutants under ionizing radiation. These findings may contribute to the future development and optimization of radiation-based approaches for the treatment of persistent chlorinated organic contaminants.

Declarations

Funding

This research did not receive funding.

Author Contribution

Samir Karimov: Writing – review & editing, Writing – original draft, Software, Resources, Methodology, Investigation. Elshad Abdullayev: Writing – review & editing, Investigation. Muslum Gurbanov: Writing – review & editing, Investigation. Lala Gasimzada: Visualization, Conceptualization, Investigation. Gulnara

Acknowledgments

The authors express their sincere appreciation to **ASOIU** and the **Institute of Physics** for institutional support and for providing access to the γ -irradiation facilities.

Data Availability

The datasets generated and analyzed during the current study can be obtained from the corresponding author upon reasonable request.

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Figures

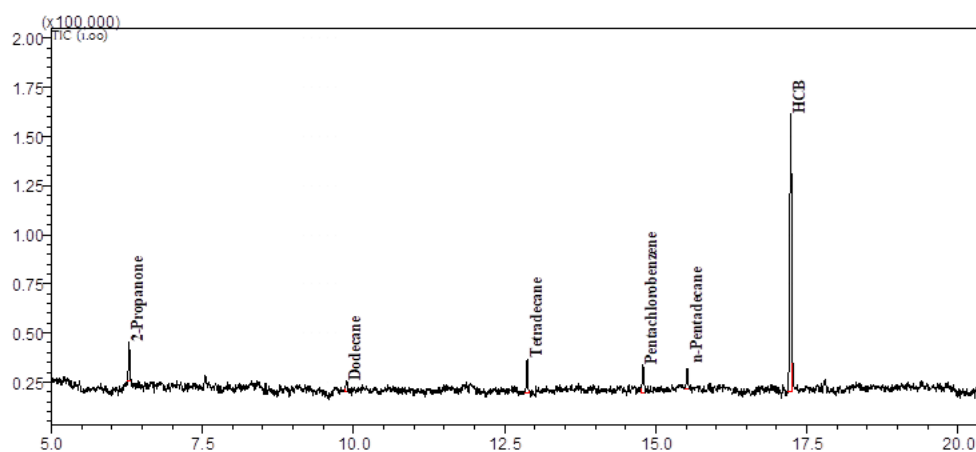


Figure 1

Representative GC–MS chromatogram of the HCB solution after 2 h of γ -irradiation, showing the separation of the identified radiolytic byproducts.

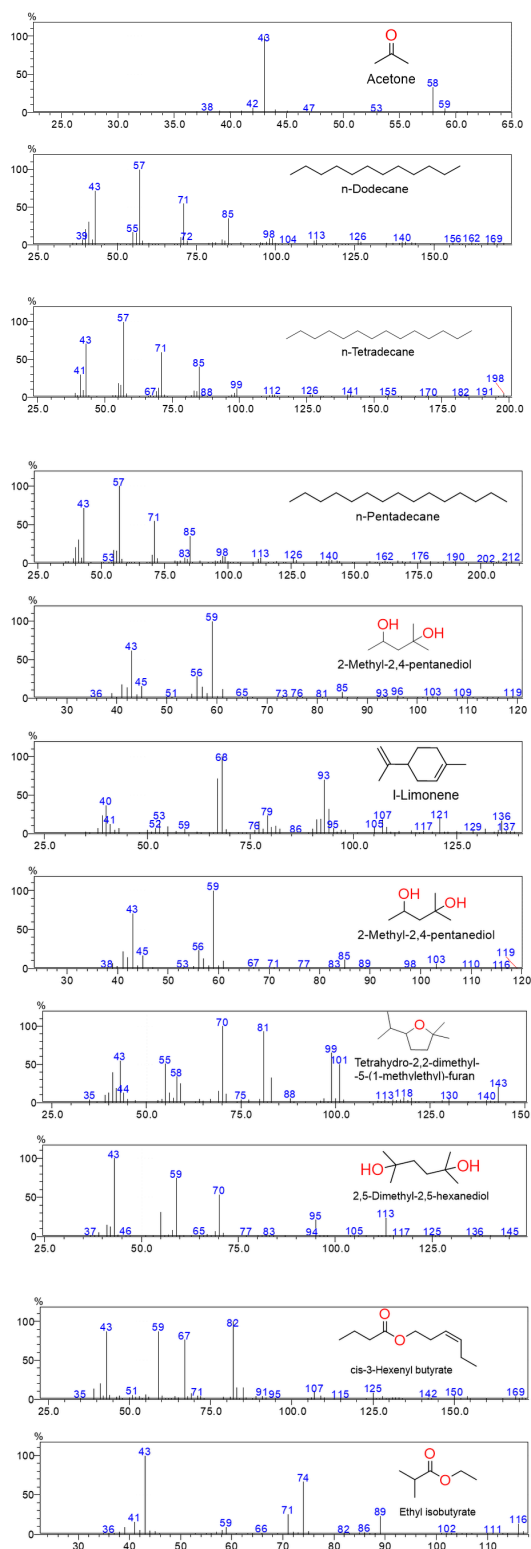


Figure 2

Experimental mass spectra of selected radiolytic products identified during the γ -irradiation of HCB in isopropanol.

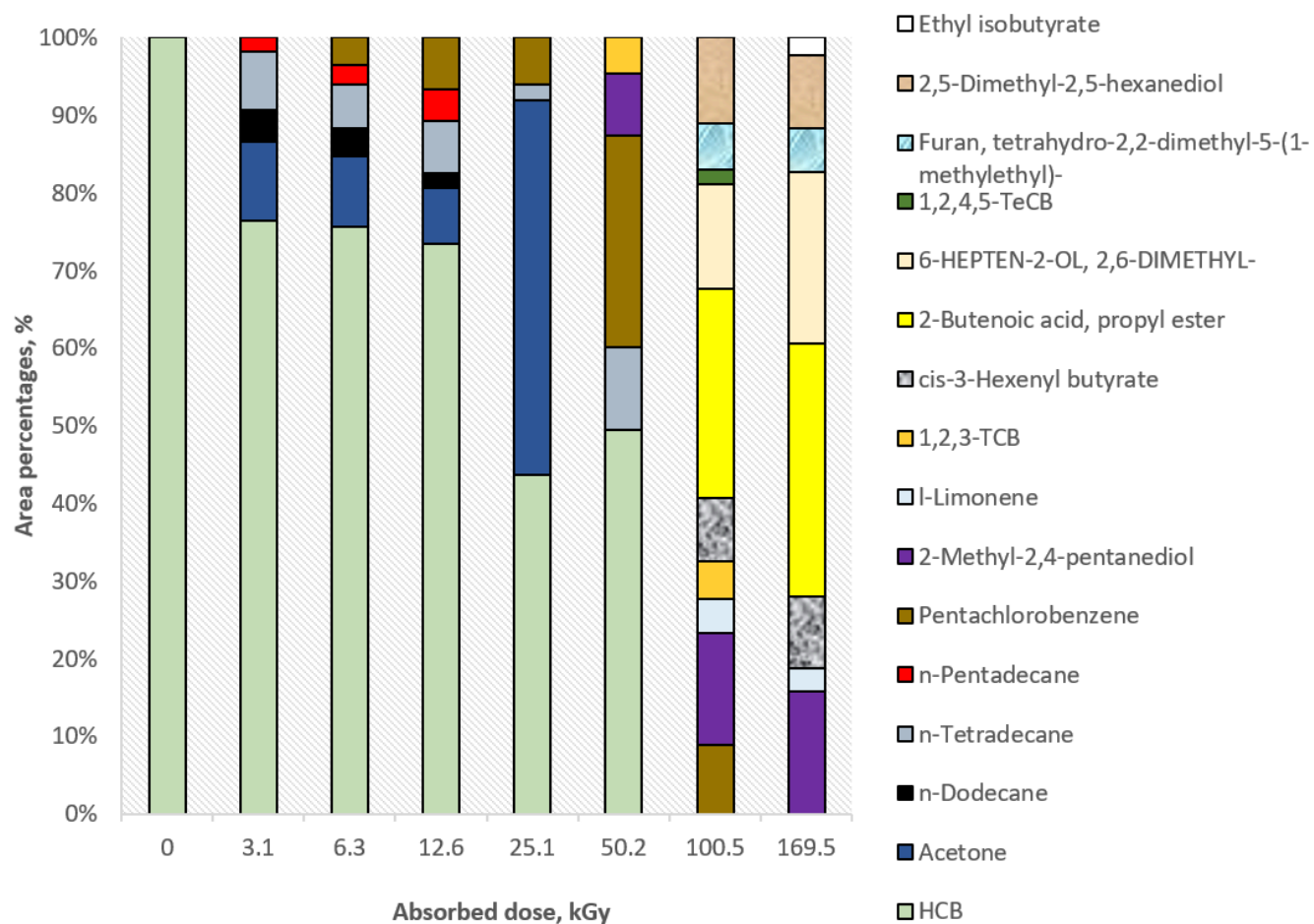


Figure 3

Evolution of the relative GC–MS peak-area composition of HCB and the identified radiolytic products during γ -irradiation of HCB in isopropanol as a function of absorbed dose.

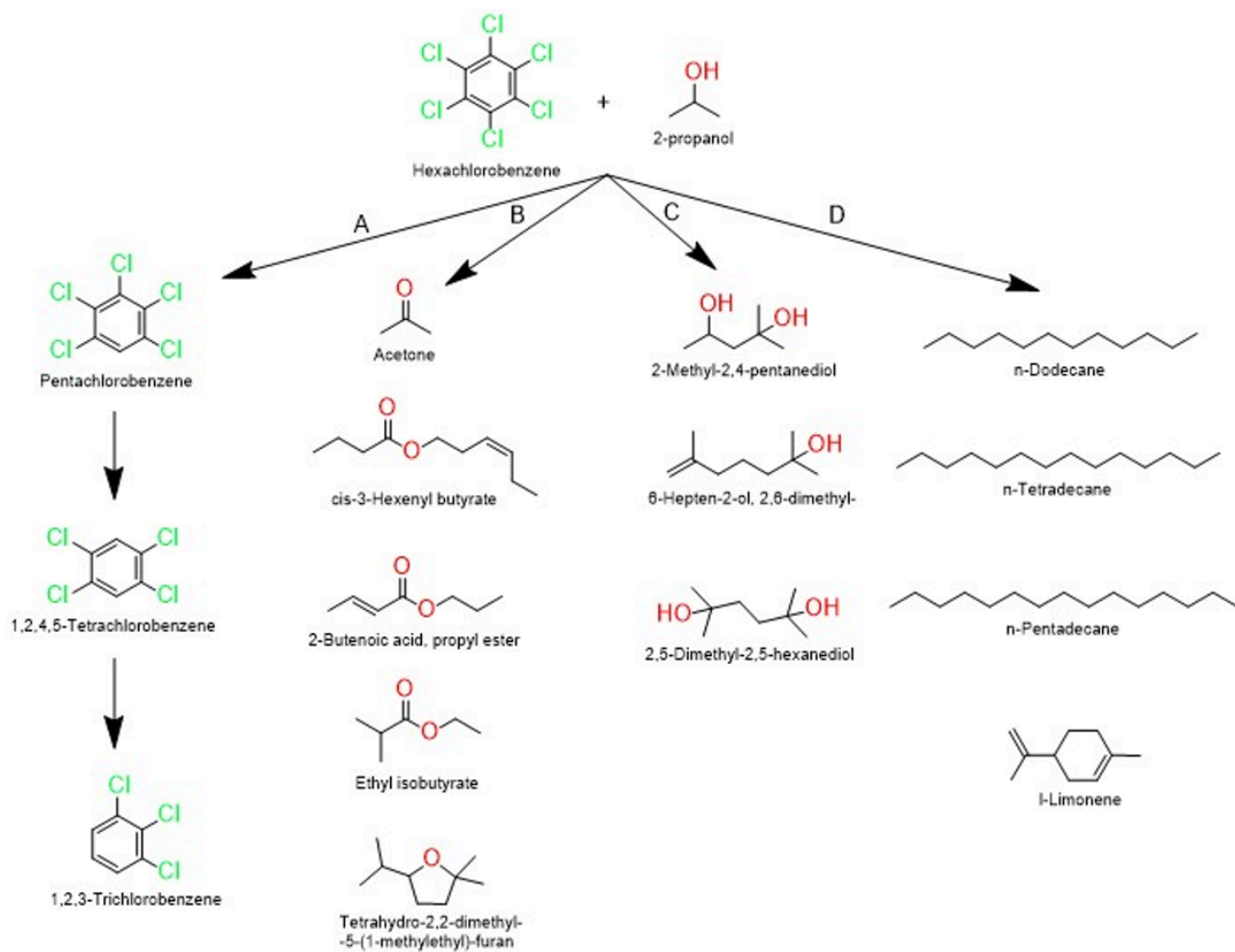


Figure 4

Proposed pathways for the formation of radiolytic products during the γ -irradiation of HCB in isopropanol: (A) successive reductive dechlorination of HCB; (B) formation of carbonyl, ester, and cyclic ether products; (C) formation of alcohols and