ECOLOGICAL FATE OF ORGANIC POLLUTANTS: HEXABROMOCYCLODODECANE

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ABSTRACT

This report discusses the physicochemical properties, applications, and environmental impacts of 1, 2, 5, 6, 9, 10-hexabromocyclododecane (HBCD), a widely used brominated flame retardant. The report highlights HBCD's persistence, bioaccumulation, long-range transport and toxicity profile, as well as its potential for water pollution and human health impacts.

KEYWORDS: Flame Retardant, Diastereomer, Persistence, Bioaccumulation, Solubility, Distribution Coefficient, Butadiene, Photochemical Decomposition, Biodegradation, Half-Life, Dehalogenation, Bioconcentration Factor, Expanded Polystyrene, Phenolic Foam, Fire Safety.

INTRODUCTION

The compound 1,2,5,6,9,10 hexabromocyclododecane (HBCD, C12H18Br6) has been produced since the 1960s and is the most used cycloaliphatic brominated flame retardant (BFR) additive today. The main industry that uses HBCD is construction, where extruded or expanded polystyrene foam products typically contain 3% or less by weight. Upholstered furniture, car seats, interior fabrics, and electrical and electronic devices are some examples of recycling. Global production of HBCD was 31,000 tonnes in 2011, of which 11,000 tonnes were used in the EU in 2007 (POPRC, 2011).

End products include insulation and packaging materials, electrical and electronic parts (as well as textile coatings (polymer dispersions). According to industry information, HBCD is mainly used (90%) for flame retardant polystyrene (EU, 2008), predominantly foaming polystyrene (EPS) and extruded polystyrene (XPS). While the production of polymer dispersions for textiles may be more limited. This category is important to consider as the dominant category of emissions into wastewater and surface waters is estimated to be textile backsheet (industrial) (EU, 2008).

Technical HBCD (t-HBCD), which is produced by adding bromine to 1,5 9-cyclododecatriene, mainly consists of diastereomers: α , β and γ . The proportions of these isomers in t-HBCD ultimately vary depending on the manufacturer, although the γ -isomer makes up more than 70% of the total, while the α - and β -isomers make up approximately 10% and 6% of the mixture, respectively. T-HBCD contains traces of other diastereomers (such as δ and ϵ), but quantitative

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data are still not available. Due to its persistence, bioaccumulation, long-range transport and toxicity profile, HBCD was placed on the US Environmental Protection Agency's List of Chemicals of Concern in 2010, and in the same year HBCD was nominated and is being considered for listing. persistent organic pollutants (POPs) by the Persistent Organic Pollutants Review Committee (POPRC) under the Stockholm Convention (Encyclopedia of Toxicology, 3rd edition, 2014).



 α -HBCD (CAS 134237-50-6), the line indicates a mirror plane

 β -HBCD (134237-51-7), the line indicates a mirror plane



 γ -HBCD (134237-52-8), the line indicates a mirror plane

Figure 1. Three major diastereomers (pairs of enantiomers) of t-HBCD (reconstructed from EC (2008); data from (Heebetal. 2005)).

PHYSICOCHEMICAL CHARACTERISTICS

The CAS registry lists two numbers for unspecified combinations of commercial or t-HBCD: 25637-99-4 (without bromine substitution pattern location numbering) and 3194-55-6 for 1,2,5,6, 9, and 10 HBCD. HBCD has complex stereochemistry, comprising 16 stereoisomers, and can isomerize both during the manufacturing process and outside the environment. Most of the chemical characterization and toxicity tests published to date are on t-HBCD, a mixture of isomers.

"REACH" link Application, §	Property	Meaning	Comments
VII, 7.1	Physical state at 20 and 101.3 kPa	White solid, odorless	
VII, 7.2	Melting temperature	fluctuates around: 172–184 °C as the average was used as input to the EU risk assessment	Smith et al. (2005)
		179-181 °Cα-HBCD 170-172 °Cβ-HBCD 207-209 °C γ-HBCD	Smith et al. (2005)

TABLE 1. SUMMARY OF PHYSICAL AND CHEMICAL PROPERTIES

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VII, 7.3	Boiling temperature	Decomposes at >190°C	Peled et al. (1995)
VII, 7.5	Gas pressure	6.3 x 10 ⁻⁵ Па (21°С)	Stenzel and Nixon (1997)
VII, 7.7	Solubility	48.8±1.9 μg/lα-HBCD 14.7±0.5 μg/lβ-HBCD 2.1±0.2μg/l γ-HBCD 65.6 μg/l (Technical product HBCD, sum of the above)	Mac Gregor and Nixon (2004)
VII, 7.8	n-octanol/water partition coefficient (log value)	5.07±0.09 α-HBCD 5.12±0.09 β-HBCD 5.47±0.10 γ-HBCD 5.625 (technical product)	Mac Gregor and Nixon (1997) Hayward et al. (2006)

HBCD is predominantly formed by the addition of bromine to trans, trans, cis-1,5,9cyclododecatriene, a cyclic trimer of butadiene. The hexabromocyclododecane produced by this reaction consists of a mixture of 1,2,5,6,9,10 hexabromocyclododecane stereoisomers ranging from oils to an isomer with a melting point of 205-208 °C. Commercially available hexabromocyclododecane 180 °C (185–195 °C). (Ullmann's Encyclopedia of Industrial Chemistry. 6th ed. Vol. 1: Federal Republic of Germany: Wiley-VCHVerlagGmbH&Co. 2003 to present, p. V5 625 (2003)).

CHARACTERISTICS OF TRANSFORMATION IN THE ENVIRONMENT

DEGRADATION

Based on the estimated half-life of the reaction with OH radicals of 3.2 days using AOP v1.91 (24 hours per day; 5*10-3 OH cm), indirect photochemical decay in the atmosphere is believed to occur slowly. Using the same model but with different variables, Wania (2003) calculated a photochemical degradation half-life of 51.2 hours.

HBCD was also observed to degrade during abiotic control biodegradation experiments.

Due to the extremely low solubility of HBCD in water, hydrolysis is not considered a significant source of environmental degradation.

Biodegradation. There are two large standard studies modeling HBCD degradation for sediments and soils (Davisetal., 2003a, b and Davisetal., 2004).

(Davisetal. 2004) found no evidence of aerobic soil degradation. Davisetal. (2003b) reported a half-life of 119 days; however, this number may be too low since only the disappearance of HBCD was assessed. Unlike Davisetal. (2004), noticeably faster extinction has been reported in sediment studies conducted by Davisetal. (2003a). Based on the results of Davisetal. (2004), the half-life of aerobic sediments at 12 °C was 214 days (α -HBCD), 129 days (β -HBCD) and 197 days (γ -HBCD), and for anaerobic sediments it was 210 days. 80 d and 125 d respectively. According to Davisetal. (2004), Stepwise reductive dehalogenation of HBCD through tetrabromocyclododecene and dibromocyclododecadiene to 1,5,9-cyclododecatriene (CDT) occurs in both aerobic and anaerobic sediments. Since no CO2 or other volatiles were produced

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during the study, there was no indication that 1,5,9-cyclododecatriene was undergoing further transformation. Modeling and testing experiments for HBCD degradation did not reveal any mineralization, although primary degradation and even mineralization were observed in two credible biodegradation tests using CDT. This may be explained by the short testing period for HBCD, which may not have been sufficient to detect any mineralization even under ideal conditions when CDT was produced in significant quantities from HBCD. Moreover, significant degradation of HBCD to CDT was observed only under anaerobic conditions, although it is likely that aerobic conditions are required for further degradation of CDT. As a result, it is not possible to determine the overall environmental degradation potential of CDT from HBCD degradation data and vice versa. (ECNA, 2008)

In addition to the experimental results, the sediment core samples examined indicate that HBCD degrades more slowly in sediments than predicted by modeling studies. Moreover, many time series show that HBCD concentrations in biota are increasing, and this has been detected in biotic and abiotic samples from even the most distant locations. (ECNA, 2008)

It is concluded that HBCD meets the criteria for persistence in soil and sediments, although disappearance or degradation was observed in several experimental conditions.



Figure 2. Stepwise dehalogenation of HBCD (Davisetal., 2004)

DISTRIBUTION IN THE ENVIRONMENT

There are no experimental data on adsorption. According to EURAR, 2008, the log partition coefficient of organic carbon (logKoc) is 4.66, indicating an extremely high adsorption potential. It is reasonable to assume that HBCD has relatively little mobility in soil and sediment. According to the observed vapor pressure, HBCD is very slightly volatile (6.3x10-5 Pa at 20 °C). The Henry's law constant at 20–25 °C is 0.75 Pa mol, based on the sum of the solubilities of the individual diastereomers ($66 \mu g/L$). Thus, HBCD has a low likelihood of evaporating from water surfaces. Vaporization of HBCD appears to be a less significant distribution route due to its low volatility and high particulate adsorption capacity.

PROPAGATION MODELING

According to the EUSES modeling used to assess the risk of HBCD in the EU (ECHA, 2008), the steady state mass fractions of HBCD at regional level are as follows:

Fresh water 0.003% Sea water 0.0003%

Air 0.00003%

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Agricultural soil 45%

Industrial soil 0.015%

Freshwater sediment. 0.02%

Sea water sediment 0.0003%

Assuming equal releases to air, water and soil, the Tier IIIEpiwin 3.20 fugacity model gives the following distribution of HBCD:

Air 0.03%

Water 8.1%

Soil 83%

Sediment 9.1%

HBCD degrades very slowly in the atmosphere (half-life > 2 days), suggesting the possibility of long-range atmospheric transport in the vapor phase. Despite this, most long-range transport of HBCD into the environment is predicted to occur in aerosol form due to its low volatility and strong adsorption potential (Wania, 2003). The scientists say that among the compounds they studied, HBCD exhibits one of the strongest long-range transport abilities. However, it should be emphasized that for HBCD the significance of the correlation between distance and concentration was quite low (r2 = 0.45; p = 0.33), and the standard errors of the estimates were quite high, possibly due to the small number of sites included (four sites were used). as a basis for regression). However, when the results for HBCD are considered in conjunction with the results for the other organohalogen chemicals studied, the results for Uenoetal. (2006) can be taken as evidence of the powerful long-range transport capacity of HBCD.

BIOACCUMULATION

Aquatic bioaccumulation. The technical product has a measured logarithmic octanol-water partition coefficient (logKow) of 5.625. Another study (Hayward et al. 2006) predicted logKow to be 5.07 for α -, 5.12 for β -, and 5.47 for γ -HBCD for each unique diastereomer. Given these logKow values, BCFwin (v 2.17) predicts bioconcentration factor (BCF) values of 4240, 1600, 1750, and 3250 for the technical product and the α -, β -, and γ diastereomers, respectively.

Terrestrial bioaccumulation. There are no studies of BCF in earthworms. However, HBCD content in earthworms was measured in an earthworm survival and reproduction study (Aufderheide et al., 2003). The study found bioaccumulation factors based on wet mass concentrations of worms and soil to range from 0.03 to 0.08. In worm tissue, the α -HBCD fraction constitutes about 60% of the total HBCD concentration, while the α -diastereomer constitutes approximately 6% of the total concentration in soil. For α -HBCD (0.3–0.8), the diastereomeric specific bioconcentration coefficient is more than an order of magnitude higher than for γ -HBCD (0.005–0.02). This is consistent with what has been observed in other biotas, such as mammals and fish, where α -HBCD is the dominant diastereomer. It is unknown why this difference exists. This may be due, for example, to greater absorption of the α -diastereomer or differences in metabolism between diastereomers.

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Other. Based on concentration measurements carried out in European surface waters and freshwater fish collected in EURAR (2008), HBCD accumulates in fish in the wild. Recent HBCD values in filtered water samples from European surface waters (n = 14) range from 0.016 (or below the detection limit) to 1.5 µg/L (point source-receiver, Skern River). Another important thing a study was published by Law in 2006: it measured HBCD in the fat of 85 porpoises that stranded or died in the United Kingdom between 1994 and 2003. The average concentration increased from 100 g kg-1 live weight in the mid-1990s to 9400 g kg-1 live weight in 2003. The increase was particularly noticeable between 2000 and 2003. In addition, abiotic samples do not typically include the dominant species -HBCD. The predominance of α -HBCD in biota may be due to many reasons. Firstly, given its higher water solubility and lower logKow value, mass transfer limitations are the lowest for α-HBCD among the three diastereomers. Thanks to these characteristics, it is more easily absorbed from the gastrointestinal tract and the environment. Based on in vitro experiments in mammals and fish, a-HBCD is the least metabolizable (Zegersetal., 2005; Janáketal., 2005b). In microsomal preparations of the liver of an ordinary Janáketal smear. (2005b) found that α -HBCD was the least biotransformable of the three major diastereomers studied. Modeling in the Davisetal article. (2004) degradation also suggests that of the three diastereomers, α -HBCD will degrade the slowest. Moreover, it has been noted that fish are able to bioisomerize γ -HBCD and β -HBCD into α -HBCD (Lawetal., 2006a).

Human exposure to HBCD through food is an important route of exposure because it is a persistent and bioaccumulative chemical released from both point and diffuse sources. EURAR determined a maximum intake of 22 ng HBCD/kg/day based on studies of food samples purchased in Swedish grocery stores and including fish, meat, chicken, milk and eggs. The average value was 10 times lower (European Commission, 2008).

In the risk assessment, a bioconcentration factor value of 18,100 was chosen as a representative value (European Commission, 2008). In addition, a significant collection of field measurements of biota indicates that HBCD is biomagnified. HBCD concentrations have been increasing at several times, including among birds and marine mammals. There are no bioconcentration factors specific for diastereomers. Although α -HBCD is present in commercial HBCD at its lowest concentration, it usually has the highest concentration of the three major diastereomers in biota. However, there may be several reasons for the differences in diastereomer distribution between biota and industrial products. Conclusion: HBCD has a very high bioaccumulation potential.

HAZARD ASSESSMENT TO HUMAN HEALTH

TOXICOKINETICS

There are limited data available on the toxicokinetics of HBCD. The largest amounts of properly dissolved HBCD are likely to be found in adipose tissue and muscle, followed by the liver and, in much smaller quantities, the lungs, kidneys, blood and brain of rodents. Although the exact percentage of oral absorption is unknown, it is most likely between 50% and 100%. However, 100% oral absorption is assumed to calculate DNEL. Although women reach higher levels than men, the chemical accumulates in both sexes. Accumulation of α -diastereomer is significantly higher than the accumulation of the other two HBCD diastereomers in the technical product,

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especially at higher exposure levels. It looks like it will take months to reach a steady state. Although the full extent of technical metabolism of HBCD is unknown, exposure to -HBCD resulted in the detection of three polar metabolites, as well as non-extractable compounds in feces and urine. The stepwise reductive debromination of HBCD via tetrabromocyclododecene and dibromocyclododecadiene to 1,5,9-cyclododecatriene, which appears to be the end product of degradation in environmental samples, was identified as the only biodegradation route in environmental biodegradation studies to date. (ECNA, 2008).

TOXICITY

ECHA has reported the following acute toxicity data:

The minimum lethal dose by oral exposure in rats exceeds 20 g/kg.

The minimum lethal dose by inhalation in rats exceeds 200 mg/l.

The minimum lethal dose for dermal exposure in rabbits exceeds 20 g/kg. (ECNA, 2008)

Their conclusion: Currently available acute toxicity data indicate very low acute toxicity and do not support classification of HBCD according to EU standards. (ECNA, 2008)

In addition, the substance does not cause eye irritation or corrosive effects on the skin. (ECNA, 2008)

Available data on repeated dose toxicity and carcinogenicity do not suggest classification of HBCD according to EU criteria.

HBCD was negative in the in vitro and in vivo micronucleus aberration tests and did not cause mutations in the Ames test. Therefore, it can be said that HBCD has negligible genotoxic potential both in vitro and in vivo. Currently available mutagenicity data do not support the EU classification of HBCD. (ECNA, 2008)

ECOLOGICAL HAZARD ASSESSMENT

ECHA has proposed the following environmental classification: N; R50-53: Very toxic to aquatic life may cause long-term adverse effects in the aquatic environment.

As stated in the proposal for maximum concentration limits for highly hazardous compounds (ECBI/65/99 Add.10), the stated L (E) C50 range of 10-100 μ g/L would result in the following drug concentration limits:

Substance concentration limits Drug classification

 $C \ge 2.5\%$ N; R50-53

 $C \ge 0.25\%$ N; P51-53

C ≥0.025% R52-53

The proposal is based on the extremely high bioconcentration factor (18,100), the bioconcentration factor found in the fish study, the toxic effects observed in the 72-hour study of the seaweed Skeletonemacostatum (EC50 52 μ g/l), and the lack of biodegradation shown in the standard test . The results of the 21-day Daphnia magna life cycle test, in which the lowest

observed effect concentration was estimated at 5.6 μ g/L based on lower mean lengths, support the proposed classification. (ECNA, 2008)

- Likewise, the U.S. Environmental Protection Agency (EPA) has also concluded that HBCD may cause adverse effects on a variety of organisms, including algae, fish, invertebrates, and soil organisms at environmentally significant concentrations. "HBCD is toxic to algae and extremely toxic to fish embryos (Desjardins, et al., 2004; Deng, et al., 2009)." ("Hexabromocyclododecane (HBCD) Action Plan - US Environmental Protection Agency") The US EPA report presented the following results to support its conclusion:
- Fish have also been found to experience a number of sub-lethal effects such as changes in thyroid status, protein metabolism, oxidative stress and reproductive activity (Palace, et al., 2008; KlingandFörlin, 2009; Zhang, etal., 2008; KlingandFörlin, 2009; Zhang, et al., 2008; Ronish et al., 2004).
- 3) According to Drottar and Kruger (1998), the first and second generations of Daphnia descendants turned out to be smaller and smaller in number.
- 4) Tadpoles (Xenopuslaevis) treated with HBCD showed developmental effects dependent on thyroid hormones (Schriks, et al., 2006).
- 5) HBCD has been noted to reduce the biomass and egg production of soil organisms (Lumbriculus variegatus) (Oetken, et al., 2001).
- 6) When HBCD was administered to chicken embryonic hepatocytes (Gallus domesticus) in vitro, the expression of genes (mRNAs) related to thyroid and liver function was significantly altered (Crump, et al., 2008).
- 7) Thinner eggshells have been measured in American kestrels exposed to a combination of PBDEs and HBCD (Fernie, et al., 2009).

8) ASSESSMENT BY THE CRITERIA "PERSISTENT, BIOACUMULATIVE, TOXIC"

The European Chemicals Agency (ECHA), after comparison with Annex XIII criteria, concluded that hexabromocyclododecane (HBCD) meets both the "Bioaccumulative" and "Very Bioaccumulative" criteria, based on experimental data (bioconcentration factor = 18100) and biota measurement data . At a concentration without an observable effect of $3.1 \,\mu$ g/l for daphnia, the "Toxic" criterion is also met. The "persistent" criterion in the soil is met, as soil degradation modeling estimates the half-life of HBCD in aerobic soil to be >120 days. Additionally, phosphorus requirements for sediments are supported by dated sediment cores and sediment degradation modeling experiments, both of which show slow rates of HBCD degradation. The fact that HBCD is consistently detected in abiotic samples and biota from distant locations further supports the idea that this substance is persistent in the environment and is transported over long distances. According to the European Chemicals Agency (ECHA), HBCD is "persistent, bioaccumulative, toxic"; this conclusion is also supported by data from the US Environmental Protection Agency (EPA, 2010) and Swedish monitoring studies (Sellstrometal. 1998).

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ALTERNATIVE PRODUCTS

There appears to be no flame retardant that is equally well suited for the major applications of HBCD, especially in expanded polystyrene (EPS) and extruded polystyrene (XPS). The main advantage of HBCD is that it provides exceptional performance to polystyrene foam as it is effective at low concentrations (EURAR, 2008). For use as building insulation, there are commercially available EPS/XPS equivalents that are technically feasible in many, but not necessarily all, situations. The fact that mineral wool is now widely used and accounts for at least 30% of the building insulation industry in Europe suggests that it is both a technically and financially feasible alternative to EPS/XPS in many applications. However, in some cases the lower mass density of EPS/XPS or superior moisture resistance can be critical. Another product available in the market that meets fire safety standards is phenolic foam. It is low density, moisture resistant and very thermally efficient. It accounts for only a small part of the European insulation market, which is mainly used in situations where moisture resistance or thickness is critical. This may indicate that it is relatively expensive. Although the chemicals used to make phenolic foam are hazardous to human health, these alternative insulation materials are not particularly harmful to the environment or human health (ECHA/2008/02/SR4/ECA.226). There are commercially available substitutes for HBCD in impact resistant polystyrene and textile coatings that provide the same level of fire protection and functionality. Such substitutes include non-halogenated flame retardants. (ECHA/2008/02/SR4/ECA.226)

CONCLUSION

The compound 1,2,5,6,9,10-hexabromocyclododecane (HBCD, C12H18Br6) is primarily used as a flame retardant and is an odorless white solid. In addition, it is used in expandable, extruded, impact-resistant polystyrenes and polymer dispersions for textiles. The synthesis of technical HBCD (t-HBCD) is carried out by adding bromine to 1,5,9-cyclododecatriene. T-HBCD is mainly composed of three diastereomers: α , β and γ , with the latter accounting for more than 70%. It has been established that in reaction with OH radicals, HBCD decomposes within a few days, but it is believed that indirect photochemical decomposition in the atmosphere occurs slowly. Moreover, hydrolysis is not considered a major source of environmental degradation because HBCD has extremely low solubility in water. Following an environmental risk assessment, the European Chemicals Agency concluded in a report in 2008 that this substance meets all the criteria to be considered "Persistent, Bioaccumulative, Toxic", which is also supported by American and Swedish colleagues. In addition to its persistence, bioaccumulation and toxicity, HBCD has a strong ability to be transferred to long distances because it decomposes very slowly in the atmosphere. With regard to the danger to human health, the substance has very low acute toxicity and is not carcinogenic. However, HBCD is highly toxic to aquatic organisms and may have long-term negative effects on the aquatic ecosystem. There are several alternative products available to replace HBCD, namely mineral wool and phenolic foam, but these products do not work equally well as a fire retardant in major applications such as EPS and XPS. However, non-halogenated flame retardants are commercially available substitutes and provide equivalent functionality and fire safety in impact resistant polystyrene and textile coatings.

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