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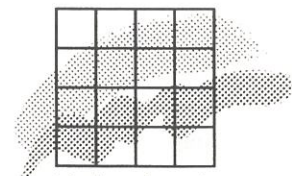
Bulk Deposition of Hexachloro- cyclohexane

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Data sheet

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Abstract

Bulk deposition of gamma-HCH (Lindane) and alpha-HCH has been measured monthly at two locations in Western Jutland, Denmark. Bulk deposition samplers collect wet deposition and some dry deposition. Each sampler consisted of a glass funnel mounted on a glass bottle. At the laboratory the samples were extracted with dichloromethane, cleaned up on a Florisil column and alpha- and gamma-HCH were quantified using capillary GC with EC detection.

Results from 1990 are presented. The total deposition for the two stations shows similarity. An estimate of a one-year- deposition to the area is approximately 2-3 $\mu\text{g}/(\text{m}^2 \text{ year})$ of alpha-HCH and 15-17 $\mu\text{g}/(\text{m}^2 \text{ year})$ of gamma-HCH. The deposition to the North Sea is estimated about 10 tons HCH (alpha+gamma) per year.

The two stations roughly follows same pattern of HCH deposition, indicating the absence of a pronounced local source. The concentrations and deposition of alpha-HCH varies less than that of gamma-HCH, the latter being between a factor 1 and 20 higher than alpha-HCH. Large values of gamma-HCH are seen during spring and early summer probably caused by use of Lindane and by long range transport.

1 Introduction

HCH

Hexachlorocyclohexane (HCH) is a persistent chlorinated organic compound, which may undergo atmospheric long range transport before wet and dry deposition. In ecosystems it may accumulate and cause biological effects.

HCH isomers

HCH exists as several isomers each with different orientation of the chlorine atoms. The two most important isomers, the alpha- and gamma-forms (Lindane) are shown in Figure 1

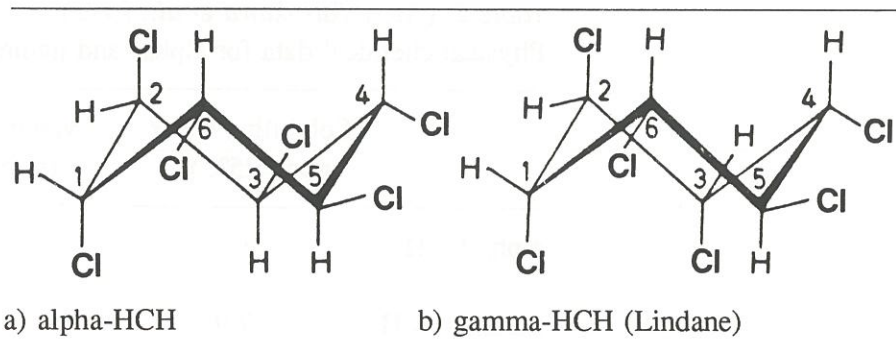


Figure 1. Structure and orientation of atoms in alpha- and gamma-HCH.

1.1 Use of Lindane

Use of Lindane

Lindane is still used in Denmark as an insecticide, but in quite small amounts. From 1976 to 1990 between 5 and 15 tons per year were traded. Lindane is mainly used for seed coating, but is also used in forests, for gardening, for preservation of timber, and in minor amounts against e.g. ants and fleas. In Table 1 the yearly sales from 1976 to 1990 is shown (Bendixen, pers. com.).

Table 1. Sale of Lindane in Denmark in tons per year (Bendixen, pers. com.).

year	1976	1977	1978	1979	1980	1981	1982	1983
tons	11.6	9.1	11.4	11.3	12.4	9.0	8.7	7.3
year	1984	1985	1986	1987	1988	1989	1990	
tons	5.8	8.4	5.2	10.4	8.1	14.6	8.4	

In Western Europe Lindane (~99 % gamma-HCH) is used, but in other countries the technical mixture of HCH is used. This consists of about 82% alpha, 13% gamma and 5% other isomers (Agarwal *et al.*, 1987), gamma-HCH being the active insecticide.

1.2 Physical and chemical properties of Lindane

The vapor pressures of alpha- and gamma-HCH are of same size while the water solubility of gamma-HCH is about 5 times larger than that of alpha-HCH (Tatsukawa *et al.*, 1972) (See Table 2).

Table 2. (After Tatsukawa *et al.*, 1972).

Physical chemical data for alpha- and gamma-HCH.

	Solubility, water (ppm, 25°C)	vapor pressure (mm Hg, 20°C)
alpha-HCH	1.6	0.02
gamma-HCH	7.9	0.03

1.3 Earlier studies

Wash out

Several authors have found a higher gamma/alpha ratio in precipitation compared to air (Atlas & Giam, 1981; Bidleman & Leonard, 1982; Brorström-Lundén, 1991). This may indicate that gamma-HCH is more effectively washed out of the atmosphere compared to alpha-HCH.

gas/particle fraction

In studies with glass fiber filters followed by an adsorbent a minor part of the HCH is generally found on the filter indicating a large part in the gas phase (*e.g.* Brorström-Lundén, 1991; Oehme & Stray, 1982). This effect may however be caused by blow off from the filter to the adsorbent.

Johnson *et al.* 1986 used a diffusion denuder method in sampling HCH from ambient air at a suburban location near Lake Ontario, Canada. This method has the ability to distinguish between gas and particle phase. They found a varying part of the air concentration in the particle and vapor phase. The mean values were 22 % alpha-HCH and 36 % gamma-HCH in the particle-associated fraction. Lindane was predominantly found in the vapor phase near a suspected source.

Concentrations in ambient air

In measurements of air concentrations alpha-HCH is generally found at higher levels than gamma-HCH (Oehme & Stray, 1982; Bidleman et al., 1987; Selenka & Eckrich, 1983; Johnson et al., 1986). Typical HCH concentrations measured in ambient air are roughly found at a level between 0.1 and 10 ng/m³ for both alpha- and gamma-HCH in Europe and North America. Lower levels are found in background areas and much higher levels are found in India (Kaushik et al., 1987).

Concentrations in precipitation

Alpha- and gamma-HCH in precipitation are found in varying ratios with a tendency to highest levels of gamma-HCH in Western Europe (Brorström-Lundén, 1991; Villeneuve & Cattani, 1986) and highest levels of alpha-HCH in American studies (Strachan et al., 1980; Pankow et al., 1984; Strachan & Huneault, 1984; Ligocki et al., 1985) and in background areas (Bidleman & Leonard, 1982; Atlas & Giam, 1981; Pacyna et al., 1986). Typical levels in precipitation are between 1 and 50 ng/L for alpha- and gamma-HCH, much higher levels are found in India (Agarwal et al., 1987).

gamma: alpha ratio

According to Pacyna et al. (1986) the gamma:alpha ratio seems to be useful to distinguish between sources of air pollutants measured in the Arctic. Gamma:alpha ratios ranging from 1:4 to 1:1 were found for the "European" air, 1:10 to 1:7 for North America and 1:10 to less than 1:100 for the former Soviet Union.

Sampling of wet deposition

In sampling of wet deposition two principally different kinds of samplers are used: wet-only samplers, which are only open when it is raining, and bulk samplers which are open all the time. The magnitude of the dry deposition depends on the surface characteristics and a funnel is less effective in sampling dry deposition compared to many natural surfaces. Dry deposition to a bulk sampler outside an emission influenced area is normally small compared to wet deposition.

HCH in precipitation can be sampled in bottles and determined after extraction of the rain water in the laboratory (Brorström-Lundén, 1991; Villeneuve & Cattani, 1986) or after in situ concentration on an adsorbent (Wells & Johnstone, 1978; Pankow et al., 1984; Strachan & Huneault, 1984). A review of sampling methods is made by Egebäck (1991).

Looking at alpha- and gamma-HCH together is a very complex task because:

- the sources can be pure gamma-HCH (Lindane) or the technical mixture of HCH (mainly alpha-HCH).
- photochemical isomerization may occur mainly resulting in conversion of the gamma form to the alpha form (Malaiyandi & Shah, 1984).
- gamma-HCH might be deposited faster than alpha-HCH.
- after deposition HCH might undergo microbial isomerization before reemission.

2 Experimental

2.1 Sampling Stations

Sampling Stations

The sampling stations are placed in forest clearings far from local sources of contamination such as industry and agricultural activity.

The samplers are placed in the terrain partly protected against high winds. This is done to minimize turbulent wind fields around the funnels leading to a under catch of small rain drops.

The location of the sampling stations is shown in Figure 2.

Husby is placed in a fly sand area now covered with heath and low pine plantation 1 km from the coast.

Ulborg is placed in a forest clearing 3 km to nearest farmland and 19 km from the coast.

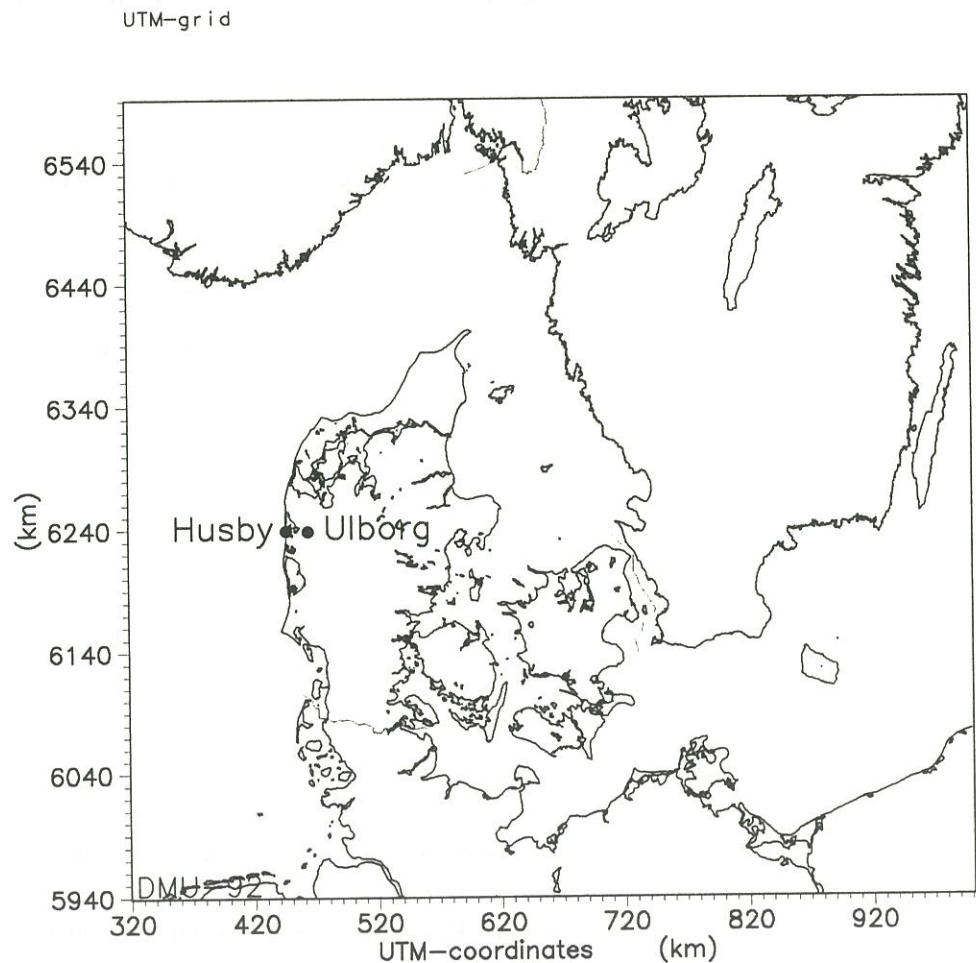


Figure 2. Location of measuring stations: Ulborg and Husby Klit.

2.2 Sampling and analysis

Sampling

Bulk deposition is collected at the two stations.

At each station four samplers consisting of a glass funnel (diameter 20 cm) mounted to a glass bottle (2 liters) were used in a parallel. The edges of the funnels were placed 1.5 m above the ground. The bottles were changed twice a month and the funnels were changed once a month.

In the winter time the bottles were protected from freezing by adding sodium chloride.

Two bottles from the first half of the month and two bottles from the second half were combined, resulting in two replicates per month. The sample volume was typical between 2 and 6 liters (four bottles).

Analysis

At the laboratory the samples were extracted in the bottles, three times with dichloromethane on a magnetic stirrer for 30 minutes. The extracts from the four bottles were combined and concentrated (vacuum) to about 1 mL. Then the samples were added to a Florisil column for clean up. The first fraction were eluted with n-hexane, alpha- and gamma-HCH were eluted in the second fraction by using 1:1 dichloromethane:n-hexane. Dodecane was added as a keeper before the eluates were concentrated (nitrogen) to dryness and soluted in 1 mL of iso-octane. Pentachlorobenzene was used as internal standard (*Poulsen, 1991*).

The quantification of alpha- and gamma-HCH was made with non-linear multi-level calibration on a HP 5890 series II gaschromatograph (GC) with an electron capture detector (*Storr-Hansen, 1991a*). The column was a 60 m x 0.25 mm i.d. fused silica with 0.1 µm 5% cross-linked phenylmethylsilicone (DB-5) and the GC was equipped with a HP 7673 autosampler and a HP Vectra PC QS/20 for data analysis. The identification was checked on a 60 m x 0.25 mm i.d. fused silica column with 0.15 µm 14% cross-linked cyanopropylphenyl (DB-1701). A part of the samples were analyzed on a system with two parallel columns DBS, DB 1701) operated with a glass T-split. (*Storr-Hansen, 1991b*). Chromatograms are shown in Appendix.

The detection limit was below 0.5 ng/L rain water for a 2 liter sample. Blank values (purified water in bottles following the normal procedure) was not detectable. The recovery for the clean up procedure and the GC analysis was 80% ± 8% (s.d., n=5) for alpha-HCH and 80% ± 6% (s.d., n=5) for gamma-HCH; the results were corrected with this figures. Furthermore there was a loss in the extraction step at about 20 % for alpha-HCH while there was no detectable loss of gamma-HCH.

3 Results and discussion

Results from bulk deposition measurements of alpha- and gamma-HCH at the two stations Ulborg and Husby Klit, Western Jutland, Denmark, as monthly figures (1990) are shown in Figures 3 - 7.

Concentrations

Figure 3 shows the concentration of alpha- and gamma-HCH at the Husby and Ulborg station during the year 1990. The two stations follow the same pattern: gamma-HCH gives higher values than alpha-HCH. Especially in spring and early summer a large peak was found for gamma-HCH.

In Figure 4 (below) gamma-HCH is plotted for the two stations together. It is seen that the peak is a little bit higher for the Husby station.

Figure 4 (above) shows the concentration of alpha-HCH in a smaller scale. Even though the concentrations are highest in April and May the concentration during the year is much more constant compared to gamma-HCH.

Gamma: Alpha ratio

The ratio between gamma- and alpha-HCH is plotted in Figure 5. Gamma:alpha ratios between 1:1 and 20:1 are found with highest ratio in spring and early summer.

Precipitation

The amount of precipitation sampled at the two stations is shown in Figure 6. The annual precipitation rate is highest at Ulborg, as Husby is closer to the coast.

Deposition

The deposition of gamma-HCH is shown in Figure 7 (below). A peak in the spring and early summer is caused by high concentrations and a smaller peak in September-October is caused by large amounts of precipitation.

Figure 7 (above) shows the deposition of alpha-HCH. Like the concentrations the deposition is more constant during the year compared to gamma-HCH.

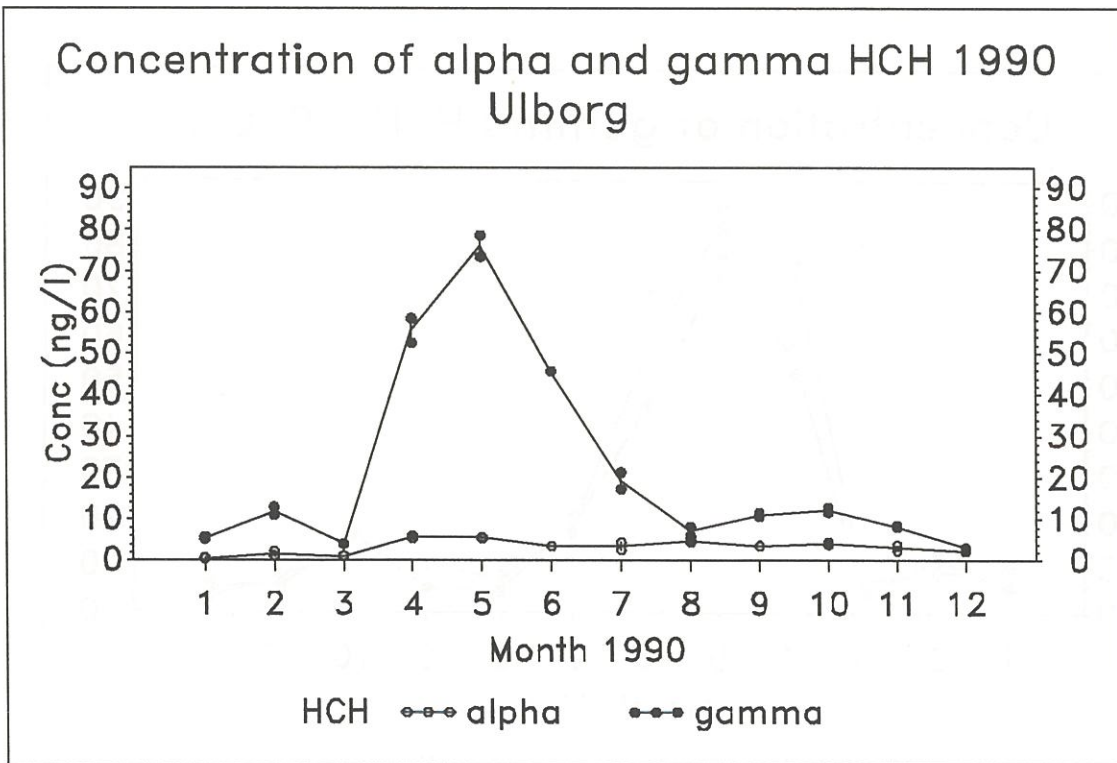
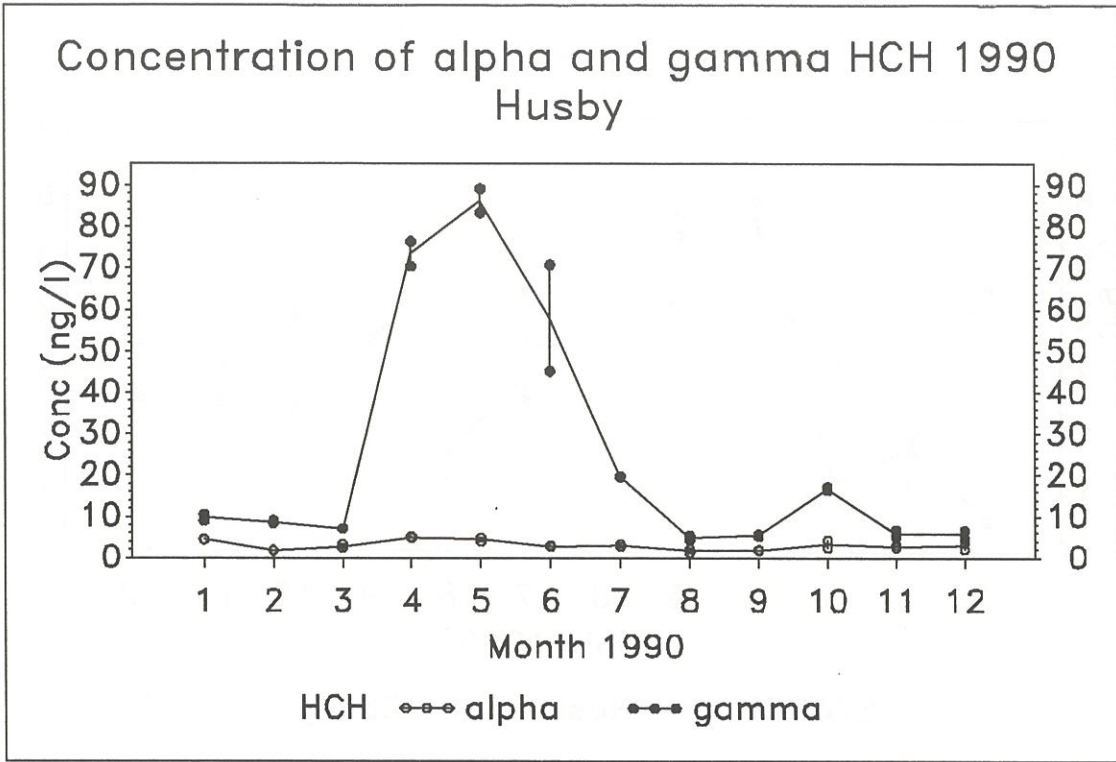


Figure 3. HCH concentrations (ng/L) measured in bulk deposition sampled at Husby (above) and Ulborg (below) 1990.

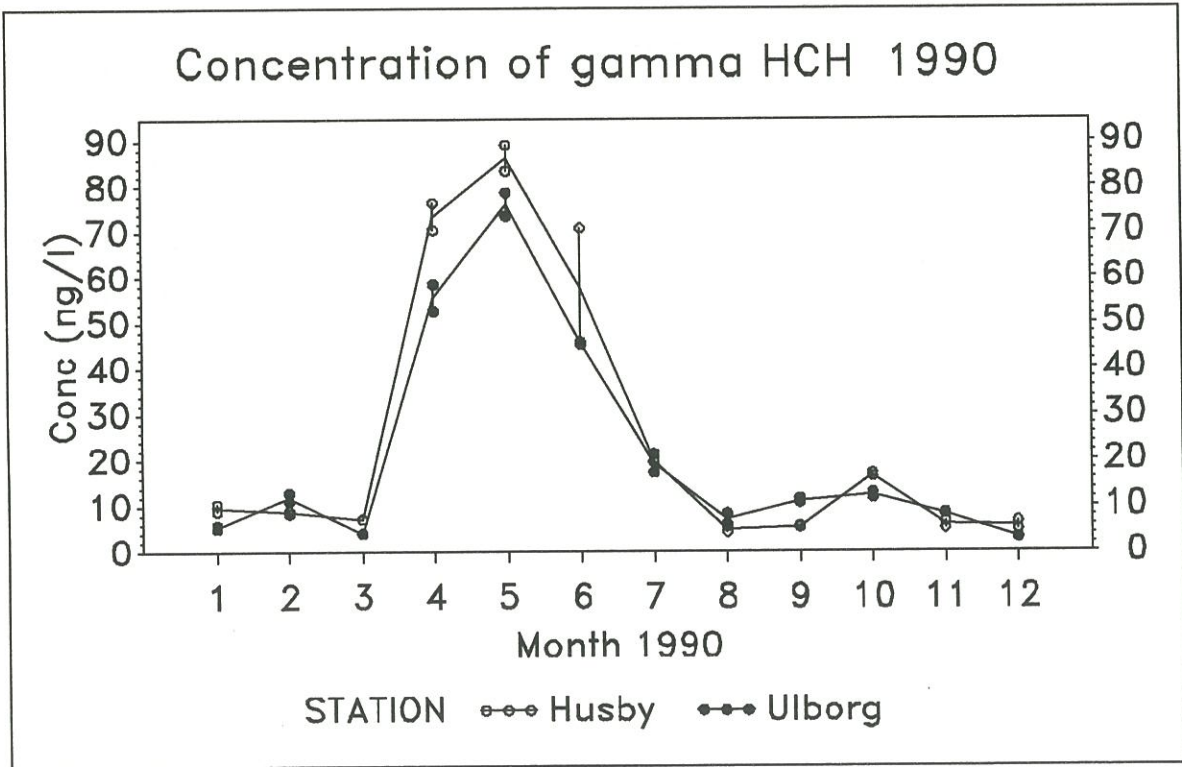
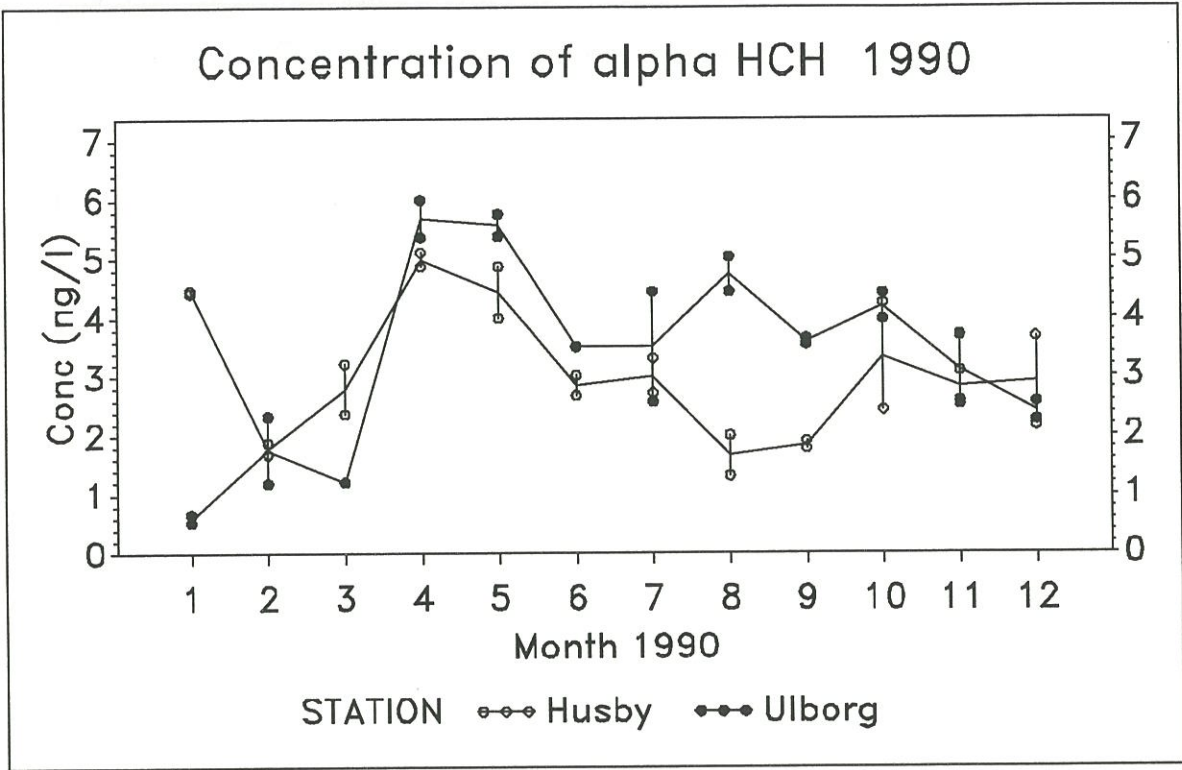


Figure 4. Concentrations of alpha-HCH (above) and gamma-HCH (below) (ng/L) in bulk deposition at Husby and Ulborg 1990.

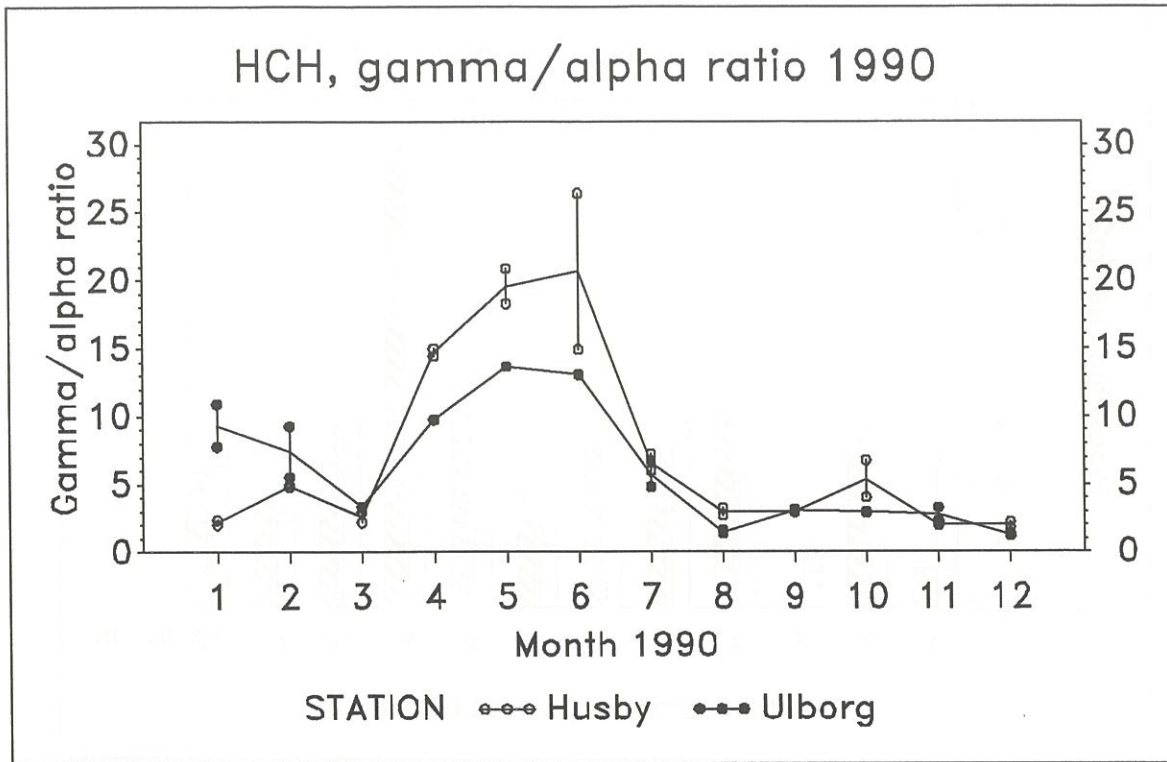


Figure 5. Ratio between gamma- and alpha-HCH found in bulk deposition at Husby and Ulborg 1990.

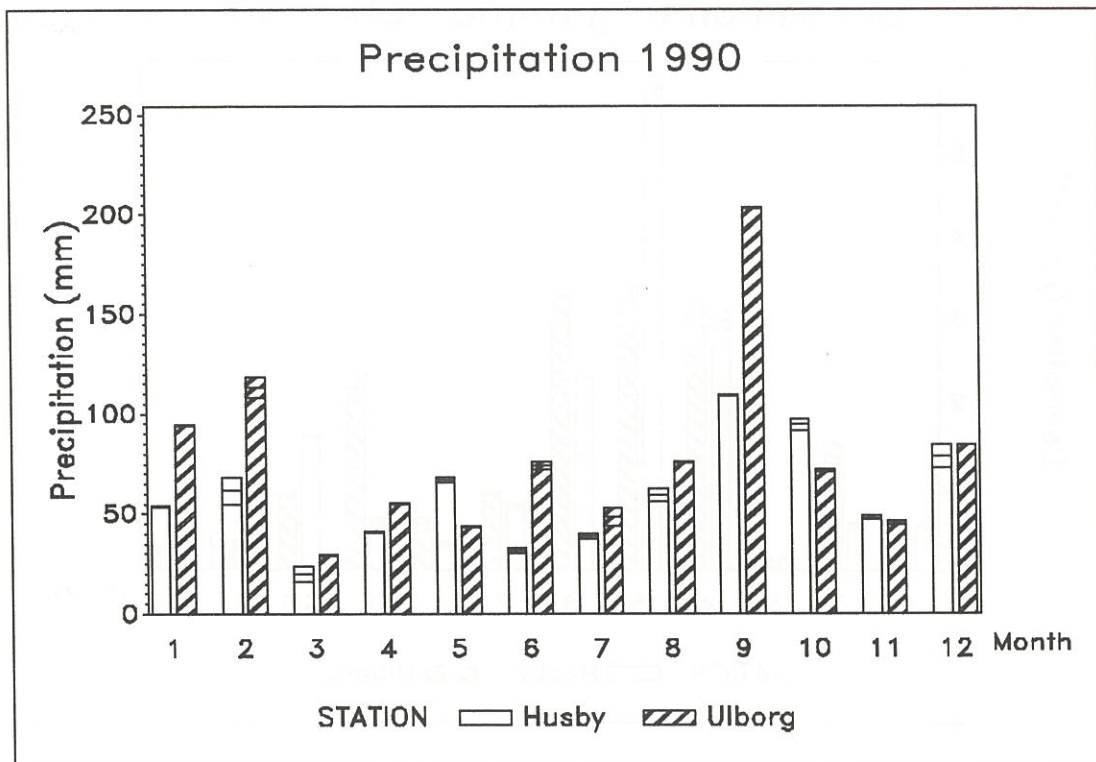


Figure 6. Amounts of precipitation (mm) sampled at Husby and Ulborg 1990. (Minimum, mean and maximum value indicated for each month).

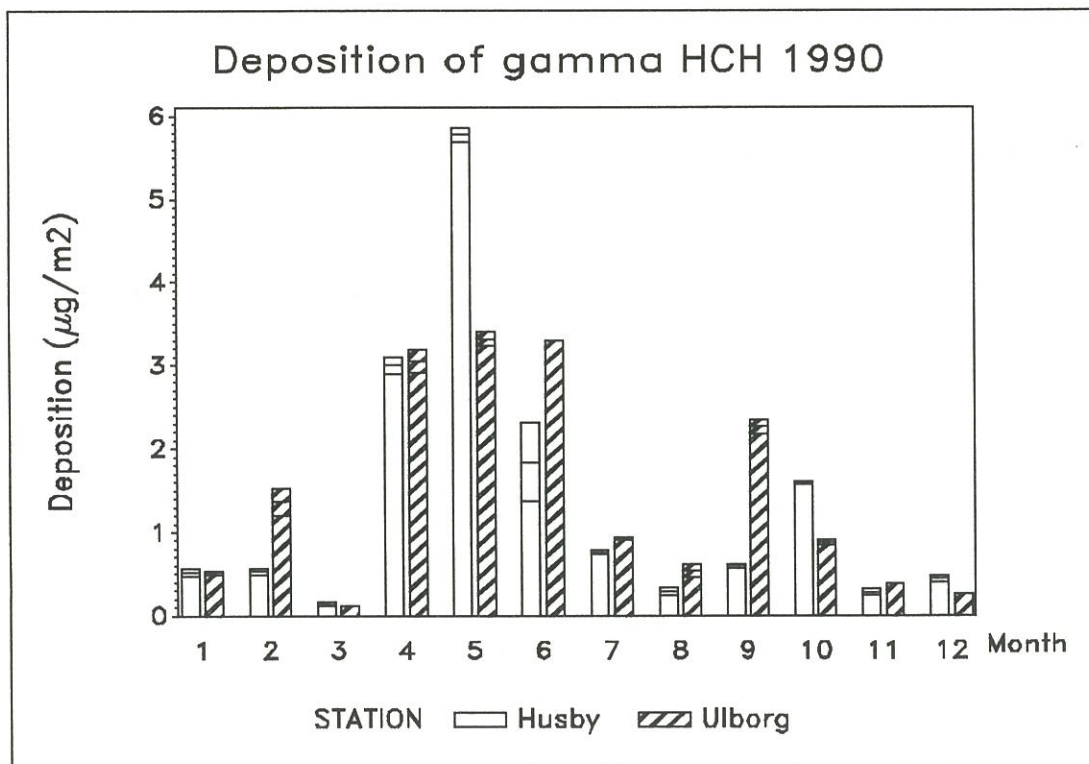
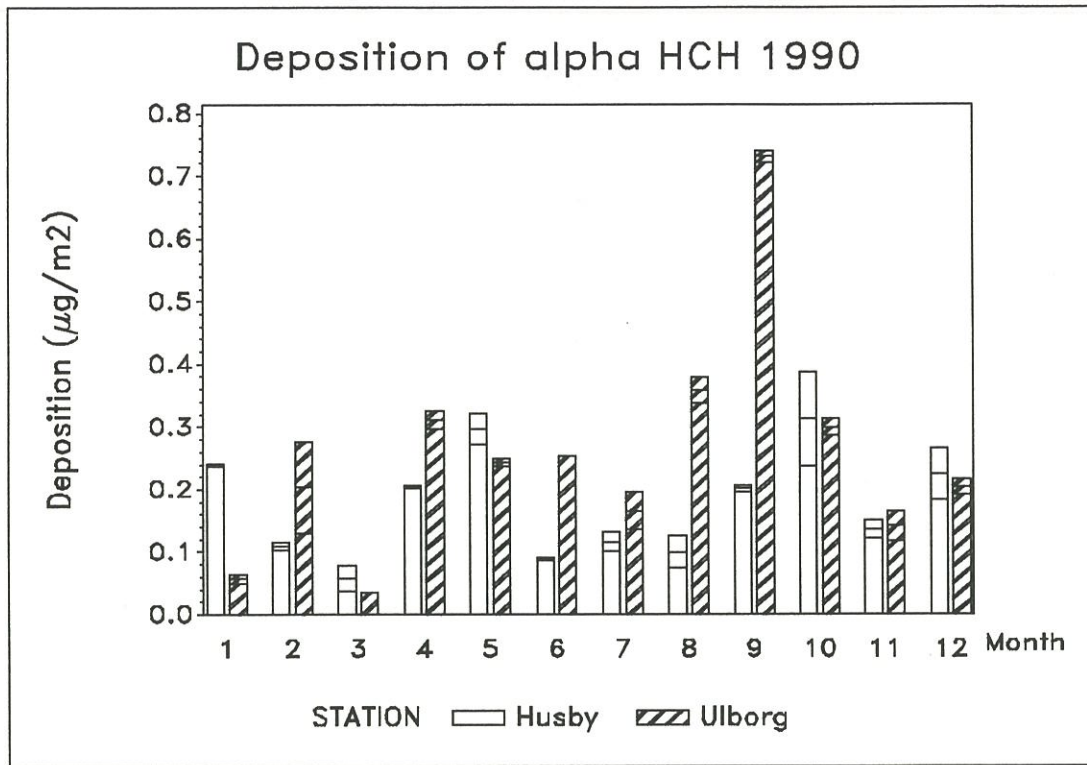


Figure 7. Bulk depositions of alpha-HCH (above) and gamma-HCH (below), $\mu\text{g}/(\text{m}^2 \text{ month})$, at Husby and Ulborg 1990. (Minimum, mean and maximum value indicated for each month).

Gamma-HCH peak

There are several possible explanations for the gamma-HCH peak in spring and summer:

- a local source of Lindane: The equal patterns of the two stations indicate the absence of a pronounced local source.
- reemission of earlier deposited HCH when the weather gets warm in the spring: If this was the case we would probably find the same pattern for alpha-HCH too.
- wind directions varying during the year: This is very difficult to look at with only monthly values. There is a tendency to more eastern wind directions in April and May, but not in June).
- use of Lindane during spring and summer followed by long range transport: This might be a proper explanation.

The maximum concentration is slightly higher at Husby compared to Ulborg. In Husby precipitation is lesser than in Ulborg. Assuming same air concentrations in Husby and Ulborg, the precipitation in Husby might get higher gamma-HCH concentrations caused by wash out of gamma-HCH from the air.

Mean concentration and total deposition

Table 3 shows the precipitation weighted mean concentrations for the year and the total deposition with standard deviations. The alpha-HCH concentrations are alike for the two stations but the gamma-HCH concentration shows slightly higher values for Husby. Ulborg has more precipitation than Husby, which results in a relatively higher deposition.

Table 3. Precipitation weighted mean concentration and total deposition (with standard deviations) for the year 1990.

Total 1990		Husby		Ulborg
Precipitation (mm)		702	± 11	938 ± 7
Concentration (ng/L)	alpha	3.0	± 0.2	3.2 ± 0.1
	gamma	22.5	± 1.1	18.0 ± 0.6
Deposition (µg/m ²)	alpha	2.1	± 0.1	3.0 ± 0.1
	gamma	15.8	± 0.5	16.9 ± 0.4

*Deposition to
Denmark*

The deposition of HCH (alpha + gamma) was found to about 19 $\mu\text{g}/(\text{m}^2 \text{ year})$. Assuming this deposition representative it corresponds to 1.5 tons per year to the area of Denmark (~80.000 km^2 , including sea area). This figure is about 18 % of the amount traded in Denmark in 1990 (8.4 tons). A deposition of about 18 % of the amount used (mainly seed coating) indicates long range transport.

*Deposition to the
North Sea*

From the measurements in this study, bulk deposition of HCH to the North Sea (525.000 km^2) is estimated to 10 tons/year. An earlier estimate by Wells & Johnstone (1978) is 2.1 tons/year. (Aalst et al. 1983) has estimated the dry deposition to between 10 and 100 tons/year, derived from measurements in Delft, Netherlands. These atmospheric inputs should be compared to an estimated river input of 2.5 tons/year (Aalst et al., 1983).

4 Conclusions

- gamma-HCH is found in precipitation at higher levels than alpha-HCH.
- there is observed a peak for gamma-HCH in spring and early summer probably caused by use of Lindane followed by long range transport.
- The two stations Husby and Ulborg follows the same pattern and have almost the same levels. This indicates the absence of a pronounced local source.
- the deposition for the area is measured to 2-3 $\mu\text{g}/(\text{m}^2 \text{ year})$ for alpha-HCH and 15-17 $\mu\text{g}/(\text{m}^2 \text{ year})$ for gamma-HCH.
- The estimated deposition of HCH (alpha + gamma) to the area of Denmark is about 18 % of the amount traded in 1990 (mainly seed coating) indicating long range transport.
- deposition to the North Sea is from the measured deposition estimated to about 10 tons per year.

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Appendix

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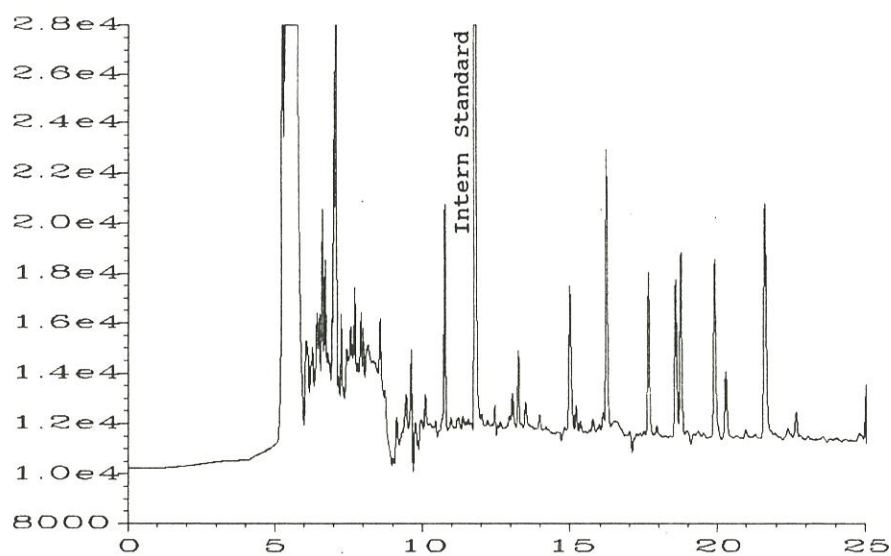


Figure 1. Chromatogram of blank containing internal standard (50 ng/ml), analysed on DB 5 column.

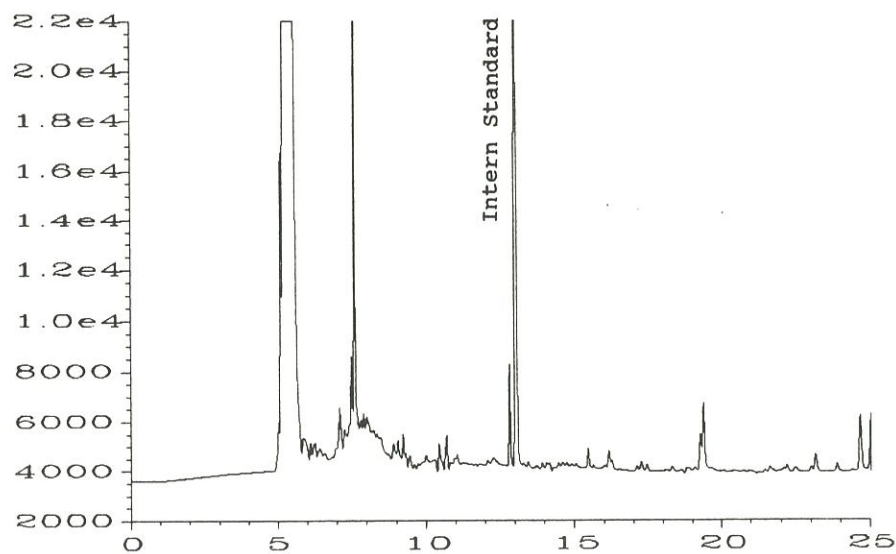


Figure 2. Chromatogram of blank containing internal standard (50 ng/ml), analysed on a DB 1701 column.

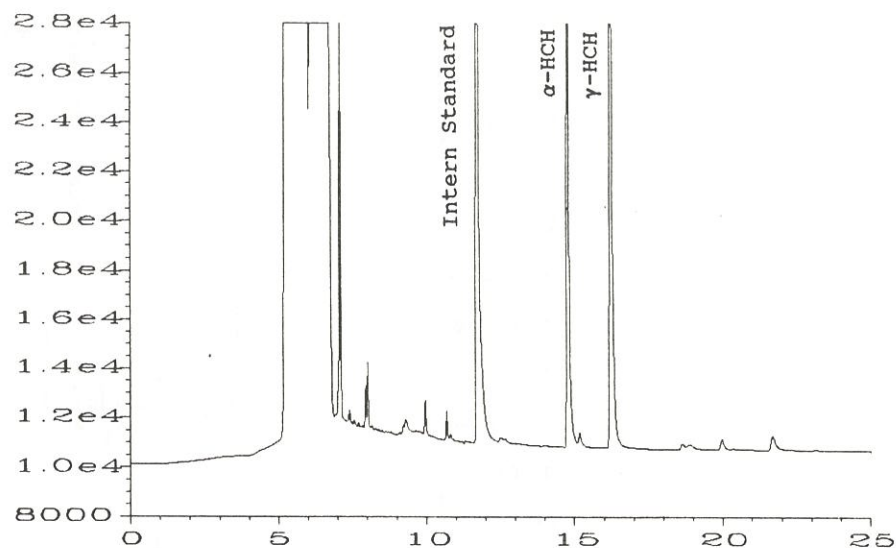


Figure 3. Chromatogram of standard solution containing internal standard (50 ng/ml), alpha-HCH (20 ng/ml) and gamma-HCH (20 ng/ml), analysed on a DB 5 column.

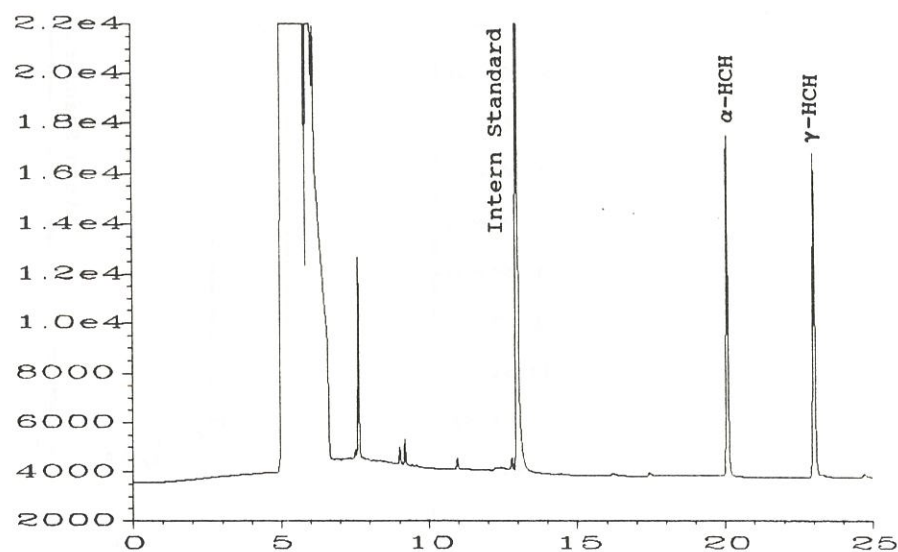


Figure 4. Chromatogram of standard solution containing internal standard (50 ng/ml), alpha-HCH (20 ng/ml) and gamma-HCH (20 ng/ml), analysed on a DB 1701 column.

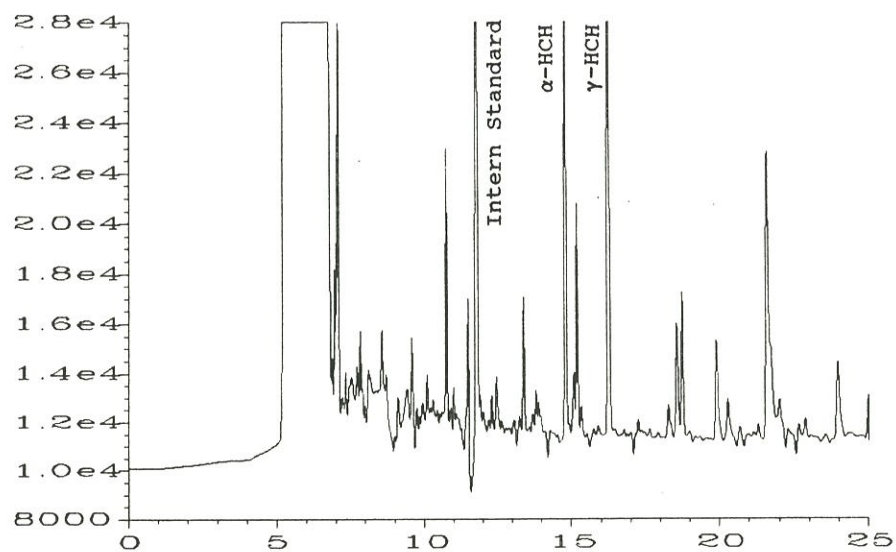


Figure 5. Chromatogram of rainwater sample from Ulborg november 1990, containing internal standard (50 ng/ml), analysed on a DB 5 column.

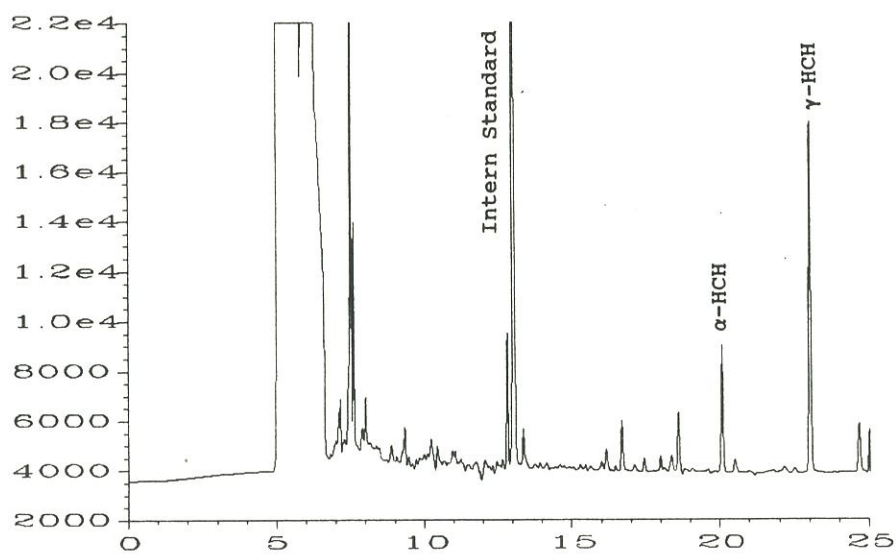


Figure 6. Chromatogram of rainwater sample from Ulborg november 1990, containing internal standard (50 ng/ml), analysed on a DB 1701 column.

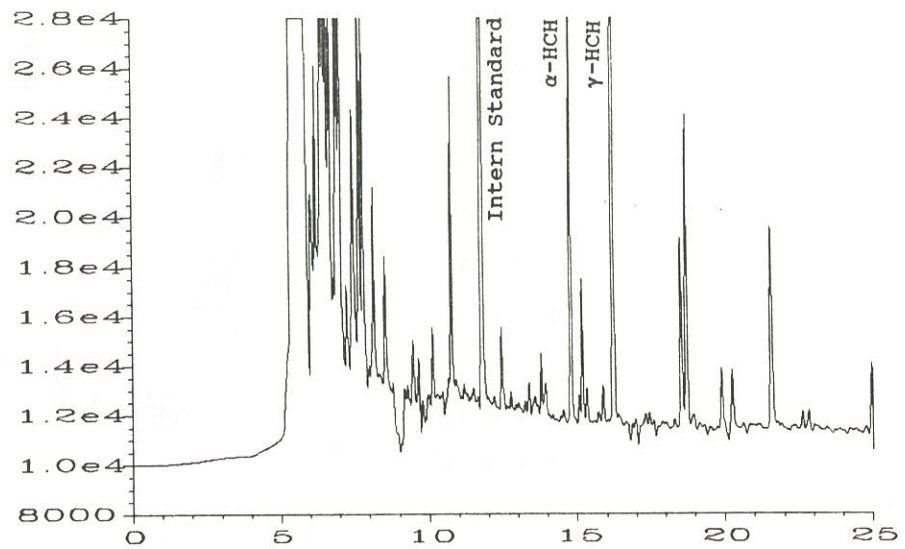


Figure 7. Chromatogram of rainwater sample from Husby december 1990, containing internal standard (50 ng/ml), analysed on a DB 5 column.

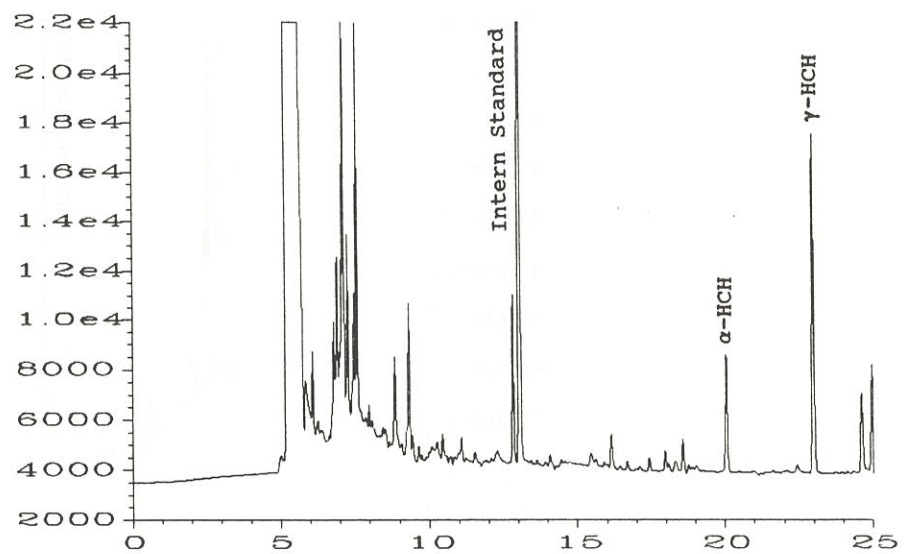


Figure 8. Chromatogram of rainwater sample from Husby december 1990, containing internal standard (50 ng/ml), analysed on a DB 1701 column.

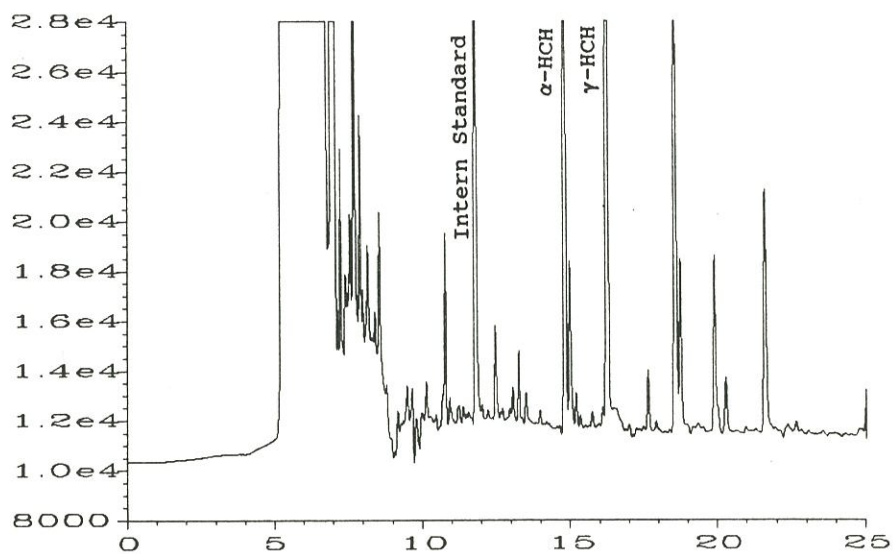


Figure 9. Chromatogram of recovery, containing internal standard (50 ng/ml), analysed on a DB 5 column.

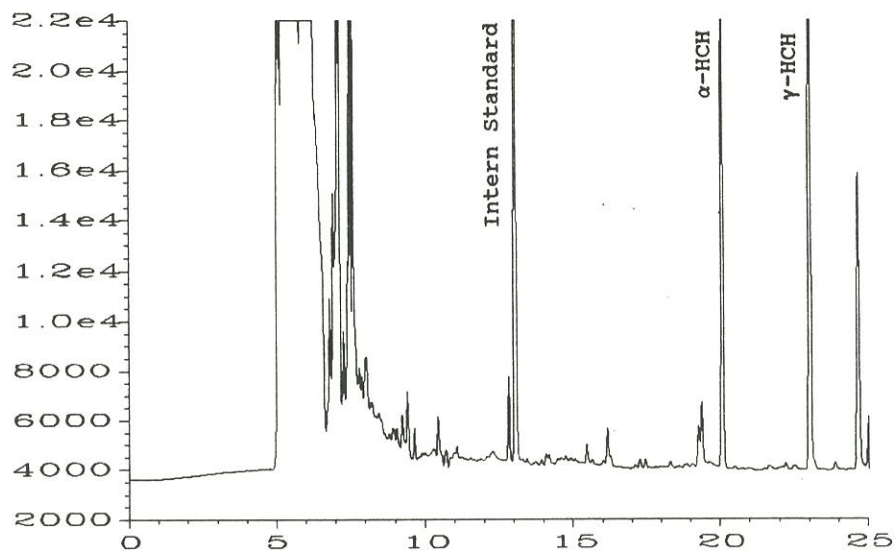


Figure 10. Chromatogram of recovery, containing internal standard (50 ng/ml), analysed on a DB 1701 column.

National Environmental Research Institute

The National Environmental Research Institute - NERI - is a research institute of the Ministry of the Environment. Neri's tasks are primarily to do research, collect data and give advice on problems related to the environment and nature.

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