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Toxaphene and Other Organochlorines in Arctic Ocean Fauna: Evidence for Atmospheric Delivery

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ABSTRACT. Residues of the insecticide toxaphene (polychlorinated camphenes, PCCs) and other organochlorines (OCs) were determined in air, snow, seawater, zooplankton, and benthic amphipods collected from an ice island in the Canadian Arctic. The simultaneous determination of OCs in the atmospheric, hydrologic, and biologic compartments provided evidence of an atmospheric link to polar food chains. PCCs were identified and quantified using capillary gas chromatography — negative ion mass spectrometry. The order of OC abundance in arctic air was: hexachlorocyclohexanes (HCHs) > hexachlorobenzene > PCCs > polychlorinated biphenyls (PCBs) > chlordanes > DDTs. In seawater, PCCs were exceeded only by the HCHs. Concentrations of PCBs and PCCs in two samples of benthic amphipods were the highest of the OCs detected.

Key words: Arctic, Canada, pollution, organochlorines, air, water, biota

RÉSUMÉ. On a déterminé les résidus de l'insecticide toxaphène (camphènes polychlorés) et d'autres organochlorés dans l'air, la neige, l'eau de mer, le zooplancton et les amphipodes benthiques recueillis dans une île de glace de l'Arctique canadien. La détermination simultanée des organochlorés dans les secteurs atmosphérique, hydrologique et biologique, a fourni la preuve d'un lien atmosphérique dans les chaînes alimentaires polaires. On a identifié et quantifié les camphènes polychlorés en utilisant la chromatographie par capillarité en phase gazeuse-spectrométrie de masse à ions négatifs. L'ordre d'importance des organochlorés dans l'air arctique était le suivant: hexachlorocyclohexanes (H.C.H.) > hexachlorobenzène > camphènes polychlorés > biphényles polychlorés (P.C.B.) > chlordanes > D.D.T. Dans l'eau de mer, les camphènes polychlorés n'étaient surpassés que par les H.C.H. Dans deux échantillons d'amphipodes benthiques, les concentrations de P.C.B. et de camphènes polychlorés étaient les plus élevées de tous les organochlorés détectés.

Mots clés: Arctique, Canada, pollution, organochlorés, air, eau, biote

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INTRODUCTION

Contamination of the Arctic by synthetic organochlorine (OC) compounds has been recognized since pesticides and polychlorinated biphenyls (PCBs) were found in birds, mammals, and fish collected in the middle 1960s through the early 1970s (Addison and Smith, 1974; Bowes and Jonkel, 1975; Cade *et al.*, 1968, 1971). Pesticides reported in these studies were DDT, its transformation products DDE and DDD, and dieldrin. Recent investigations of OCs in arctic fauna have expanded the list to include chlorobenzenes and the insecticides hexachlorocyclohexane (HCHs), components of technical chlordane and their metabolites, and toxaphene (polychlorinated camphenes, PCCs) (Andersson *et al.*, 1988; Kawano *et al.*, 1988; Muir *et al.*, 1988, 1989; Norstrom *et al.*, 1988). Surveys of OCs in fish across the Canadian Northwest Territories have shown that PCCs are the most abundant pesticide residue (Muir *et al.*, 1988, 1989; Norstrom *et al.*, 1988). Muir and co-workers (1989) noted that in some locations consumption of fish and their livers by native people could lead to an intake of PCCs that exceeds the U.S. National Academy of Sciences acceptable daily intake of 1.25 $\mu g \cdot kg^{-1} \cdot d^{-1}$.

The widespread distribution of PCCs and other OC pesticides in northern ecosystems points strongly to eolian transport from lower latitudes. Goldberg (1975) coined the term "global distillation" to describe the process by which pesticides are volatilized from temperate and tropical zones and redeposited in colder regions. Several of the above-mentioned OCs have been found in the lower troposphere of the Arctic (Hargrave *et al.*, 1988; Pacyna and Oehme, 1988; Hoff and Chan, 1986; Oehme and Ottar, 1984; Oehme and Stray, 1982; Tanabe and Tatsukawa, 1980). OC pesticides were reported in snow from east-central Ellesmere Island (McNeely and Gummer, 1984) and more recently from large areas of the Keewatin and High Arctic districts of the Northwest Territories (Gregor and Gummer, 1989). Addison and co-workers (1986) noted that over the last decade PCBs have declined more rapidly than total DDT compounds in arctic ringed seal (*Phoca hispada*), compared to two seal populations from eastern Canada (*Halichoerus grypus* and *Pagophilus groenlandicus*). Furthermore, levels of untransformed p, p′-DDT in ringed seal were not significantly different between 1969 and 1981. The authors suggested that DDT input continued for a longer time in the Arctic via air transport from eastern countries.

Toxaphene and similar PCC products are complex mixtures, and their analysis by conventional gas chromatography with electron capture detection (GC-ECD) is difficult. Oehme and Stray (1982) showed mass spectral evidence of PCCs in one air sample from Spitzbergen, but the results were not quantified. In the summers of 1986-87 we collected samples of air, snow, seawater, zooplankton, and benthic amphipods in the Canadian Arctic to investigate OC input to and transfer through polar food chains. Details of the atmospheric study and evidence for PCCs in air are presented elsewhere (Patton *et al.*, 1989). PCCs were the second most

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abundant pesticide in air (after the HCHs), ranking ahead of chlordanes, dieldrin, and DDT compounds. Here we report that PCCs are also major OCs in snow and seawater and are bioaccumulated by zooplankton and amphipods to concentrations similar to PCBs and exceeding those of other pesticides. The simultaneous determination of PCCs in the atmospheric, hydrologic, and biologic compartments provides the first direct evidence for an atmospheric link of PCCs to arctic food chains.

METHODS

Sampling of air, water, zooplankton, and amphipods was carried out from a 4 km × 7 km × 45 m thick tabular ice island drifting in the Arctic Ocean. The Canadian Polar Continental Shelf Project has established a base on the island for oceanographic studies of the Arctic Ocean. It was located off the coast of Axel Heiberg Island at 81°N, 101°W at the time of our study. Air samples were taken at sea level by drawing 1500-3000 m^3 air through glass fiber filter–polyurethane foam traps. Water volumes of 300-550 L were drawn and extracted *in situ* with columns of XAD-2 resin, using a Seastar™ sampler. Zooplankton were collected by vertical net hauls between 150 and 290 m to the surface. Benthic lysianassid amphipods were obtained with traps deployed on the bottom with bait enclosed by Nitex™ mesh to prevent ingestion. Snow representing the annual accumulation was obtained from the ice island, from the Devon Island ice cap (75°30′N, 87°40′W), and from Resolute Bay on Cornwallis Island (74°42′N, 94°45′W).

Samples were extracted with organic solvents, subjected to cleanup and fractionation steps, and analyzed by capillary GC on polydimethylsiloxane bonded-phase columns. OCs were determined using GC-ECD and, for PCCs, negative ion mass spectrometry (GC-NIMS) with multiple ion detection. Details of sampling and analytical methods are given elsewhere (Gregor and Gummer, 1989; Hargrave *et al.*, 1988; Patton *et al.*, 1989).

RESULTS AND DISCUSSION

PCCs in individual air samples have been reported by Patton *et al.* (1989), and mean values are given here in Table 1. Comparison of the PCC congener distributions in air and

TABLE 1. Organochlorines in the Canadian Arctic[a]

	PCCs[b]	HCB	HCHs	CHLOR	NCHLOR[b]	DDE	DDT	PCBs
Air,[c] pg·m^{-3}								
August 1986	44	189	577	3.9	1.9	0.1	<0.9	14
June 1987	36	147	385	6.3	4.4	2.9	2.3	20
Snow, pg·L^{-1}								
May 1986								
Ice Island	307	<2	2340	128	19	<7	<1.6	160
Ice Island	85	<2	1720	51	9	<8	<1.7	86
Devon Island	1700	100	1300	181	81	20	<10	670
Resolute Bay	600	50	9800	69	55	50	100	1800
Seawater, pg·L^{-1}								
May 1986, 10 m	400	<0.5	5590	3	0.5	<0.6	—	<14
May 1986, 225 m	105	11	510	5	0.3	<2	—	<14
August 1986, 10 m	360	28	4300	4	<0.7	<0.3	<1	<8
June 1987, 270 m	32	5	190	3	0.6	<0.4	<3	<2
Zooplankton,[d] ng·g^{-1}								
August 1986								
Dry wt.	12	1.6	20	3.1	1.6	1.2	1.9	6.6
Lipid wt. (49% dry wt.)	24	3.3	41	6.3	3.3	2.4	3.9	13
June 1987								
Dry wt.	39	2.4	6.7	7	5.3	3.2	12	27
Lipid wt. (24.3% dry wt.)	160	10	28	29	22	13	49	110
Amphipods,[e] ng·g^{-1}								
August 1986								
Dry wt.	440	18	47	45	220	59	130	480
Lipid wt. (29.2% dry wt.)	1500	62	160	150	750	200	440	1600
June 1987								
Dry wt.	1730	26	33	130	390	540	180	3000
Lipid wt. (20.8% dry wt.)	8300	120	160	620	1900	2600	860	14000

[a] Abbreviations: PCCs — polychlorinated camphenes; HCB — hexachlorobenzene; HCHs — sum of alpha- and gamma-hexachlorocyclohexanes; CHLOR — sum of cis- and trans-chlordane; NCHLOR — sum of cis- and trans-nonachlor; DDE and DDT — p,p′-DDE and p,p′-DDT; PCBs — polychlorinated biphenyls as Aroclor 1254 (water, zooplankton, amphipods), Aroclors 1242 + 1254 + 1260 (snow) or Aroclors 1242 + 1254 (air).
[b] PCCs and NCHLOR determined by GC-NIMS, other OC by GC-ECD.
[c] Mean of 7 samples in 1986 and 6 samples in 1987 (Patton *et al.*, 1989).
[d] Plankton composed of *Calanus hyperboreus*, *Metridia langa*, and *Xanthocalanus borealis*.
[e] Lysianassid amphipods *Anonyx sarsi* from 190 m in August 1986 and *Tmetonyx cicada* from 310 m in June 1987.

in a technical toxaphene standard supplied by the U.S. Environmental Protection Agency showed that the most volatile constituents were preferentially transported to the Arctic. PCC concentrations in air exceeded those of PCBs, components of technical chlordane, and DDT compounds (Table 1).

Reconstructed ion chromatograms of PCCs in air from the Ice Island and in snow from Devon Island show that the heavier congeners were enriched in snow (Fig. 1). This might arise from enhanced deposition of the higher molecular weight PCCs from the atmosphere or revolatilization of the lighter PCCs. PCCs in water, zooplankton, and amphipods are shown in Figures 2 and 3. After the HCHs, PCCs were the most abundant OC pesticide in surface (10 m) and deep (270 m) water samples. Levels of PCCs in zooplankton and amphipods were comparable to those of PCBs (Table 1). PCCs in one amphipod sample were sufficiently high that full-scan spectra of some Cl_8 and Cl_9 bornanes, major components of toxaphene (Swackhamer *et al.*, 1987), could be obtained (Figs. 2 and 4).

As is the case of other organic compounds, the heavier, less water soluble PCCs should be preferentially accumulated in lipids by aquatic organisms (Mackay, 1982) and more susceptible to vertical transport on sinking particles (Burns *et al.*, 1985; Knap *et al.*, 1986; Tanabe and Tatsukawa, 1983). This trend is shown by the relative proportions (based on total peak area) of Cl_7 : Cl_8 : Cl_9 PCCs in water (1.0 : 0.73 : 0.04), zooplankton (1.0 : 1.7 : 0.30), and amphipods (1.0 : 3.8 : 3.6). The same ratios in technical toxaphene (1.0 : 1.2 : 0.34) are in good agreement with those found by Swackhamer and co-workers (1987).

The Arctic Ocean is characterized by a low-salinity (30-32 parts-per-thousand, ppt) surface layer uniformly mixed to 50 m, which is separated from more saline (34-35 ppt) water with origins in the Atlantic Ocean by a broad pycnocline extending from 50 to 150 m depth (Coachman and Barnes, 1961). The density gradient and vertical stability of the surface layer inhibit vertical exchange of OCs. Concentrations of PCCs and HCHs in the upper mixed layer sample were higher than those at depth (Table 1), indicative of input at the surface. Other mixed layer and deep samples showed the same trend for HCHs, although PCCs were not determined (Hargrave *et al.*, 1988). PCCs and other OCs are probably transported to deeper water on sinking particles, as occurs in other marine areas (Burns *et al.*, 1985; Knap *et al.*, 1986; Tanabe and Tatsukawa, 1983) and through zooplankton vertical migration and feeding (Harding, 1986). Zooplankton produce large fecal pellets rich in contaminants that sink rapidly (Burns *et al.*, 1985).

PCCs may also reach the bottom and be incorporated into the benthic food web through other biological pathways, as is evident from the high concentration found in benthic amphipods (Table 1). These crustaceans, attracted to bait by smell, feed rapidly to satiation and disperse (Sainte-Marie and Hargrave, 1987). Hundreds of individuals, primarily of the two species *Tmetonyx cicada* and *Anonyx sarsi*, were obtained by exposure of baited traps on the bottom under the ice island for several hours. Feeding must normally occur on carcasses of larger organisms, since the amphipod mouthparts are adapted for cutting. The high lipid levels (>20% of dry weight), typical of Arctic Ocean fauna that store energy for long periods of starvation, could lead to the bioaccumulation of OCs.

Atmospheric loading of PCCs and other OCs to the Arctic can occur through a variety of processes, but little is known about their relative importance. From approximately November through May, influx of pollutants from lower latitudes causes the Arctic to be shrouded in a haze blanket. The haze is a mixture of accumulation mode aerosols (0.1–1.0 μm diameter) containing sulfate, ammonium, soot carbon, trace elements, and other anthropogenic substances and larger particles composed mainly of crustal and sea salt elements (Barrie, 1986). Due to a combination of meteorological conditions and source strengths, Europe and Asia contribute the major share of haze material to the Arctic (Rahn, 1981; Barrie, 1986). Adsorption of high molecular weight organic vapors to atmospheric particulate matter is enhanced by low temperatures (Bidleman, 1988). During the winter OCs may attach to haze aerosols, accumulate on land and at the frozen ocean surface by dry deposition and snow scavenging, then enter the ocean through summer melting of pack ice and continental runoff.

In the summer, precipitation scavenging and direct air-to-water transfer of gaseous PCCs may also contribute to the annual deposition. The air-water partition coefficient (Henry's Law constant, H) of toxaphene at 20°C is 6.1×10^{-6} atm-m^3·mol^{-1} in fresh water (Murphy *et al.*, 1987). The variations in this constant with temperature and salinity have not been experimentally determined, but H can be estimated as 8.9×10^{-7} atm-m^3·mol^{-1} under Arctic Ocean surface conditions (−2°C, 32 ppt salinity) using the assumptions made for HCHs (Patton *et al.*, 1989). Based on the latter H value, equilibration of surface water with the average concentration of PCCs in the atmosphere (40 pg·m^{-3}) would result in 1000 pg·L^{-1} dissolved PCCs. Our summertime measurement of PCCs at 10 m depth was about one-third of this concentration (Table 1).

The potential atmospheric sources of PCCs in the Canadian Arctic are diverse. Toxaphene was banned in the U.S. near the end of 1982 following evidence of its mammalian carcinogenicity and acute and chronic toxicity to aquatic life (U.S. Government, 1982). Volatilization of toxaphene may still be occurring from soils in the United States, where over 1.6×10^5 tonnes were applied between 1972 and 1982 (Bidleman *et al.*, 1988), or from Mexico, where use continues (FAO, 1986). The presence of PCCs in fish from the Great Lakes (Rice and Evans, 1984; Swackhamer *et al.*, 1987; Swackhamer and Hites, 1988) and in peat bogs from the Great Lakes region and northeastern Canada (Rapaport and Eisenreich, 1988) attests to their atmospheric deposition at least as far north as the U.S.–Canada border.

However North America may not be the primary source of PCCs to the Arctic. PCCs have been found in birds and aquatic life from the Baltic Sea (Andersson *et al.*, 1988; Jansson and Wideqvist, 1983; Pyysalo and Antervo, 1985), in air and rain from southern Sweden (Bidleman *et al.*, 1987; Sundström, 1981), and in human milk from Sweden (Vaz and Blomqvist, 1985) and Finland (Pyysalo and Antervo, 1985), even though PCCs are not used in Scandinavia. PCCs were among the major OCs in rain in the western Mediterranean Sea area (Villeneuve and Cattini, 1986), and they have been found in fish from alpine lakes (Zell and Ballschmiter, 1980).

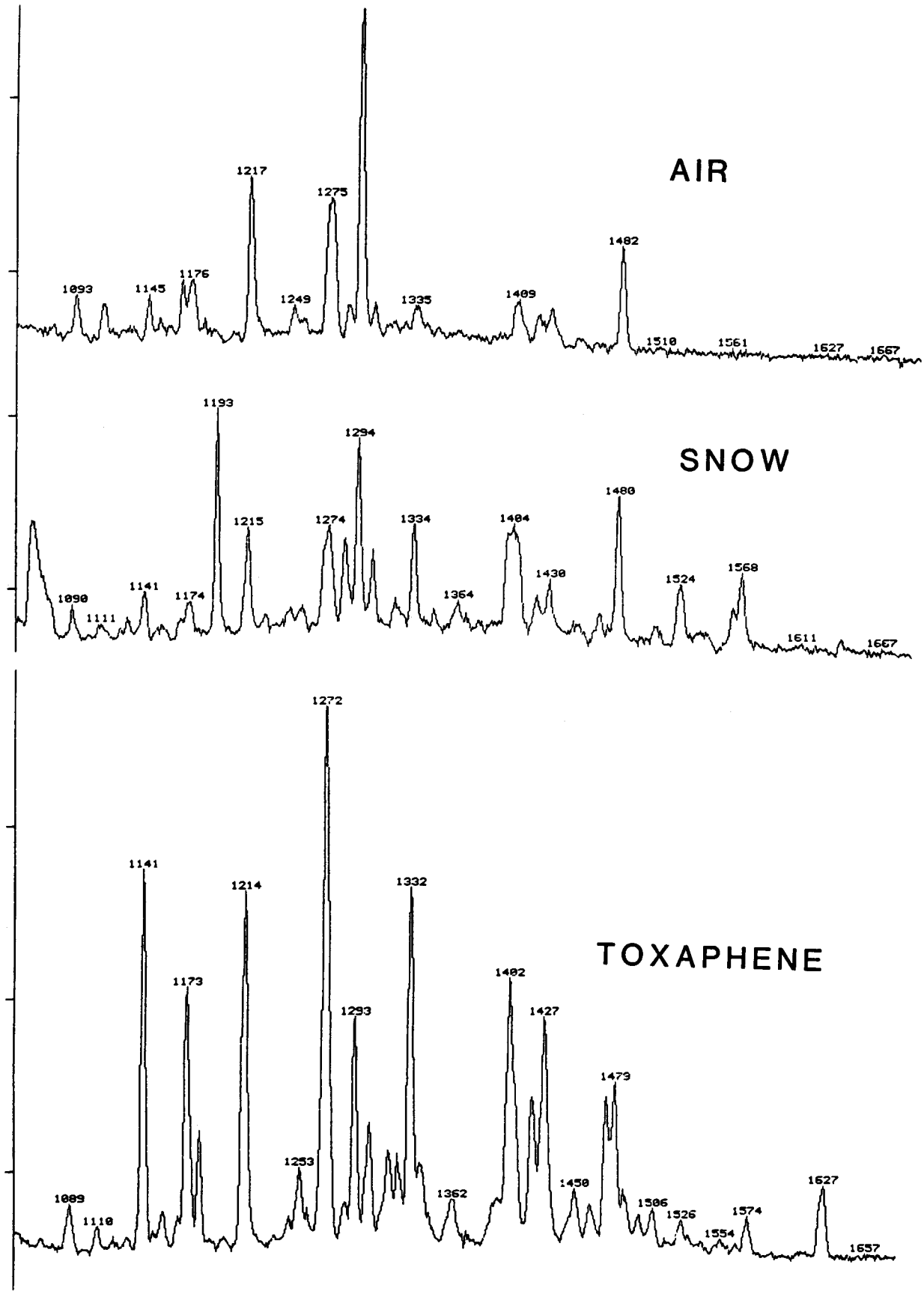

FIG 1. Reconstructed GC-NIMS chromatograms of PCCs in an air sample from the Ice Island, snow from Devon Island, and a toxaphene standard.

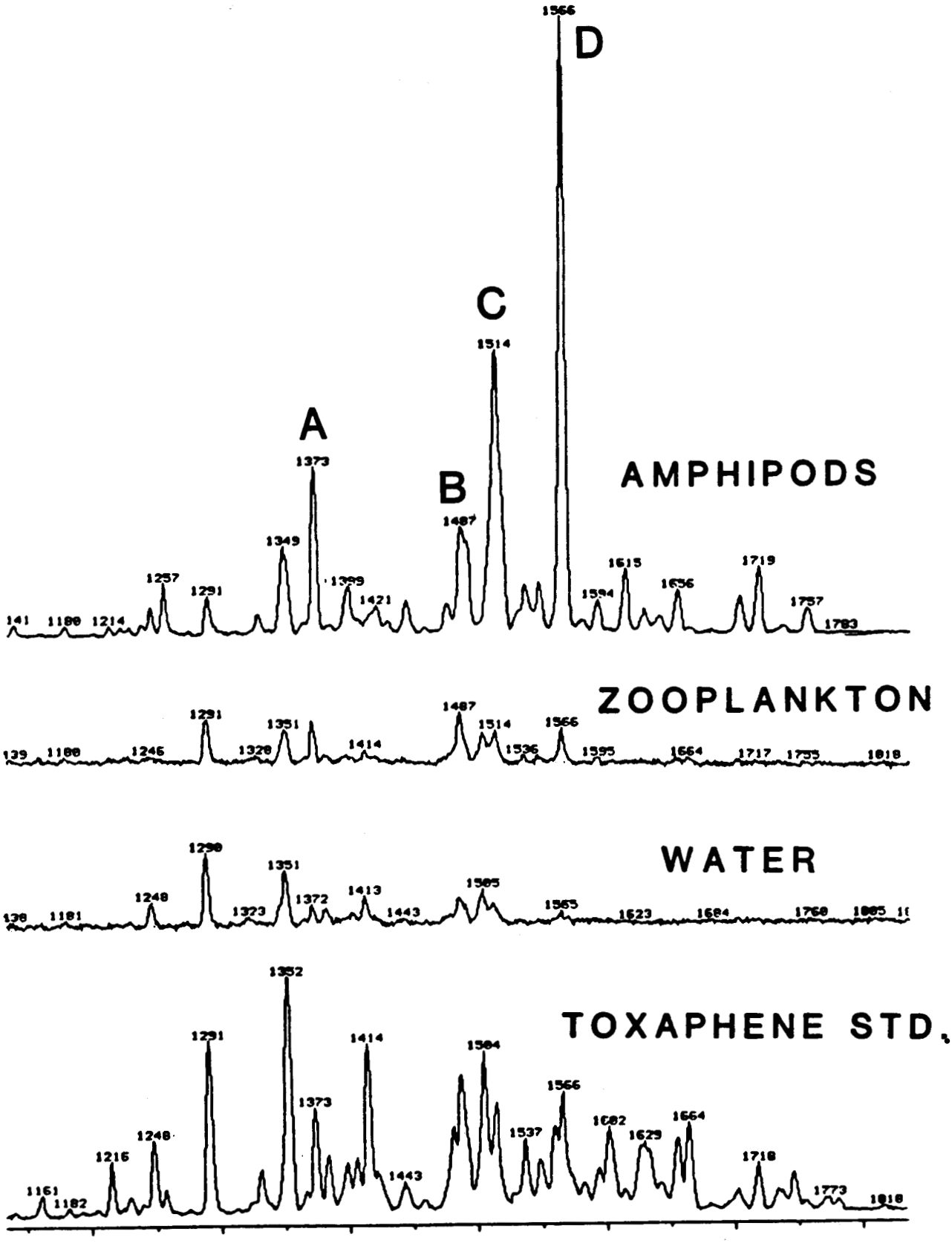

FIG 2. Reconstructed GC-NIMS chromatograms of PCCs in amphipods, zooplankton, and water from the Ice Island. Full-scan spectra were obtained for peaks A-D in the amphipod sample. M/Z and abundances (found)(theoretical) for the $(M\text{-}Cl)^-$ clusters of peak C were: 375 (39.1)(44.6), 377 (100)(100), 379 (91.8)(95.9), 381 (42.4)(51.1), 383 (8.8)(16.3). Similarly for peak D: 409 (30.0)(34.9), 411 (88.2)(89.3), 413 (100)(100), 415 (65.5)(64.0), 417 (20.6)(25.6). These are consistent with the assignments of $C_{10}H_{10}Cl_8$ for peak C and $C_{10}H_9Cl_9$ for peak D. Peaks A and B were also identified as $C_{10}H_{10}Cl_8$.

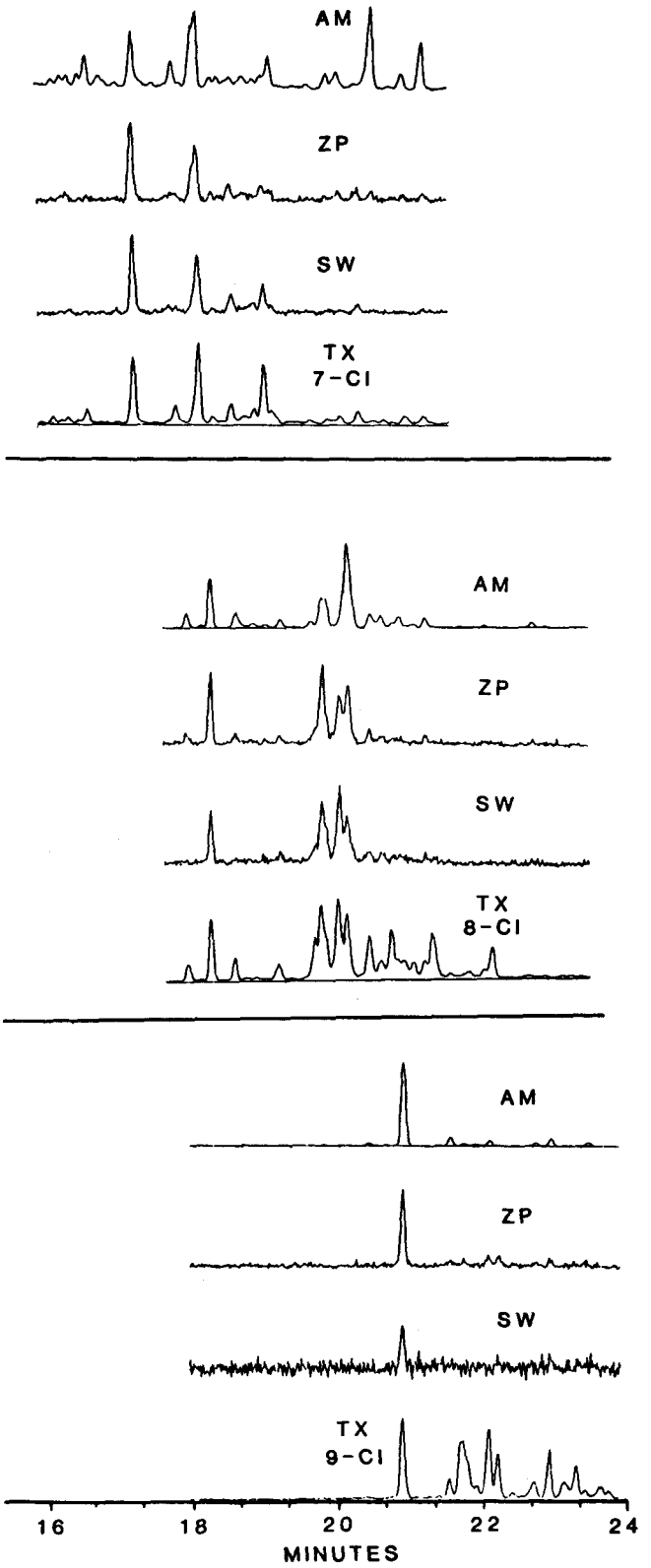

FIG 3. Upper: PCCs containing seven chlorines in amphipods (AM), zooplankton (ZP), seawater (SW), and toxaphene standard (TX); $(M\text{-}Cl)^-$ SIM ions 343, 345. Middle: as above, for PCCs containing eight chlorines; ions 379, 381. Bottom: as above, for PCCs containing nine chlorines; ions 413, 415. Chromatogram attenuations have been varied to facilitate peak display; relative proportions of PCC homologs are given in the text. Mass spectrum of Cl_9 PCC in AM (*) is shown in Figure 4.

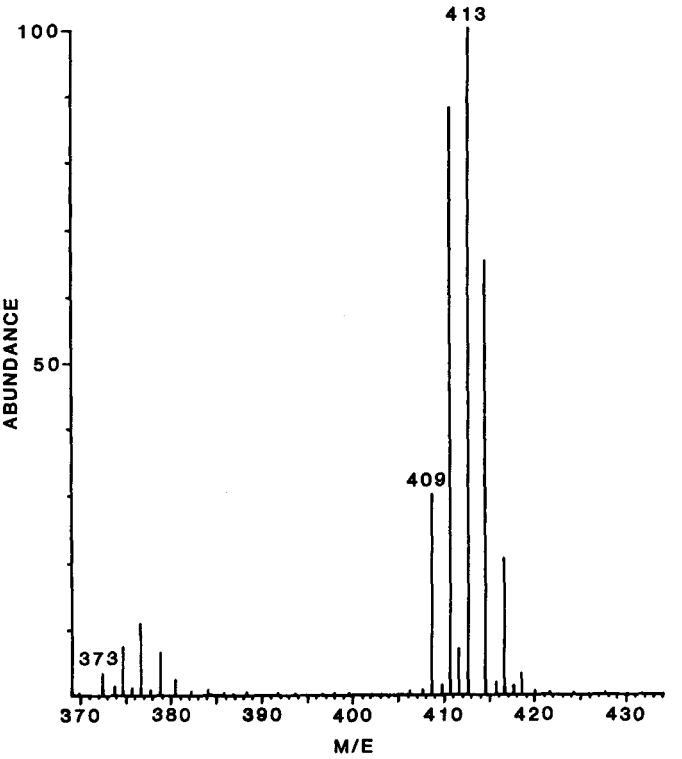

FIG 4. NI mass spectrum of Cl_9 PCC component in amphipod sample (peak marked D in Figure 2, and * in Figure 3). Relative ion abundances given in Figure 2.

Use of PCCs has been reported by Czechoslovakia, Poland, and Hungary (FAO, 1986) and by the Soviet Union (Izmerov, 1983). Since transport of combustion-related pollutants to the Arctic is predominantly from Eurasia, the supply of PCCs and other OCs from eastern vs. western hemispheres merits further investigation.

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