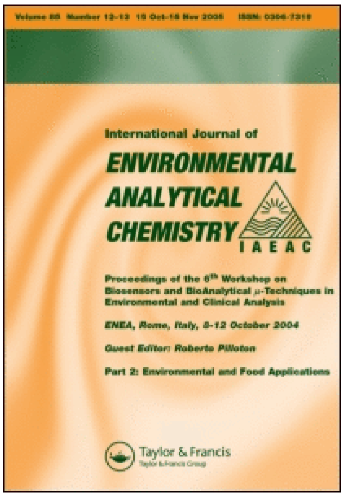

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ORGANIC COMPOUNDS IN SURFACE AND DEEP ANTARCTIC SNOW

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Eight surface snow samples taken during the 1987/88, 1988/89 and 1990/91 Italian Antarctic Expeditions and six samples collected at different depths from two dissimilar sites during the 1990/91 Expedition, were analyzed for the non-chlorinated organic content using the GC capillary columns technique and GC-MS. Several biogenic and anthropogenic classes of organic compounds were identified and quantitatively determined. The data obtained give a more complete picture of the pollution level in Antarctica.

KEY WORDS: Antarctica, snow, organics, pollutants, chromatographic analysis.

INTRODUCTION

Snow and ice are certainly the matrices that best characterise the Antarctic continent and are fundamental in the study of the input mechanism of transferring pollutants originating from distant areas through the atmosphere. Snow is the only means of wet deposition in Antarctica and is therefore the means of transferring pollutants from the atmosphere to the continent, together with dry deposition. Despite its importance, it has not been intensively studied for its organic content until now. The only research available concerns the analysis of chlorinated organic compounds (DDTs, HCHs, PCBs) used as pollution markers[1-3]. The aim of this work is the systematic study of non-chlorinated compounds, both individually and in groups, present in surface snow samples, and in snow taken at different depths during the years 1987 to 1991.

EXPERIMENTAL

Sampling

The sampling stations for surface snow were the following (see Figure 1):
2SN – Campbell Glacier, 920m, Lat. 74°11′ S; Long. 164°02′E.
4SN – Mt. Melbourne, 1130m, Lat. 74°26′S; Long. 164°45′E.
5SN – Tourmaline Plateau, 1650m, Lat. 74°11′S; Long. 163°30′E.
9SN – Mt. Crummer, 700m, Lat. 75°05′S; Long. 162°40′E.
10SN – Vegetation Island, 220m, Lat. 74°47′S; Long. 163°38′E.
17SN – Carezza Lake, Lat. 74°43′S; Long. 164°01′E.

The sampling stations for snow samples at different depths were (see Figure 1):
19SN – Styx Glacier (Plateau), 1700m, Lat. 73°52′S; Long. 163°41′E.
27SN – Mc Carthy Ridge, 700m, Lat. 74°32′S; Long. 162°56′E.

The surface snow samples were taken as follows: after eliminating the top layer (about 5cm) to avoid contamination of the sample, a scoop was used to collect a layer of snow approximately 5cm thick, so to obtain a total volume of about 40 liters.

Snowpit samples 19SN and 27SN were collected at –2m, –1m and at the surface, after eliminating the layer exposed to the atmosphere. For the –1m and –2m samples, a horizontal layer about 10cm thick was collected, so to obtain a total volume of about 40 liters.

The samples were immediately frozen and kept at –30°C until the time of analysis.

Reagents and materials

20 Liter stainless steel reservoirs (Inox Sabat, Bologna, Italy) were used for storage of the snow samples. Solvents (*n*-hexane, methylene chloride, acetone) were all pesticide grade purchased from Merck (GFR). Anhydrous sodium sulphate was heated for 12 hours at 450°C to remove any organic matter and then kept at 120°C until use. All apparatus was cleaned before use by repeatedly washing with chromic and concentrated sulphuric acid mixture, purified water (UHQ – Elgastat system, England), acetone, methylene chloride and *n*-hexane. Standard organic compounds are commercially available from Supelco (USA) and Alltech (USA).

Analysis

Only the internal part of each sample, which did not come into contact with the walls of the container was analyzed to avoid any eventual contamination. The samples were melted at room temperature in a glass column (14cm ID), kept under a nitrogen (chromatography grade) flow. The walls of the column were washed with 20ml acetone after the melted snow was taken out of the column, and the resulting water/acetone mixture was extracted with 3ml of *n*-hexane. The extraction of the melted snow was effected by the replicated extractant

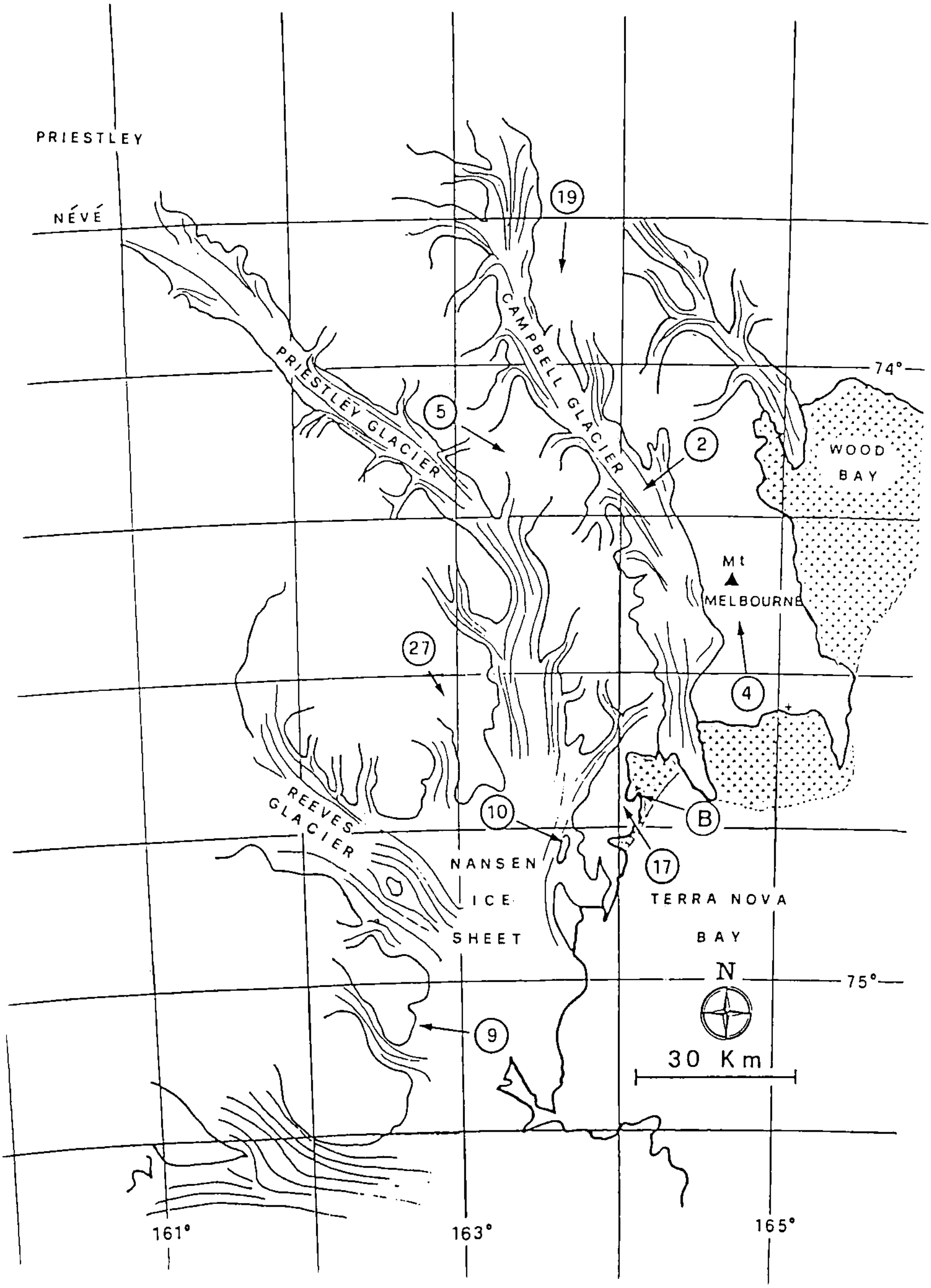

Figure 1 Snow sampling points of the three Italian Antarctic Expeditions 1987/88, 1988/89, 1990/91.

enrichment method, especially designed for Antarctic aqueous matrices[4]. 3ml of *n*-hexane were used to extract three successive aliquots of 1 liter of unfiltered samples. The 3ml of *n*-hexane used to treat the water/acetone mixture were added to the above mentioned extracts and the resulting volume was dried under sodium sulphate and cold evaporated to 100μl under a nitrogen flow in standardized conditions[5]. The entire analytical procedure was repeated five times for every sample.

The total recovery for the different classes, calculated by using a standard mixture containing aliphatic and aromatic hydrocarbons and heterocompounds (phthalates and other compounds containing O, S or N atoms) at concentration levels of 10ng/l, was the following:

Classes of organic compounds	Recovery (%)	St.Dev.
Aliphatic hydrocarbons	85	5
Alkyl benzenes	70	10
Polycyclic aromatic hydrocarbons	80	7
Heterocompounds	70	10

The detection limit of the method was 5ng/l.

In contrast with other matrices (sea water, sea ice and sediments), surface and deep snow do not require a preliminary fractionation of the organic compounds in homogeneous classes for their identification and quantitative determination.

Gc and GC-MS analysis

For the identification and quantitative determination of organic compounds, an HRGC-5160 Mega Series (Carlo Erba, Italy) gas-chromatograph equipped with a FID detector was used. The injection was made by using a Cold-SSL injector (Carlo Erba) according to the following temperature program: injection at 40°C, then a rapid increase in temperature to 300°C and splitting after 30sec. Column temperature program: 40°C for 1min., then linear increase to 300°C at 4°C/min., and finally isotherm at 300°C for 15min. Supelco PTE-5 capillary columns (30m, 0.25mm ID, 0.25μm thickness) were used; carrier gas: hydrogen. The chromatographic peaks were analyzed with a Mega-2 computer system (Carlo Erba) with Spectra Physics software. GC-MS analyses were performed on a Varian 3400 gas-chromatograph coupled with a Finnigan ITD mass detector; carrier gas: helium. An injector SPI (Varian) was used according to the following temperature program: injection at 40°C, then a rapid increase to 300°C. The column temperature program was the same as that described above.

Identification of organic compounds

The identification was realized using the following two methods:

a) by employing a dedicated software using the gas-chromatographic retention indices with eight *n*-alkanes (C-8, C-12, C-16, C-24, C-28, C-32, C-34) as standards and a reference calibration table;

b) by comparing mass spectra of compounds using the N.B.S. library and a second library made in our laboratory on ITD by using suitable standards.

The exact name is reported in Tables 1–4 only for those compounds positively identified with both methods, while only the belonging class is given for the others.

RESULTS AND DISCUSSION

Surface snow

Tables 1 and 2 report the identified organic compounds and their concentrations in eight surface snow samples taken during the three Italian Antarctic Expeditions. The results clearly show that the surface snow is a matrix particularly rich in both biogenic and anthropogenic organic compounds. The biggest group contains *n*-alkanes included between C-9 and C-32. The three classes of anthropogenic compounds (alkyl benzenes, PAHs and phthalates) are present at high levels in most of the samples. The analyzed surface snow contains a great variety of biogenic compounds: *n*-alkanes, aldehydes, fatty acids, alcohols, squalene and cholesterol. Their concentrations, however, change noticeably from sample to sample with the exception of *n*-alkanes which can be both biogenic and anthropogenic. The average concentration value of the eight snow samples is 642 ng/l for *n*-alkanes in a range of 369–1231 ng/l. These results confirm the validity of the analytical method used, considering that these data refer to samples collected from different sites and in different years. Sample 17SN (Carezza Lake), taken in the sampling area nearest to the Italian Base and to the coast, characterized by the presence of Skua birds, is valid for evaluating the contribution of the local fauna to biogenic compounds. This sample contains the highest concentrations of aliphatic aldehydes and alcohols and, above all, of fatty acids (11482 ng/l) and cholesterol (2390 ng/l). It is interesting to note the presence of some biogenic compounds, even if in small amounts, in samples collected in areas far from the influence of animals; for example, 4SN (Mt. Melbourne) and 5SN (Tourmaline Plateau). This fact may indicate a contribution by air currents coming from the sea, and/or marine aerosol, since these compounds are commonly present in marine and coastal matrices[6–8].

As far as the anthropogenic compounds are concerned, a quite homogeneous concentration in all samples is to be noted (see Table 2). In addition, some compounds like meta- and para-xylene, trimethylbenzenes, diiso-, di-n-butyl and bis(2-ethylhexyl)phthalate, are present in all of the samples at much higher levels than the detection limit of the method (5 ng/l). The most abounding groups are the alkyl benzenes and phthalates, as found for the other environmental antarctic matrices[6–8]. The results we obtained clearly show that these compounds are new classes of antarctic pollutants. The histograms of Figure 2 point out the ratio of the concentrations of the three classes of anthropogenic compounds present in the eight surface snow samples. It should be noted that the 10SN samples taken from the same site in different years, show a rather constant concentration of alkyl benzenes and phthalate esters. This fact indicates a rather homogeneous pollution level in the area studied, probably due

Table 1 Organic compounds in surface Antarctic snow (ng/l); medium values of five determinations ± standard deviation; * = detection limit; bdl = below detection limit.

	1987/88		*1988/89*		*1990/91*			
Compounds	*9SN*	*2SN*	*10SN*	*10SN*	*5SN*	*10SN*	*4SN*	*17SN*
n-ALKANES								
C-9	10±3	bdl	bdl	bdl	bdl	bdl	10±1	26±3
C-10	51±5	6±1	29±3	40±4	bdl	50±6	35±4	95±10
C-12	10±1	5*	bdl	bdl	bdl	bdl	5*	39±4
C-13	bdl	bdl	bdl	5*	bdl	bdl	bdl	42±4
C-14	47±5	bdl	bdl	7±1	40±4	5*	5*	116±10
C-15	55±5	19±2	44±4	26±3	41±4	26±3	20±2	171±15
C-16	110±10	70±8	62±6	40±4	60±6	12±1	7±1	64±6
C-17	60±6	19±2	52±5	26±3	47±5	52±5	24±2	bdl
C-18	38±4	5*	8±1	19±2	22±2	10±1	20±2	10±1
C-19	10±1	5*	28±3	14±2	bdl	22±2	bdl	bdl
C-20	5*	bdl	10±1	16±2	5*	20±2	7±1	bdl
C-21	5*	5*	17±2	20±2	10±1	24±3	20±2	bdl
C-22	5*	5*	6±1	28±3	10±1	24±2	22±2	bdl
C-23	10±1	bdl	bdl	30±3	10±1	26±3	25±3	46±5
C-24	24±2	34±3	10±1	24±3	10±2	48±5	bdl	30±3
C-25	24±2	33±3	25±3	20±2	23±2	113±10	44±4	12±1
C-26	20±2	36±4	30±3	28±3	14±2	113±10	60±6	38±4
C-27	17±2	37±4	29±3	25±3	16±2	120±10	45±5	33±3
C-28	14±1	37±4	31±3	20±2	15±2	135±11	48±5	31±3
C-29	27±3	45±4	33±3	27±3	20±2	130±11	51±5	85±8
C-30	11±1	54±5	27±3	12±1	10±1	118±10	52±5	45±4
C-31	5*	56±6	21±2	10±1	16±2	97±10	37±4	41±4
C-32	5*	49±5	5*	8±1	bdl	86±9	52±5	37±4
Total *n*-alkanes	563	517	467	445	369	1231	589	961
ALDEHYDES								
Nonanal	bdl	13±2	139±15	94±15	546±50	140±18	90±14	393±40
Undecanal	11±2	7±2	13±3	5*	18±4	20±5	bdl	139±15
Dodecanal	bdl	bdl	bdl	bdl	21±4	bdl	bdl	16±3
Tetradecanal	bdl	bdl	bdl	bdl	bdl	bdl	bdl	65±12
Total aliphatic aldehydes	11	20	152	99	585	160	90	613

FATTY ACIDS								
Tetradecanoic acid	bdl	bdl	bdl	bdl	bdl	bdl	bdl	3630±320
Hexadecanoic acid	10±2	800±80	15±2	23±2	bdl	bdl	bdl	7300±500
Octadecanoic acid	bdl	1500±100	10±2	75±8	bdl	bdl	bdl	552±50
Total fatty acids	10	1300	35	98	bdl	bdl	bdl	11482
Total fatty acid esters	bdl	bdl	20	bdl	bdl	bdl	6	bdl
ALCOHOLS								
1-Octanol	bdl	bdl	bdl	bdl	32±6	bdl	bdl	bdl
1-Nonanol	bdl	bdl	bdl	bdl	27±5	bdl	bdl	bdl
1-Decanol	bdl	bdl	bdl	bdl	12±3	bdl	bdl	bdl
1-Dodecanol	bdl	bdl	bdl	bdl	bdl	bdl	bdl	203±35
1-Tridecanol	bdl	bdl	bdl	bdl	bdl	bdl	bdl	112±20
1-Tetradecanol	bdl	bdl	bdl	bdl	bdl	bdl	bdl	387±50
1-Pentadecanol	bld	bld	bdl	bdl	bdl	bdl	bdl	32±6
Total aliphatic alcohols	bdl	bdl	bdl	bdl	71	bdl	bdl	734
Squalene	bdl	bdl	bdl	bdl	144±10	30±3	121±10	255±20
Cholesterol	10±2	50±10	60±10	53±10	30±7	107±20	53±10	2390±350
Sulphur	5	bdl	bdl	bdl	bdl	5	5	5

Table 2 Anthropogenic organic compounds in surface Antarctic snow (ng/l); medium values of five determinations ± standard deviation; * = detection limit; bdl = below detection limit.

		1987/88			1988/89			1990/91
Compounds	*9SN*	*2SN*	*10SN*	*10SN*	*5SN*	*10SN*	*4SN*	*17SN*
BENZENES								
Ethylbenzene	15±3	bdl	13±3	bdl	22±5	16±5	5*	44±9
m/p-Xylene	58±12	12±2	37±8	40±8	93±20	12±3	69±15	198±35
o-Xylene	19±4	8±2	9±2	5*	33±6	bdl	19±5	80±17
Cumene	8±2	5*	bdl	16±4	bdl	bdl	bdl	bdl
Propylbenzene	9±2	5*	24±6	bdl	5*	bdl	bdl	13±5
m/p-Ethyltoluene	30±6	16±4	22±5	19±4	20±4	bdl	10±2	75±15
Mesytilene	31±6	7±2	9±2	27±7	17±4	20±5	bdl	30±6
o-Ethyltoluene	27±6	15±3	20±4	61±10	13±3	16±4	bdl	31±6
1,2,4-Trimethylbenzene	108±18	8±2	20±4	26±5	69±15	80±15	44±10	180±30
1,2,3-Trimethylbenzene	51±12	5*	27±6	13±3	27±6	16±3	10±2	55±12
1,2,3,5-Tetramethylbenzene	18±4	8±2	9±2	9±3	bdl	bdl	bdl	21±4
Butylbenzene	bdl	bdl	bdl	bdl	bdl	bdl	bdl	15±5
1-Methyl-2-nPropylbenzene	bdl	bdl	bdl	bdl	bdl	bdl	bdl	17±4
1,4-Dimethyl-2-Ethylbenzene	104±18	8±2	13±2	bdl	bdl	10±2	bdl	17±4
1,2-Dimethyl-4-Ethylbenzene	47±10	13±3	35±7	bdl	bdl	20±4	bdl	43±8
1,2,4,5-Tetramethylbenzene	bdl	bdl	bdl	bdl	bdl	bdl	bdl	20±4
C4-Benzene	43±8	bdl	bdl	bdl	8±2	bdl	bdl	28±6
1,2,3,4-Tetramethylbenzene	8±2	bdl	bdl	5*	bdl	bdl	bdl	bdl bdl
C5-Benzene	23±4	5*	12±2	bdl	bdl	45±8	bdl	bdl
Total alkyl benzenes 576	115	250	221	307	235	157	867	

POLYCYCLIC AROMATICS								
1,2,3,4-Tetrahydronaphthalene	21±4	5*	7±2	9±2	bdl	7±2	bdl	bdl
Naphthalene	29±5	bdl	5*	27±4	19±3	50±6	12±3	43±8
2-Methylnaphthalene	16±3	10±2	9±2	5*	5*	15±3	bdl	bdl
1-Methylnaphthalene	11±2	7±2	13±2	5*	18±3	20±3	bdl	bdl
Alkylnaphthalene	17±3	5*	14±2	bdl	bdl	bdl	bdl	bdl
Total PAHs	94	27	48	46	42	92	12	43
PHTHALATES								
Di-*iso*-butylphthalate	102±20	126±20	355±50	85±15	121±20	160±25	61±12	142±20
Di-*n*-butylphthalate	15±3	120±20	118±20	280±35	32±6	32±6	28±6	37±7
Bis(2-ethylhexyl)phthalate	bdl	88±15	66±12	147±24	126±20	289±35	173±15	205±30
Total phthalates	117	334	539	512	279	481	262	384

Table 3 Organic compounds in surface and deep snow (ng/l); medium values of five determinations ± standard deviation; * = detection limit; bdl = below detection limit.

Compounds	*19SN(−2)*	*19SN(−1)*	*19SN(0)*	*27SN(−2)*	*27SN(−1)*	*27SN(0)*
n-ALKANES						
C-9	5*	5*	bdl	bdl	7±1	10±1
C-10	5*	bdl	37±4	bdl	bdl	5*
C-11	70±7	70±7	137±12	65±7	58±6	80±8
C-12	bdl	101±10	117±12	bdl	bdl	bdl
C-13	bdl	25±3	20±2	7±1	bdl	bdl
C-14	13±2	39±3	25±3	bdl	bdl	12±1
C-15	bdl	bdl	20±2	7±1	bdl	13±1
C-16	30±3	187±12	40±4	40±4	38±4	5*
C-17	31±3	15±2	59±6	24±2	32±3	5*
C-18	10±1	bdl	87±9	bdl	18±2	bdl
C-19	30±3	22±2	76±8	5*	25±2	20±2
C-20	bdl	5*	bdl	bdl	5*	10±1
C-21	45±4	43±4	21±2	14±1	13±1	20±2
C-22	31±3	24±2	13±1	14±1	7±1	15±1
C-23	39±4	35±4	39±4	14±1	13±1	30±3
C-24	80±8	60±6	110±10	17±2	21±2	49±5
C-25	41±4	57±6	78±8	21±2	8±1	57±5
C-26	122±10	67±6	92±9	25±2	19±2	83±9
C-27	143±10	62±6	70±7	38±4	9±1	53±5
C-28	155±10	66±6	91±9	20±2	19±2	61±6
C-29	176±12	38±4	80±8	31±3	19±2	61±6
C-30	200±12	49±4	48±5	31±3	10±1	50±5
C-31	180±12	40±4	38±4	21±2	7±1	31±3
C-32	175±12	48±4	34±3	7±1	6±1	30±3
C-33	111±10	27±3	bdl	15±1	bdl	22±2
C-34	104±10	9±1	bdl	bdl	bdl	10±1
C-35	64±6	bdl	bdl	bdl	bdl	bdl
Total *n*-alkanes	1790	1024	1195	351	276	652

ALDEHYDES						
Benzaldehyde	5*	5*	bdl	bdl	bdl	bdl
Octanal	5*	bdl	37±8	bdl	bdl	5*
Nonanal	70±17	70±15	137±20	65±13	58±12	80±18
Undecanal	27±6	7±2	10±2	5*	7±2	7±2
Dodecanal	11±2	bdl	bdl	8±2	8±2	bdl
Total aldehydes	118	82	184	78	73	92
FATTY ACIDS ESTERS						
Fatty acid ester	bdl	34±6	bdl	17±4	32±6	58±12
Tetradecanoic acid methyl ester	39±8	32±6	bdl	bdl	bdl	5*
Octadecanoic acid methyl ester	54±10	10±2	bdl	bdl	bdl	bdl
Total esters	93	76	bdl	17	32	63
ALCOHOLS						
1-Octanol	11±2	24±4	44±8	9±2	bdl	10±2
1-Nonanol	12±2	28±5	bdl	bdl	bdl	11±2
1-Decanol	650±80	2350±250	232±30	134	107±20	511±60
1-Tridecanol	18±4	bdl	bdl	bdl	bdl	bdl
Total alcohols	691	2402	276	143	107	532
Squalene	26±3	95±9	183±20	bdl	98±8	36±4
Cholesterol	bdl	83±20	66±15	bdl	bdl	bdl

Table 4 Anthropogenic organic compounds in surface and deep snow (ng/l); medium values of five determinations ± standard deviation; * = detection limit; bdl = below detection limit.

Compounds	*19SN(−2)*	*19SN(−1)*	*19SN(0)*	*27SN(−2)*	*27SN(−1)*	*27SN(0)*
BENZENES						
Ethylbenzene	11±2	13±3	5*	7±2	bdl	10±2
m/p-xylene 13±2	8±2	29±6	14±2	12±3	14±3	
o-xylene	11±2	13±2	9±2	12±2	7±2	10±2
Propylbenzene	bdl	bdl	bdl	bdl	bdl	bdl
m/p-Ethyltoluene	bdl	5*	bdl	bdl	bdl	10±2
Mesytilene	5*	5*	bdl	bdl	bdl	5*
o-ethyltoluene	5*	bdl	bdl	bdl	bdl	5*
1,2,4-trimethylbenzene	9±2	7±2	bdl	7±2	5*	8
1,2,3-trimethylbenzene	5*	5*	bdl	7±2	5*	5*
1,3-dimethyl-5-ethylbenzene	bdl	bdl	20±4	bdl	bdl	bdl
1,4-dimethyl-2-ethylbenzene	5*	15±3	10±2	bdl	18±4	bdl
1,2-dimethyl-4-ethylbenzene	5*	7±2	10±2	bdl	bdl	bdl
1,3-bis(1-methylethyl)benzene	bdl	bdl	38±6	bdl	bdl	bdl
Alkylbenzene	20±4	bdl	bdl	bdl	bdl	bdl
Alkylbenzene	20±4	14±2	20±4	bdl	bdl	5*
Total alkyl benzenes	116	92	141	47	47	72
POLYCYCLIC AROMATICS						
Naphthalene	20±4	18±4	13±3	14±2	11±2	21±4
2-methylnaphthalene	11±2	bdl	20±4	bdl	bdl	bdl
1-methylnaphthalene	27±4	7±2	10±2	5*	7±2	7±2
Biphenyl	bdl	bdl	29±5	bdl	bdl	bdl
Acenaphthylene	13±2	bdl	22±3	bdl	bdl	bdl
Phenanthrene	14±2	bdl	5*	bdl	bdl	bdl
Total PAHs	85	25	99	19	18	28
PHTHALATES						
Di-*iso*-butylphthalate	379±70	966±160	1422±320	119±25	137±30	92±20
Dibutylphthalate	71±15	136±20	96±20	bdl	44±8	13±3
Benzylbutylphthalate	375±65	15±3	20±4	37±8	144±27	bdl
Bis(2-ethylhexyl)phthalate	530±60	211±30	bdl	8±3	32±6	5*
Total phthalates	1355	1328	1538	164	357	110

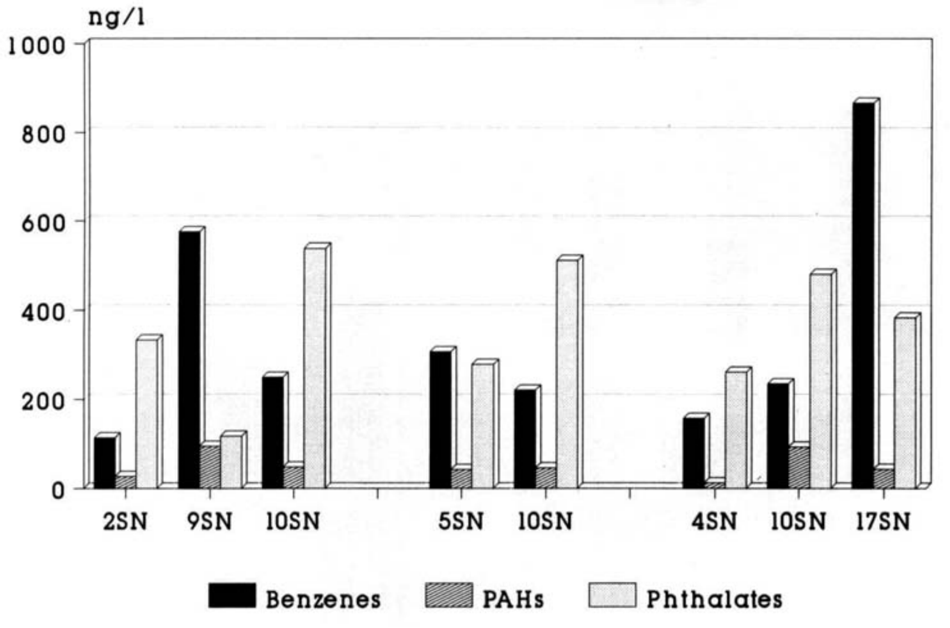

Figure 2 Total concentration of classes of anthropogenic organic compounds found in superficial snow samples collected in different years.

to long range atmospheric transport, as for the chlorinated pollutants, from industrialized regions[2,9,10]. In the case of sample 17SN, the closest to the Italian Base, there is probably local contribution also, as shown by the very high concentration of alkyl benzenes.

Deep snow

Tables 3 and 4 show the identified organic compounds and their concentrations, found in snow collected at different depths (–2m, –1m, surface) from two sites not previously sampled. The two surface snow samples, 19SN(O) and 27SN(O), contain the same organic compounds present in the above-mentioned samples (see Tables 1 and 2). Sample 27SN is the least polluted, while 19SN has a very high concentration of phthalate esters (1538 ng/l). There are some differences in the biogenic compounds, namely the lack of fatty acids. The deep snow samples taken at –1m and –2m refer respectively to the end of the winter 1989 and 1987 for 27SN and to the summer 1988/89 and 1985/86 for 19SN[11]. They contain the same anthropogenic compounds found in the corresponding surface snow at comparable concentration levels, as the histograms in Figure 3 clearly show. On the basis of these data, there is no evidence that the concentrations of the organic compounds change when the depth is increased to 2 meters.

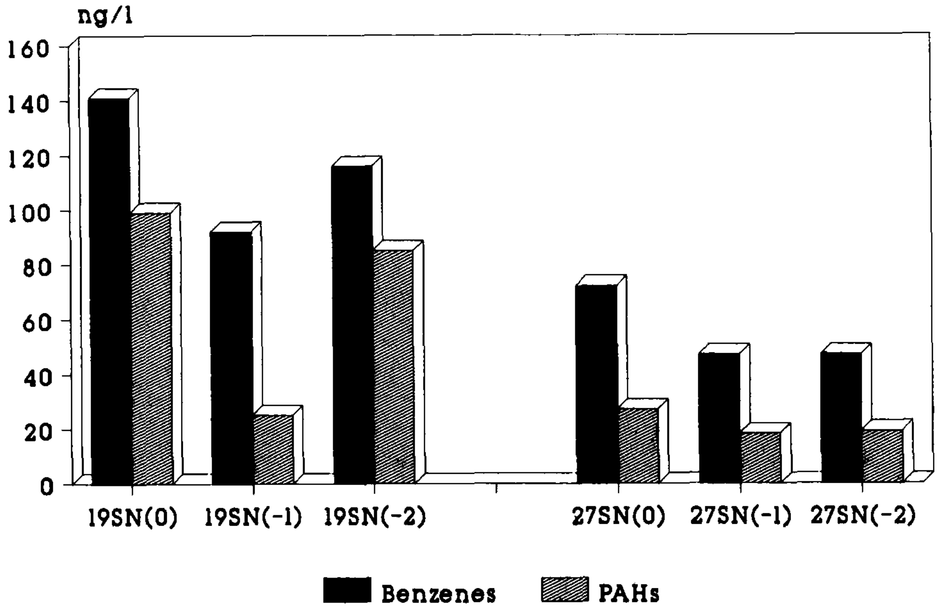

Figure 3 Total concentration of classes of aromatic hydrocarbons in snowpit samples collected at the surface,–1m and–2m during the 1990/91 expedition.

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