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Determination of Volatile Pollutants in Human and Animal Milk by GC-MS

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Volatile pollutants could readily be analysed in drinking water and blood samples (Eklund et al. 1978, Pfaffenberger et al. 1980, Kroneld & Reunanen 1983).

These substances are toxic, mutagenic, carcinogenic and teratogenic (Kuzma et al. 1977, Tardiff 1977, Tardiff et al. 1978, Page et al. 1980).

Becauce of their lipophilic nature they accumulate in the body and can also be analysed in animal tissues (Vogt et al. 1980, Bauer 1981). Although there are no reports of volatile halocarbons in milk samples, several papers deal with organochlorine compounds in milk samples (Skaare 1981, Rogirst et al. 1983, Wolff 1983).

Therefore, we considered it reasonable to modify and adapt our method for water and blood samples for analyses of possible volatile pollutants in milk samples and milk products.

MATERIAL AND METHODS

Twenty-five milk samples of human origin and ten samples from cows, pigs and sheep were collected both from the suburban area of Turku and from the countryside. Samples were also taken from pasteurized milk to evaluate whether the contamination of water by volatile pollutants could affect the milk. The samples from Turku and suburban areas were taken from humans and animals served by chlorinated drinking water. The samples from humans and animals taken from the countryside were, however, served by well water free from volatile halocarbons.

The samples (5 ml) were extracted with n-pentane (0.5 ml) containing a known amount of 1-chlorohexane as an internal standard. After centrifugation, the pentane

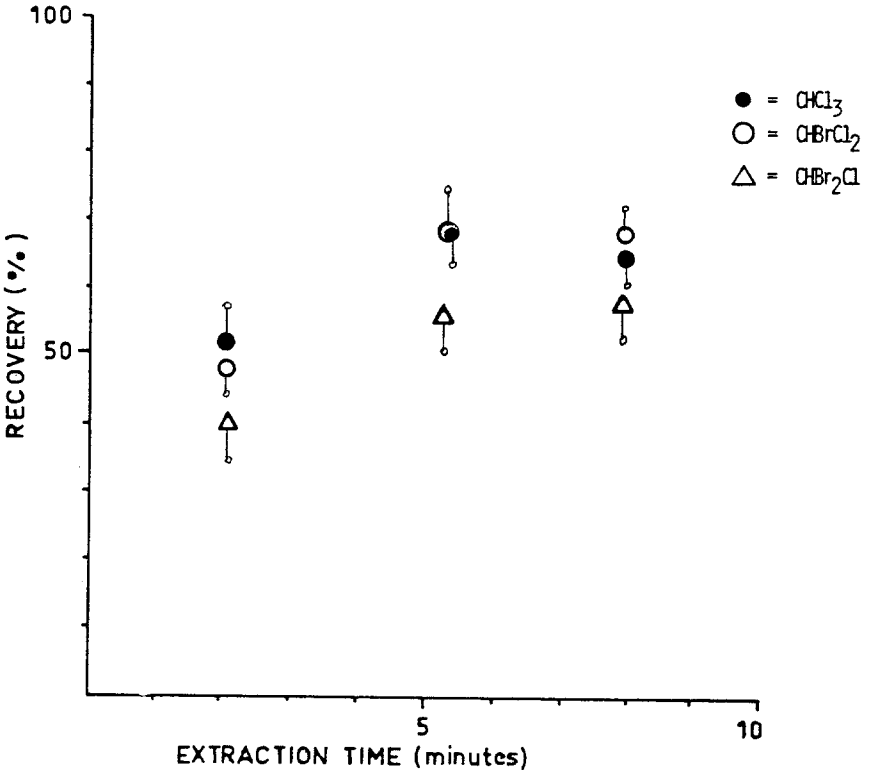

Figure 1. Recovery of volatile pollutants by means of pentane extraction from milk. Spike level for $CHCl_3$ (•) is 10 µg/l and for both $CHBrCl_2$ (o) and $CHBr_2Cl$ (△) 2 µg/l (h=12).

phase was analysed by gas chromatography-mass spectrometry (GC-MS) in selected ion monitoring mode (SIM).

Calibration of the system, as well as a test for extraction efficiency, was performed with spiked milk samples.

The recovery from milk, as a function of time, was analysed (Figure 1). The increase in the elution process was significant ($p < 0.001$) to five and eight minutes. Continued extraction for more than five minutes did not increase the recovery in any significant way (N.S.), which means that this was the minimum extraction time needed for elution of milk samples.

The chromatograms show volatile pollutants in a spiked milk sample (Figure 2). The method modified for milk analyses, was hence found to be as reliable as the one developed for blood samples.

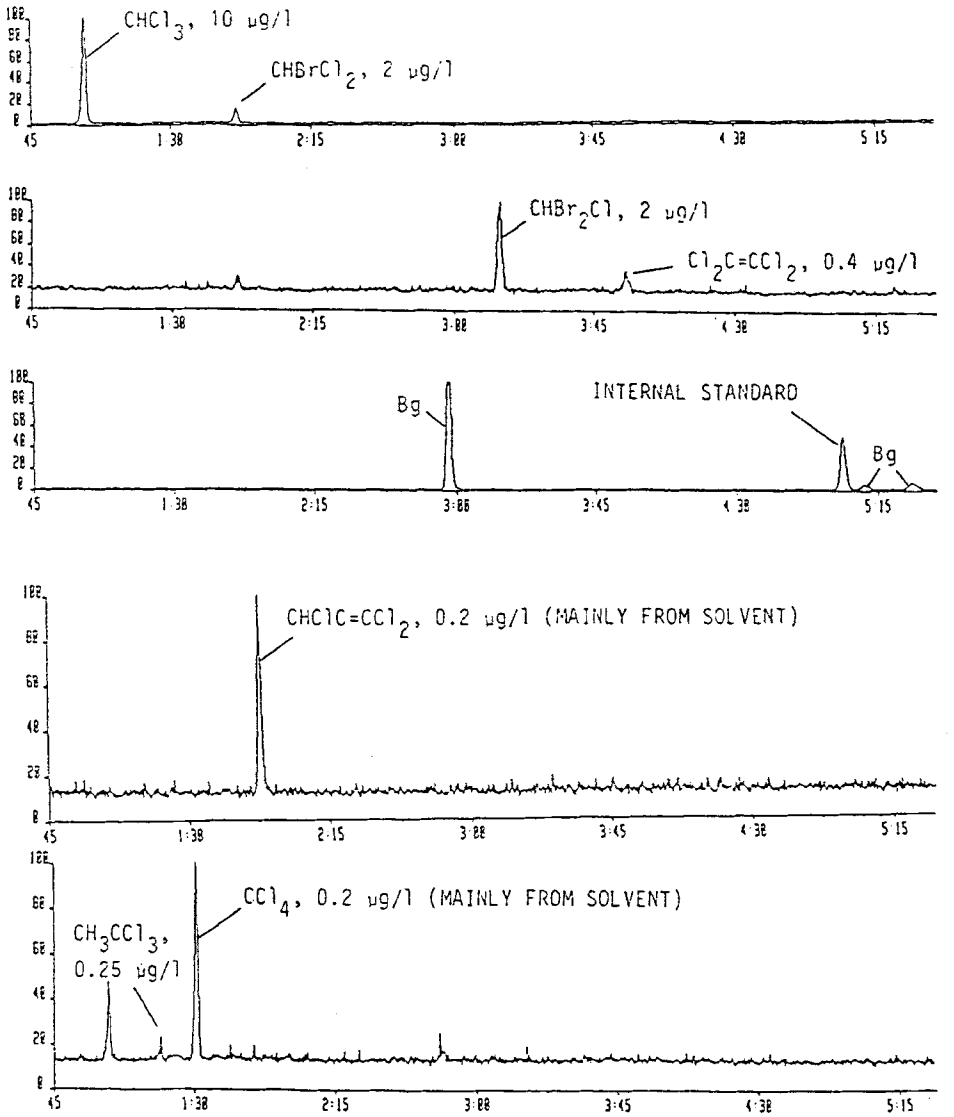

Figure 2. SIM-chromatograms from a spiked milk sample. Bg, solvent impurities.

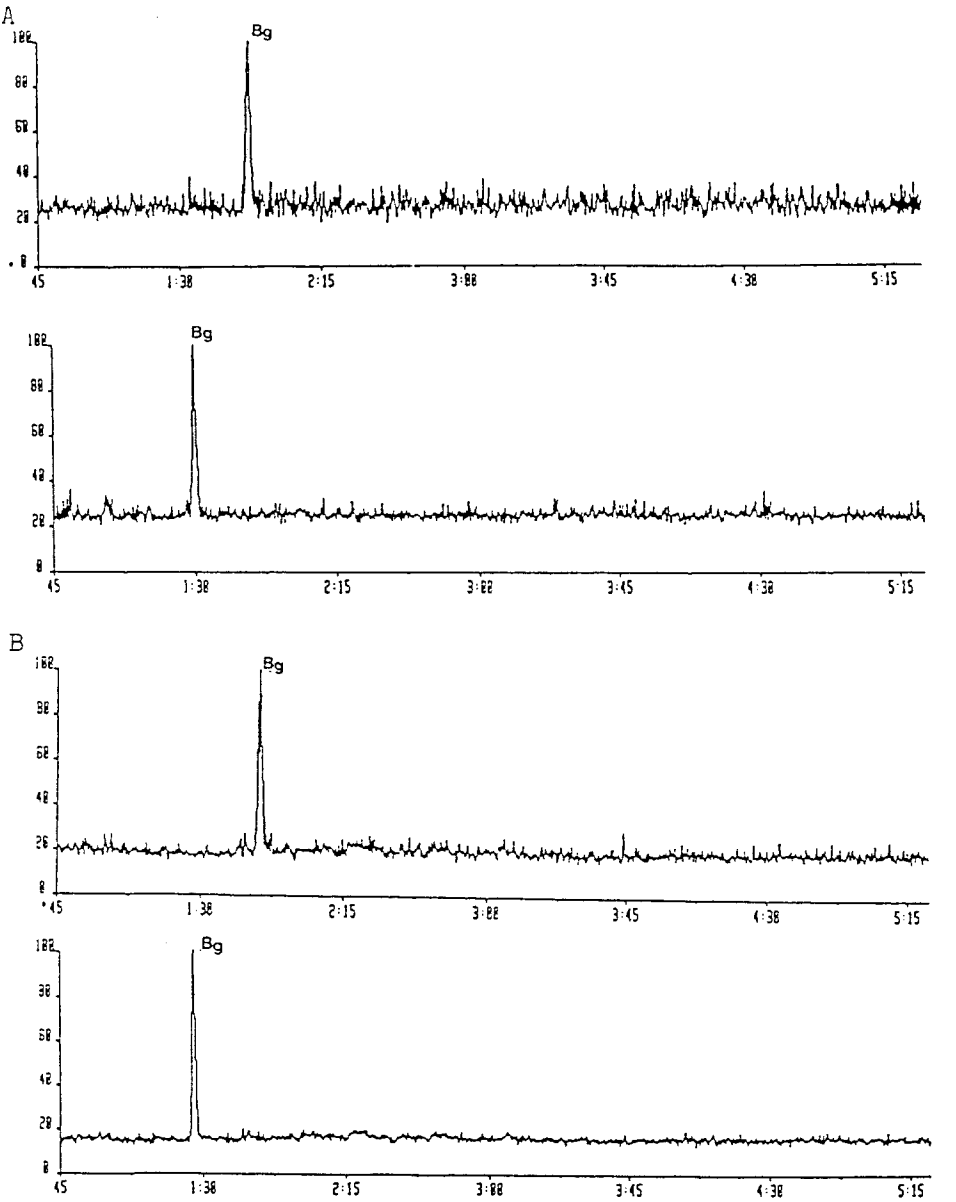

Figure 3. Milk samples for human milk from the countryside (A) and from the suburban area (B). Bg = solvent impurities.

The chromatograms from human milk are described in Figure 3 and of animal milk in Figure 4.

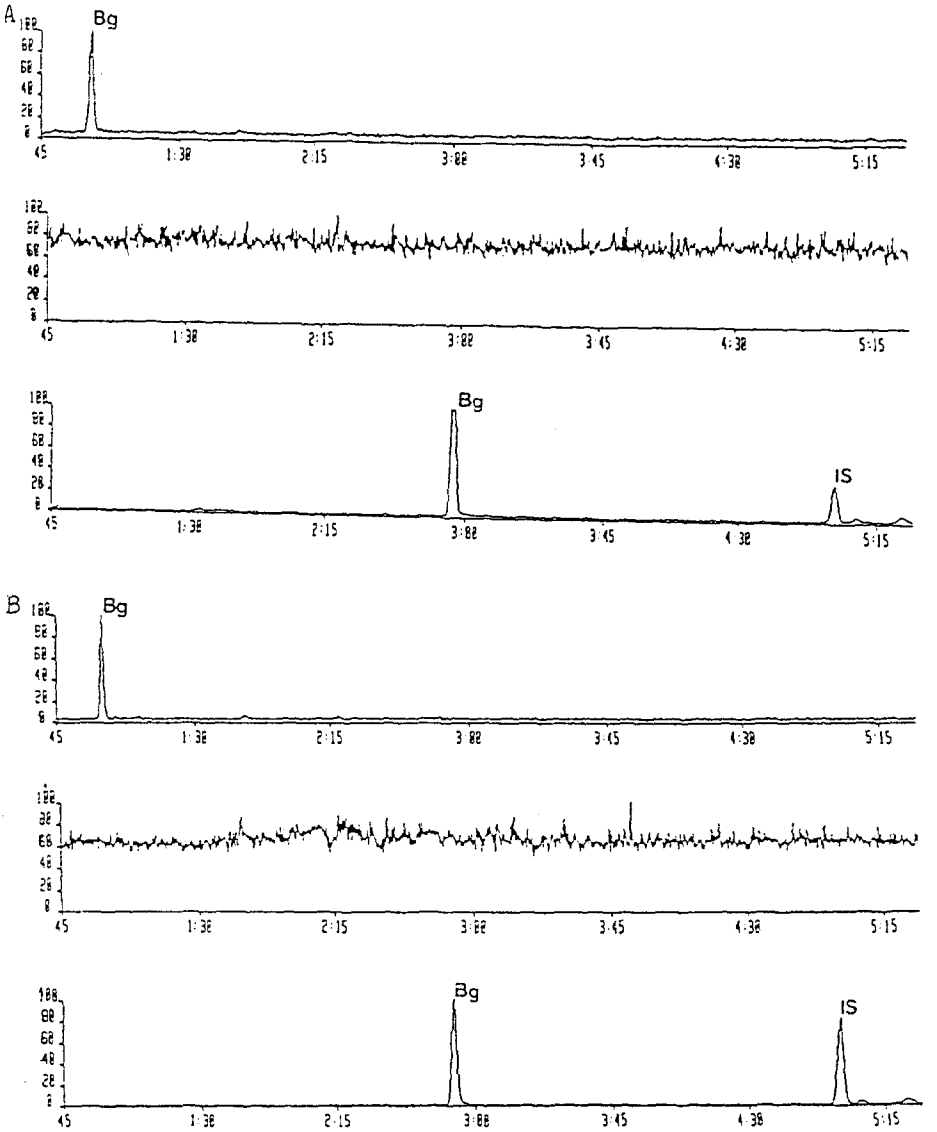

Figure 4. Milk samples from animal milk from the countryside (A) and from suburban area (B). Bg = impurities, IS = internal standard.

RESULTS AND DISCUSSION

The results of the study are shown in Table 1. Volatile compounds could hence be found only as traces in milk samples of human origin from the suburban area.

Volatile pollutants were otherwise not found in milk samples from humans or animals.

Table 1. Volatile halocarbons in human and animal milk samples from suburban areas and the countryside and in pasteurized milk.

Volatile compound µg/l	human		Milk						
	suburban area	country-side	suburban area	country-side	suburban area	country-side	suburban area	country-side	Milk, pasteurized suburban area
n	25	25	50	10	10	10	10	10	100
$CHCl_3$	+	ND	ND	ND	ND	ND	ND	ND	2.2 N.D-3.1
CH_3CCl_3	ND	ND	ND	ND	ND	ND	ND	ND	0.008 N.D-0.03
CCl_4	ND	ND	ND	ND	ND	ND	ND	ND	0.07 N.D-0.2
$ClCH=CCl_2$	ND	ND	ND	ND	ND	ND	ND	ND	+
$CHBrCl_2$	+	ND	ND	ND	ND	ND	ND	ND	0.008 N.D-0.07
$CBrCl_3$	ND	ND	ND	ND	ND	ND	ND	ND	+
$CHBr_2Cl$	+	ND	ND	ND	ND	ND	ND	ND	0.05 N.D-0.3
$BrCH_2CH_2Br$	ND	ND	ND	ND	ND	ND	ND	ND	+
$Cl_2C=CCl_2$	ND	ND	ND	ND	ND	ND	ND	ND	+
$CHBr_3$	ND	ND	ND	ND	ND	ND	ND	ND	+
toluene	ND	ND	ND	ND	ND	ND	ND	ND	+
m+p xylene	ND	ND	ND	ND	ND	ND	ND	ND	+
o-xylene	ND	ND	ND	ND	ND	ND	ND	ND	+

+ = traces

ND = not detectable

Volatile halocarbons were, however, found in food products as pasteurized milk. It is reasonable to assume that these halocarbons are due to contamination of chlorinated drinking water.

In earlier studies (Kroneld & Reunanen 1985, Pfaffenberger et al. 1980) it has been shown that these compounds could be found both in water and serum. The lipophilic nature of these compounds (Vogt et al. 1980) and having a low molecular weight easily penetrating the cell membranes, also makes it reasonable to assume that they could be found in milk.

A higher exposure of humans and animals might also be expected to make it possible to analyse such compounds in milk samples from areas with chlorinated drinking water.

The concentrations in drinking water, however, vary at different seasons between 25-130 µg/l. This means that the exposure level is quite low and comparable to blood sample analyses, where only haemodialysis patients showed an exposure level following the

seasonal changes in the water.

If volatile halocarbons in milk do accumulate, however, the authors would have expected to find small concentrations in both human and animal milk samples from the suburban area.

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