6. ANALYTICAL METHODS

6.1 BIOLOGICAL MATERIALS

The analytical methods for the determination of 1,2-dichloropropane in biological matrices are given in Table 6-l. The purge and trap method used for environmental samples is also commonly used for biological samples. The discussion about the methods that may be most sensitive for the determination of 1,2-dichloropropane levels in environmental samples, the advantages and disadvantages of the commonly used methods, and the precautions required to avoid evaporation losses as given in Subsection 6.2 is also applicable for biological samples.

6.2 ENVIRONMENTAL SAMPLES

As with all extremely volatile chemicals, it is essential to take precautions during sampling, storage, and analysis to avoid loss of 1,2-dichloropropane. Analytical methods for determining 1,2-dichloropropane in environmental samples are presented in Table 6-2. The two common methods that are used for the preconcentration of 1,2-dichloropropane for the determination of its levels in air are adsorption on a sorbent column or collection in a cryogenically-cooled trap, although the use of oxygen-doped electron capture detection may eliminate the need for preconcentration (Rasmussen et al. 1980). The disadvantages of cryogenic cooling are that the method is cumbersome and that condensation of moisture in air may block the passage of further air flow through the trap. The disadvantages of the sorption tubes are that the sorption and desorption efficiencies may not be 100% and that the background impurities in the sorbent tubes may limit the detection limit for samples at low concentrations (Cox 1983).

The most common method for the determination of 1,2-dichloropropane levels in water, sediment, soil and aquatic species is the purging of the vapor from the sample or its suspension in water with an inert gas and trapping the desorbed vapors in a sorbent trap. Subsequent thermal desorption is used for the quantification of its concentration.

The two methods that provide the lowest detection limits are halide-specific detectors (e.g., Hall electrolytic conductivity detector) and mass spectrometer. The advantage of halide specific detectors are they are not only very sensitive but are also specific for halide compounds. The mass spectrometer, on the other hand, provides an additional confirmation of the presence of a compound through the ionization patterns and is desirable when a variety of compounds are required to be quantified. The disadvantage of halide-specific detectors for their inability to detect and quantify nonhalogen compounds can be greatly overcome by using other detectors (e.g., photoionization detector) in series (Lopez-Avila et al. 1987; Driscoll et al. 1987). High-resolution gas chromatography with capillary columns is a better method for volatile compounds than packed columns because they provide better resolution of closely eluting compounds and increase the

TABLE 6-1. Analytical Methods for 1,2-Dichloropropane in Biological Samples

Sample Matrix	Sample Preparation	Analytical Method	Detection Limit	Accuracy	Reference
Exhaled air	exhaled air collected by valved Teflon Spriometer mouth pieces into Tedlar bag. The content of bag sorbed in Tenax and thermally desorbed	cryofocussing HRGC-MS	NG	NG	Barkely et al., 1980
Blood and urine	sample mixed with water purged at 50°C, trap in Tenax, thermal desorption	cryofocussing HRGC-MS	NG	NG	Barkely et al., 1980
Blood	sample mixed with water purged at ambient temperature and trapped in Tenax and thermally desorbed	GC-MS	<100 ppt	55-60% at 1 ppb	Cramer et al., 1988
Urine	sample equilibrated in sealed in vial at 37°C and head space gas analyzed	HRGC-MS	NG	NG	Ghittori et al., 1987

NG - Not given; GC - gas chromatography; HRGC - high resolution gas chromatography; MS - mass spectrometry

TABLE 6-2. Analytical Methods for 1,2-Dichloropropane in Environmental Samples

Sample Matrix	Sample Preparation	Analytical Method	Detection Limit	Accuracy	Reference
Ambient air	aliquot of sample collected in Tedlar bag, concentrated in a cryogenic trap and thermally disolved	GC-ECD	0.2 ppb	NG	Shikiya et al. 1984
Ambient air	aliquot of sample collected in electropolished cylinder cryogenically preconcentrated	GC-ECD	4 ppt	85-115%	Singh et al. 1982
Indoor/Outdoor air	sample collected by adsorption through charcoal desorbed by CS_2	HRGC-ECD	10 ppb	NG	DeBortoli et al. 1986
Occupational air	sample collected by adsorption on charcoal, desorbed in acetone/ cyclohexane	GC-HELD	33 $\mu g/m^3$ (7 ppb)	<95%	NIOSH 1984; *Boyd et al.* 1981; Dillon 1981
Air from industrial and chemical water disposal sites	sample collected by adsorption on Tenax, thermally desorbed	cryofocussing HRGC-MS	<0.2 $\mu g/m^3$ (<0.04 ppb)	>75%	Pellizzani 1982
Finished drinking and raw source water	purge and trap, thermal desorption	GC-MS	<0.1 µg/L (0.1 ppb)	90%	Otson 1987
	purge at ambient temperature, trap in Tenax/Silica/Charcoal and desorb thermally	GC-HEED (EPA Method 502.1)	NG	95% at 0.4 µg/L	EPA 1986a
	purge and ambient temerature, trap in Tenax/Silica/Charcoal desorb thermally	subambient programmable GC-NC (EPA Method 524.1)	0.17 µg/L (0.17 ppb)	101% 1 µg/L	EPA 1986a
	purge at ambient temperature, trap in Tenax/Silica/Charcoal, thermally desorbed	cryofocussing (wide or HRGC-MS (EPA-Method 524.2)	0.04 µg/L (wide bore) 0.02 µg/L (narrow bore)	97% (wide bore) at 0.1-10 µg/L 96% (narrow bore) at 0.5 µg/L	EPA 1986a
Drinking/ground/ surface water	vacuum distillation with cryogenic trapping	HRGC-ECD	0.03 µg/L	81%	Comba and Kaiser 1983
Water/waste-water	purge at ambient temperature, trap in Tenax/Silica/Charcoal, thermally	GC-HECD (EPA Method 601)	0.04 µg/L	97.7% at 0.29-39.0 µg/L	EPA 1982a

TABLE 6-2 (continued)

Sample Matrix	Sample Preparation	Analytical Method	Detection Limit	Accuracy	Reference
Water/wastewater	purge at ambient temperature, trap in Tenax/Silica/Charcoal, thermally	GC-HECD (EPA Method 601)	0.04 µg/L	97.7% at 0.29-39.0 µg/L	EPA 1982a
Wastewater	purge at ambient temperature, trap in Tenax/Silica thermally desorb	GC-MS (EPA Method 624)	6 µg/L	102-103%	EPA 1982a
Groundwater/ leachate	purge at ambient temperature, trap in Tenax/Silica, desorb thermally	GC-MS (EPA-CLP method)	5 µg/L	NG	EPA 1987a
Water and fish	dry purge and trap (water), somicated fish slurry subjected to dry purge and trap	cryofocussing HRGC-HECD/ PID in series	NG	NG	Driscall et al. 1986
Sediment/fish	vacuum distillation and condensation in supercooled trap	HRGC-MS	NG	96% (sediment) 54% (fish)	Hiatt 1981; Hiatt 1983
Marine biota/ sediment	homogenized ultrasonically (fish) or water suspension (sediment) sample purged at 70°C, trapped in Tenax/Silica and thermally desorbed	cold focussing HRGC-MS	<0.2 µg/kg	NG	Ferrario et al. 1985
Fish	cut tissue purged at 50°C, trap in charcoal desorb in CS_2	HRGC-FID	NG	61%	Reinert et al. 1983
Soil/sediment	purge sample suspension in water at 50°C, trap in Tenax/Silica, thermally desorb	GC-MS (EPA-CLP method)	5 µg/kg	NG	EPA 1987a
Liquid and solid waste	solid samples dispersed in a glycol, purge at ambient temperature, trap in Tenax/silica, desorb thermally	GC-HELD (EPA methods 5030 and 8010)	0.4 µg/L (groundwater) 0.4 µg/L (soil) 20 µg/Kg (liquid waste) 50 µg/kg (soil and sludge)	44-156%	EPA 1982b, EPA 1986b
Groundwater, solid waste, or sludge	sample disposed in a glycol, purged at ambient temperature, trapped in	GC-HELD and PID in Series	1-5 µg/g (for soil)	80% (groundwater)	Lopez-Avila et al. 1987

NG - not given; GC - gas chromatography; MS - mass spectrometry; HRGC - high resolution gas chromatography; HEED - hall electrolytic conductivity detector; PID - photoionization detector; ECD - electron capture detector; FID - flame ionization detector

sensitivity of detection. In addition, purge and whole column cryotrapping eliminates the need for the conventional purge and trap unit and reduces the time of analysis (Pankow and Rosen 1988). The plugging of the trap by the condensation of moisture during cryotrapping may be avoided by the use of very wide bore capillary column, although the chromatographic resolution of such a column is inferior to narrow bore capillary columns (Pankow and Rosen 1988; Mosesman et al. 1987).

6.3 ADEQUACY OF THE DATABASE

Section 104 (i) (5) of CERCLA, directs the Administrator of ATSDR (in consultation with the Administrator of EPA and agencies and programs of the Public Health Service) to assess whether adequate information on the health effects of 1,2-dichloropropane is available. Where adequate information is not available, ATSDR, in cooperation with the National Toxicology Program (NTP), is required to assure the initiation of a program of research designed to determine these health effects (and techniques for developing methods to determine such health effects). The following discussion highlights the availability, or absence, of exposure and toxicity information applicable to human health assessment. A statement of the relevance of identified data needs is also included. In a separate effort, ATSDR, in collaboration with NTP and EPA, will prioritize data needs across chemicals that have been profiled.

6.3.1 Data Needs

Methods for Determining Parent Compounds and Metabolites in Biological Materials. The analytical methods for determining volatile chlorinated hydrocarbon levels in biological matrices are quite general. However, there is a paucity of data specific to 1,2-dichloropropane. The limited number of publications that discuss the methods for the determination of this compound in biological matrices do not report either the recovery or the detection limit of the compound in different biological matrices. The study of the levels of the parent compound in human blood, urine or other biological matrices can be useful in deriving a correlation between the level of this compound found in the environment and those found in the body. One study (Ghittori et al. 1987) reported that a correlation exists between the urinary level and the TWA level of 1,2-dichloropropane measured at the breathing zone. No metabolite of 1,2-dichloropropane from human exposure to this compound has yet been identified, although specific metabolites have been identified in the urine of rats (see Subsection 2.6.3). The changes in metabolite concentrations with time in human blood, urine, or other appropriate biological medium may be useful in estimating its rate of metabolism in humans. In some instances, metabolite levels may be useful in correlating exposed doses to human body burdens. Such studies on the levels of metabolites in human biological matrices are not available for this compound.

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Methods for Biomarkers of Exposure. No biomarker of exposure to 1,2-dichloropropane has been identified (see Subsection 2.9.2). If a biomarker for this compound in a human biological tissue or fluid were available and 'a correlation were found to exist between the level of biomarker and a certain health effect, it could be used as an indication of a health effect caused by the exposure to this chemical.

Methods for Determining Parent Compounds and Degradation Products in Environmental Media. Analytical methods are available for the quantification of 1,2-dichloropropane in environmental samples. The levels of this compound in different environmental media can be used to indicate exposure of 1,2-dichloropropane to humans through the inhalation of air and ingestion of drinking water and foods containing 1,2-dichloropropane. If a correlation with human tissue or body fluid levels was found to exist, the intake levels from different environmental sources could be used to estimate the body burden of the chemical in humans. Although the products resulting from the biotic or abiotic degredation of 1,2-dichloropropane in the environment can be inferred, there has been no systematic study of the concentrations of these reaction products in the environment. In instances where the product(s) of an environmental reaction is more toxic than the parent compound, it is important that the level of the degradation products in the environment be known. No such reaction products have been identified for 1,2-dichloropropane. Analytical methods are available for the quantification of the known reaction products of 1,2-dichloropropane in the environment.

6.3.2 On-going Studies

The Environmental Health Laboratory Sciences Division of the Center for Environmental Health and Injury Control, Centers for Disease Control, is developing methods for the analysis of 1,2-dichloropropane and other volatile organic compounds in blood. These methods use purge and trap methodology and magnetic mass spectrometry which gives detection limits in the low parts per trillion range.