6. ANALYTICAL METHODS

The purpose of this chapter is to describe the analytical methods that are available for detecting and/or measuring and monitoring 1,2-dibromo-3-chloropropane in environmental media and in biological samples. The intent is not to provide an exhaustive list of analytical methods that could be used to detect and quantify 1,2-dibromo-3-chloropropane. Rather, the intention is to identify well-established methods that are used as the standard methods of analysis. Many of the analytical methods used to detect 1,2-dibromo-3-chloropropane in environmental samples are the methods approved by federal agencies such as EPA. Additionally, analytical methods are included that refine previously used methods to obtain lower detection limits, and/or to improve accuracy and precision.

6.1 BIOLOGICAL MATERIALS

Methods for analyzing 1,2-dibromo-3-chloropropane in biological samples are presented in Table 6-1. All of the methods listed utilize gas chromatography (GC) with various detectors. For most of the methods, detection limit and recovery data are not provided. No studies that reported the analysis of 1,2-dibromo-3-chloropropane in urine were located in the literature. With suitable modifications, the methods used for the determination of this chemical in water samples may be applicable for its determination in urine samples (Section 6.2).

6.2 ENVIRONMENTAL SAMPLES

Methods for analyzing 1,2-dibromo-3-chloropropane in environmental samples are presented in Table 6-2. As with the methods for the analysis of biological samples, all of the listed methods for the analysis of environmental samples utilize GC with various detection methods. The preconcentration/pretreatment methods use either adsorption onto a sorbent column for air samples, purge-and-trap methods for environmental water, soil, and solid samples, or simple extraction for food samples. The detection systems used, which include halogen-specific detection (e.g., Hall electrolytic conductivity detector), electron capture detector (ECD), and mass spectrometry (MS), generally provide excellent detection limits. An advantage of halogen-specific detectors is that they are not only very sensitive but also are specific to halogen compounds. The mass spectrometer, on the other hand, provides additional confirmation of a compound's identity through its ion fragment patterns. High-resolution gas chromatography (HRGC) with capillary columns provides better resolution for volatile compounds than packed columns. In this method, desorbed compounds are cryogenically trapped onto the head of the capillary column.

TABLE 6-1. Analytical Methods for Determining 1,2-Dibromo-3-chloropropane in Biological Materials

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Exhaled air	Exhaled air collected by valved Teflon® Spirometer mouthpieces into Teflon® bag; contents of bag sorbed in Tenax® and thermally desorbed	Cryofocusing HRGC-MS	No data	77%-96%	Wallace et al. 1986
Exhaled air	Sorb into Tenax® column; thermally desorb	GC-MS	No data	No data	Pellizzari et al. 1985a
Blood	Pass inert gas over warmed sample; adsorb in Tenax® cartridges; thermally desorb	GC-MS	Approximately 3 ng/mL (10 mL sample)	No data	Pellizzari et al. 1985b
Rat blood	Extract with toluene	GC-ECD	No data	92.6%-102.4% (mean 96.7%)	Kastl et al. 1981
Tissues	Sample suspended in water; warmed and pass inert gas; adsorb in Tenax® cartidges; thermally desorb	GC-MS	Approximately 6 ng/g for 5g tissue	No data	Pellizzari et al. 1985b

ECD = electron capture detector; GC = gas chromatography; HRGC = high resolution gas chromatography; MS = mass spectrometry

TABLE 6-2. Analytical Methods for Determining 1,2-Dibromo-3-chloropropane in Environmental Samples

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air	Adsorb in Tenax® column; thermally desorb	Cryofocusing HRGC-MS	No data	77%-96%	Wallace et al. 1986
Air	Adsorb onto activated charcoal; desorb with hexane	GC-ECD	No data	90%	Fredrickson et al. 1985
Air	Adsorb onto Chromosorb II®; desorb with toluene	GC-ECD	0.02 ppb	>90% for 0.07-20 ppm	Mann et al. 1980
Air	Adsorb onto coconut charcoal; desorb with bezene, methanol-benzene, or methanol-toluene	GC-ECD	No data	87.7%-93.9%	Albrecht et al. 1986
Finished drinking/ raw source water/ groundwater	Extract with hexane	GC-HECD	0.01 µg/L	94%-105% at 2.0 µg/L	EPA 1986a (EPA Method 504)
Finished drinking/ raw source water	Purge and trap in Tenax®/ silica/charcoal; thermally desorb	Subambient programmable HRGC-MS	1.8 µg/L	No data	EPA 1986a (EPA method 524.1)
Finished drinking/ raw source water	Purge and trap in Tenax®/ silica/charcoal; thermally desorb	Cryofocusing (wide or narrow bore) HRGC-MS	0.26 µg/L[a]	83% at 0.5-10 µg/L[a]	EPA 1986a
			0.50 µg/L[b]	92% at 0.5 µg/L[b]	EPA 1986a (EPA method 524.2)
Drinking water	Purge and trap in Tenax®/ silica/charcoal; thermally desorb	GC-HECD and PID in series	3.0 µg/L	86%	Ho 1989
Liquid and solid waste, groundwater, soil, and sludge	Soil and viscous samples dispersed in water or methanol/water; purge and trap in Tenax®/silica/charcoal and thermally desorb	GC-HECD	No data	No data	EPA 1986b; Garman et al. 1987 (EPA method 5030 and 8010)
Food	Extract composited, table-ready food with isooctane or acetone-isooctane	GC-ECD/HECD	5 ng/g (ECD); 95 ng/g (HECD)	45%-102%	Daft 1988, 1989

TABLE 6-2 (Continued)

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Citrus fruit (lemon, orange, grapefruit)	Sample blended with water; distilled into cyclohexane in essential oil apparatus; cleanup on Florisil column; injected into GC	GC-ECD	No data	96.5%-97.1% at 0.01 ppm	Tonogai et al. 1986

[a]Wide bore capillary column
[b]Narrow bore capillary column

ECD = electron capture detector; GC = gas chromotography; HECD = Hall electron capture detector; HRGC = high resolution gas chromatography; MS = mass spectrometry; PID = photoionization detector

6.3 ADEQUACY OF THE DATABASE

Section 104(i)(5) of CERCLA, as amended, 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-dibromo-3-chloropropane is available. Where adequate information is not available, ATSDR, in conjunction with the NTP, is required to assure the initiation of a program of research designed to determine the health effects (and techniques for developing methods to determine such health effects) of 1,2-dibromo-3-chloropropane.

The following categories of possible data needs have been identified by a joint team of scientists from ATSDR, NTP, and EPA. They are defined as substance-specific informational needs that, if met, would reduce or eliminate the uncertainties of human health assessment. In the future, the identified data needs will be evaluated and prioritized, and a substance-specific research agenda will be proposed.

6.3.1 Data Needs

Methods for Determining Biomarkers of Exposure and Effect. No biomarker that can be associated quantitatively with exposure to 1,2-dibromo-3-chloropropane has been identified (Moody et al. 1984; Suzuki and Lee 1981; Tofilon et al. 1980) (Section 2.5). If a biomarker in a human tissue or fluid were available, and a correlation between the level of the biomarker and exposure existed, it could be used as an indication of the levels and extent of exposure to this chemical. If exposure to bromine compounds is limited to 1,2-dibromo-3-chloropropane, serum bromide can be used as an indication of exposure (Torkelson et al. 1961). There are accurate techniques available for this analysis.

No biomarker of effect that can be associated quantitatively and directly attributed to 1,2-dibromo-3-chloropropane exposure has been identified (Whorton et al. 1979) (Section 2.5). If biomarkers of effect were available, and a correlation existed between the level or intensity of the biomarker of effect and the level of exposure, it could be used as an indication of the levels and extent of exposure to this chemical.

Methods for Determining Parent Compounds and Degradation Products in Environmental Media. Methods for the determination of 1,2-dibromo-3-chloropropane in environmental media are generally available (Albrecht et al. 1986; Daft 1988, 1989; EPA 1986a, 1986b; Fredrickson et al. 1985; Garman et al. 1987; Ho 1989; Kastl et al. 1981; Mann et al. 1980; Pellizzari et al. 1985a, 1985b; Tonogai et al. 1986; Wallace et al. 1986). Groundwater contaminated by leached 1,2-dibromo-3-chloropropane and air contaminated by volatilization of 1,2-dibromo-3-chloropropane from soil are the media of most concern for

potential human exposure. The precision, accuracy, reliability, and specificity of the methods for environmental waters are well documented and well suited for the determination of low levels of 1,2-dibromo-3-chloropropane and levels at which health effects occur; however, these data are lacking for the soil methods.

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-dibromo-3-chloropropane and other volatile organic compounds in blood. These methods use purge-and-trap methodology and magnetic sector mass spectrometry which give detection limits in the low parts per trillion (ppt) range.

No other on-going studies were located.