

6. ANALYTICAL METHODS

6.1 BIOLOGICAL MATERIALS

There is little need or opportunity to measure BCME in biological samples because of its rapid hydrolysis in water to yield formaldehyde and chloride. The abundance of chloride and, to a lesser extent, of formaldehyde, in biological materials precludes use of these hydrolysis products as an index of exposure to BCME. Therefore, the analysis of BCME in biological samples from exposed humans is virtually impossible.

6.2 ENVIRONMENTAL SAMPLES

Methods for the determination of BCME in environmental samples are currently confined to monitoring of air. It is possible, to monitor BCME in air at extremely low levels, and several analytical methods have detection limits of a few tenths of a ppb. Most methods for the analysis of BCME in air call for collecting samples on a solid adsorbent, followed by thermal desorption and gas chromatographic analysis. A typical procedure (ASTM 1987) uses a sampling tube packed with Chromosorb 101 adsorbent through which up to 25 L of air can be drawn for a period as long as 24 hours. For analyte determination, the sampling tube is attached to a gas chromatograph so that carrier gas can be passed through it and through the analytical column. With the carrier gas flowing, the collection column is heated to 150°C for four minutes and the analyte flows onto the analytical column, which is maintained at room temperature. Following desorption of the BCME, the analytical column is heated to 130°C with a programmed temperature increase and the eluted BCME is detected and measured quantitatively by mass spectrometry.

It has been noted (Travenius 1982) that common adsorption methods of sampling BCME from air are prone to giving inaccurate results because of hydrolysis of the analyte by coadsorbed water. For this reason, most procedures for methods involving collection of BCME on solid adsorbents require that samples in collection tubes be processed within a few days and protected from humidity.

Methods for determination of BCME in air are given in Table 6-1.

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TABLE 6-1. Analytical Methods for BCME in Environmental Media

Sample type	Extraction/cleanup	Detection	Limit of Detection	References
Air	Adsorption on Chromosorb 101, thermal desorption	GC/MS	<1 ppb	ASTM 1987
Air	Direct injection without preconcentration	GC/OEEC	<2 ppb	Kallos 1981
Air	Collect on Tenax GC, thermal desorption, cryofocussing	CCGC/MS	NR	Krost et al. 1982
Air	Collect on Porapak Q	GC/MS	<1 ppb	Muller et al. 1981
Air	NR	MS	0.1 ppb	Collier 1972
Air	Collect on Chromosorb 101	GC/MS	0.5 ppb	NIOSH 1977
Air	Impinger	GC/ECD	0.5 ppb	NIOSH 1977
Air	Collect on Porapak Q	Colorimetric	0.2 ppb	Norpoth et al. 1981

Abbreviations: CCGC, capillary column gas chromatography; ECD, electron capture detector; GC, gas chromatography; MS, mass spectrometry; NR, not reported; OEEC, oxygen-enhanced electron capture detector.

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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 BCME 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 Compound and Metabolites in Biological Materials. No methods were located for determining BCME in biological samples. It does not appear that this is a significant limitation, however, since BCME is not expected to endure in tissues or fluids. Although there are adequate methods for the detection of formaldehyde and chloride, these are not likely to be useful for assessing exposure to BCME, since any change in the levels of these compounds would be well within normal biological variability.

Methods for Biomarkers of Exposure. No methods were located for measuring any biomarkers of exposure to BCME. Although covalent adducts of BCME with cellular proteins or DNA have not yet been reported, development of sensitive and specific immunological assays for such adducts would provide a valuable means of detecting and perhaps quantifying human exposure levels.

Methods for Determining Parent Compound and Degradation Products in Environmental Media. Air is the only environmental medium susceptible to significant contamination by BCME and methods for the determination of this compound in air are straightforward. The greatest need for improvement in the analysis of BCME is the development of methodologies

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that enable its efficient collection from large volumes of air without hydrolysis during collection or storage. Since health concern might extend to concentrations well below 1 ppb, improvement in sensitivity would also be valuable.

6.3.2 Ongoing Studies

Because of evidence that BCME is carcinogenic even at very low levels in the atmosphere, current studies of its analysis are concentrating on extending the detection limit to even lower levels. Because of its chlorine content, BCME can be measured with extreme sensitivity by electron capture detection after gas chromatographic separation of this analyte. Efforts are underway by Dr. Robert Sievers (University of Colorado, Boulder) to improve collection methods that meet the criteria of (1) highly efficient collection of BCME at sub-ppb levels in air, (2) no loss of analyte from hydrolysis resulting from atmospheric humidity, and (3) rapid, efficient, nondestructive desorption of analyte from the collection medium.