

Characterization of mass-labeled [$^{13}\text{C}_{14}$]-decabromodiphenylethane and its use as a surrogate standard in the analysis of sewage sludge samples

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Abstract

Very little data is available about the presence of the brominated flame retardant, DBDPE, in the environment. This study reports the characterization of [$^{13}\text{C}_{14}$]-decabromodiphenylethane and the use of this surrogate standard to positively identify and quantify the presence of DBDPE in sewage sludge samples. The large difference in response factors between BDE-209 and DBDPE predicated the use of [$^{13}\text{C}_{14}$]-decabromodiphenylethane as a surrogate standard to improve the accuracy when determining the levels of DBDPE in environmental samples.

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1. Introduction

Decabromodiphenylethane (DBDPE) is a relatively new brominated flame retardant for use in polystyrene and polyolefin-based thermoplastic formulations. DBDPE was introduced in the early 90s, and is marketed as a non-diphenyl oxide-based alternative to brominated diphenyl ethers (BDE). One study (Kierkegaard and Bjorklund, 2003) has shown that DBDPE produced no dioxins and only minor quantities of 2,3,7,8-TBDF under pyrolysis conditions. Another study (Jakab et al., 2003) has shown the formation of bromotoluenes but no furans during thermal decomposition of DBDPE.

Although currently DBDPE is not as widely used as decabromodiphenyl ether (BDE-209) because of its higher cost, it is predicted that DBDPE will soon become one of the major flame retardants used by the thermoplastics industry. The presence of DBDPE in sediments, sewage sludge and air samples was first reported by Kierkegaard and Bjorklund (2003) and Kierkegaard et al. (2004). The levels of DBDPE found in sewage sludge ranged from 33 to approximately 100 ng/g dry weight, and its ratio to BDE-209 levels in quantified samples was 0.01 in the sediment and 0.3–0.6 in the sewage sludge samples examined (Kierkegaard et al., 2004).

The lack of a surrogate standard for DBDPE poses difficulties in determining levels accurately. Differences in extraction efficiency and response factors between BDE-209 and DBDPE can cause a significant bias in results. This study had two goals: the characterization of mass-labeled DBDPE and an assessment of its utility in accurately determining DBDPE levels in environmental samples.

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2. Materials and methods

2.1. Synthesis

Native and mass labeled [$^{13}\text{C}_{14}$]-decabromodiphenylethane (MDBDPE) were prepared using proprietary methods.

2.2. High resolution gas chromatography/low resolution mass spectrometry (HRGC/LRMS)

The analyses were conducted on a Shimadzu GCMS-QP2010 using a J&W 15 m DB-5HT column (0.25 mm ID, 0.1 μm film). The injections were done in splitless mode, with the injector temperature at 300 $^{\circ}\text{C}$, and helium carrier gas flow of 1.50 ml/min. The following temperature program was used: initial oven temperature: 140 $^{\circ}\text{C}$, hold of 5 min, ramp at 10 $^{\circ}\text{C}/\text{min}$ to 325 $^{\circ}\text{C}$, hold 20 min. A full scan range of 50–1000 amu was used.

2.3. High resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS)

The analyses were conducted on a HP6890 HRGC coupled to a Waters Autospec Ultima HRMS. The GC column was a J&W 15 m DB-5HT (0.25 mm ID, 0.1 μm film). The injections were done in splitless mode, with the injector temperature at 300 $^{\circ}\text{C}$. The initial helium carrier gas flow was 10 ml/min for 1 min, then decreased at 500 ml/min 2 to 1.0 ml/min. The following temperature program was used: initial oven temperature: 140 $^{\circ}\text{C}$, hold of 5 min, ramp at 10 $^{\circ}\text{C}/\text{min}$ to 330 $^{\circ}\text{C}$, hold until after DBDPE eluted. The mass spectrometer was operated in the selected ion monitoring (SIM) mode at a resolution of at least 10000. The transfer line and source were maintained at 300 $^{\circ}\text{C}$. Additional analyses were carried out on a HP5890 HRGC coupled to a VG70SE HRMS with the same column and instrumental conditions.

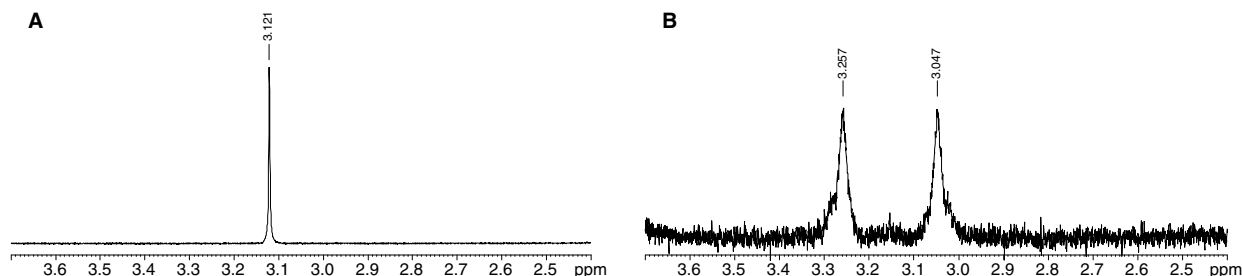


Fig. 1. ^1H NMR spectra of (A) DBDPE, (B) MDBDPE at 400 MHz.

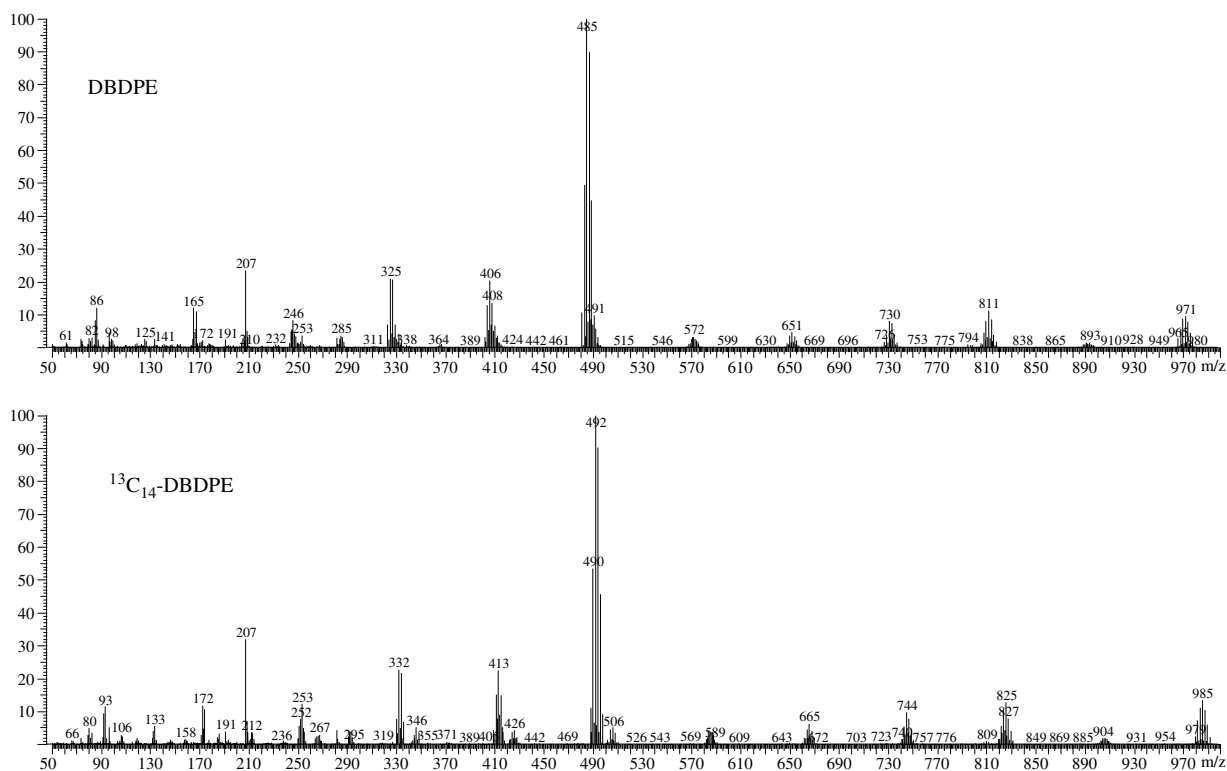


Fig. 2. Full-scan mass spectra of DBDPE and MDBDPE (LRMS).

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