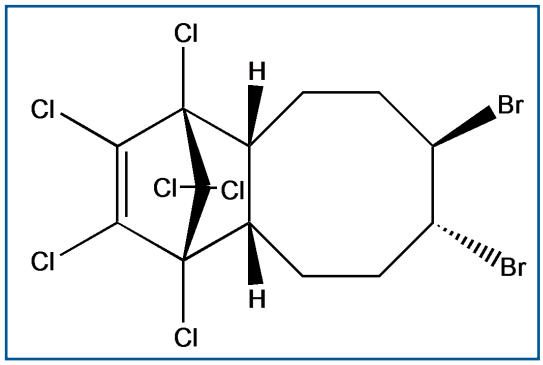
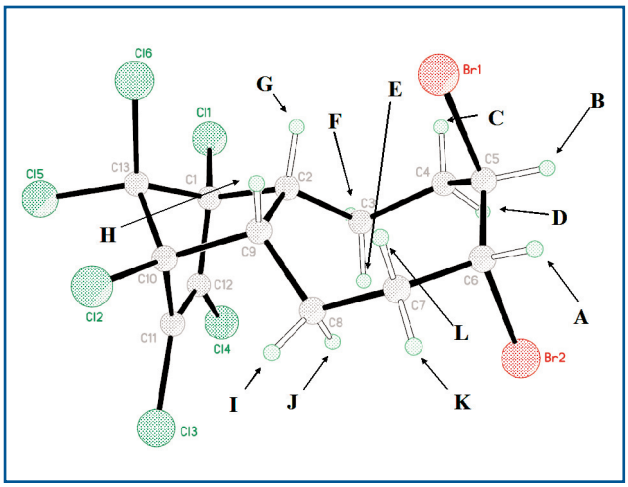
November 7, 2008

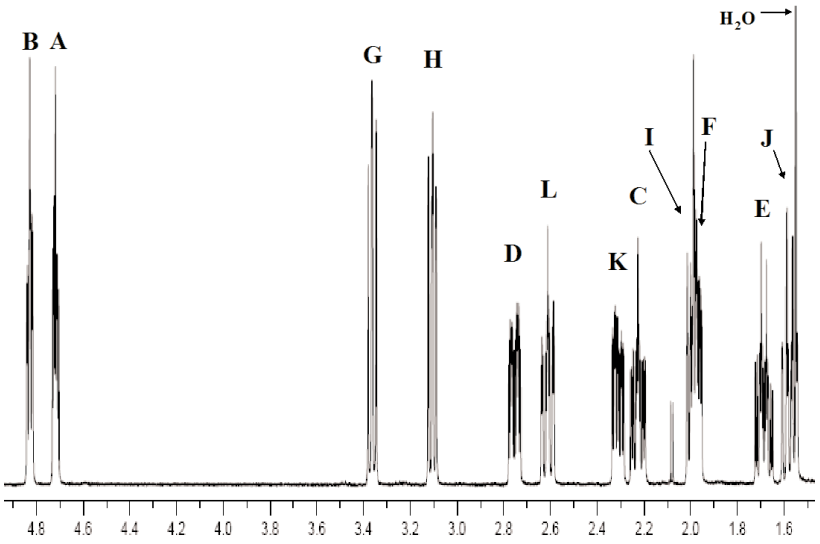
Structural Characterization of Hexachlorocyclopentenyldibromocyclooctane (HCDBCO)



HCDBCO is a commercial brominated flame retardant (BFR) used in styrenic polymers and recent work has identified it as a component in house dust. We have completed ^{1}H NMR spectroscopic studies and an x-ray structure determination to establish the conformational structure of HCDBCO in the solid state and in solution. This information should be useful to researchers wishing to model and predict the physiochemical behaviour of HCDBCO in the environment.

The ^{1}H NMR spectrum of HCDBCO is shown below. An ^{1}H-^{13}C HSQC NMR experiment allowed us to assign the pair of protons on each of the CH_2 moieties and those on the CHBr and CH moieties. An ^{1}H-^{1}H COSY experiment allowed identification of the proton sequence around the structure as: A-B-(C/D)-(E/F)-G-H-(I/J)-(K/L)-A. Most of the $3J(^{1}H\text{-}^{1}H)$ and the $2J(^{1}H\text{-}^{1}H)$ coupling constants were determined using a simulation program to help disentangle the complex second order spectra. Protons H_A and H_B are clearly those of the CHBr moieties as they would be expected to appear at lower field relative to the other protons. The measured dihedral angles from the x-ray structure correlate very well with those calculated from the proton-proton coupling constants indicating that the conformations in solution and in the solid state are very similar.





HCDBCO debrominates in the injector port at 250°C. Reducing the temperature to 200°C eliminated this problem. HCDBCO only exists as a pair of enantiomers, therefore, thermal rearrangement as seen for other cycloaliphatic polybrominated compounds such as HBCD and TBECH is not a problem when analyzing for HCDBCO by GC/MS. Nevertheless, thermal rearrangement may still exist which could create problems when determining the enantiomeric composition using a chiral column.