We developed a straightforward synthesis of a series of ten PCB sulfate monoesters from the corresponding hydroxylated PCBs for Project #3. The hydroxylated PCBs were synthesized by coupling chlorinated benzene boronic acids with appropriate brominated (chloro-)anisoles, followed by demethylation with boron tribromide. The hydroxylated PCBs were sulfated with 2,2,2-trichloroethyl chlorosulfate using DMAP as base. Deprotection with zinc powder/ammonium formate yielded the ammonium salts of the desired PCB sulfate monoesters in good yields. These compounds are now available to study their chemical stability, physicochemical properties and interaction with biological targets, such as albumin and other proteins (Li et al., Environ. Int., 2009).