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Hexabromocyclododecane(HBCD)and Tetrabromobisphenol-A (TBBPA), in Sharks and Dolphins from Florida Coastal Waters

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Top predators including bottlenose dolphin (*Tursiops truncatus*), bull shark (*Carcharhinus leucas*), and Atlantic sharpnose shark (*Rhizoprionodon terraenovae*) collected along the west and east coast of Florida between 1991 and 2004 were analyzed for tetrabromobisphenol-A (TBBPA) and hexabromocyclododecane (HBCD). Tissues of blubber from bottlenose dolphin and muscle from shark species were extracted in a Soxhlet apparatus; lipid was removed using a gel permeation chromatography column and sulfuric acid. The extracts were concentrated to dryness and reconstituted with 100 μ l of methanol, and then analyzed for HBCDs and TBBPA by a liquid chromatograph tandem mass spectrometry (LC-MS/MS) operated in electrospray negative ionization mode. TBBPA concentrations varied from 0.1 ng/g, lipid wt in bottlenose dolphin to 35.6 ng/g, lipid wt in bull shark. Mean \pm SD concentrations of TBBPA were 1.5 ± 2.9 ng/g, lipid wt for bottlenose dolphin, 9.5 ± 11.8 ng/g, lipid wt for bull shark, and 0.9 ± 0.5 for Atlantic sharpnose shark. Extracts were injected onto LC-MS/MS to separate α -, β -, and γ -HBCD isomers by C_{18} column (100x2.1 mm I.D., 5 μ m particle size) with 10 mM ammonium acetate/methanol mobile phase. Sum of HBCD isomer concentrations ranged from 0.5 ng/g, lipid wt for bottlenose dolphin to 413 ng/g, lipid wt for bull shark. Mean concentrations of total HBCDs were 7.4 ± 18.5 , 78 ± 128 , and 55 ± 88 for bottlenose dolphin, bull shark, and Atlantic sharpnose shark respectively. In addition, the temporal trends of HBCD and TBBPA concentrations for bottlenose dolphin and bull shark species collected between 1991 and 2004 were also evaluated.