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In order to develop more robust insight into the natural attenuation of polychlorinated biphenyls (PCBs), the CI isotopic compn. of residual 2,3,4,5-tetrachlorobiphenyl (2,3,4,5-CB) was monitored as it underwent microbial reductive dechlorination to 2,3,5-trichlorobiphenyl (2,3,5-CB) in lab. cultures. Reverse-phase HPLC was used to isolate the former compd. from the exptl. matrix for d37Cl measurement. No detectable isotopic fractionation was obsd. over the 90-day incubation with sterile control, std., and inoculated samples all exhibiting d37Cl values with a range of .apprx.0.5.permill.. These results show that this type of biol. activity can be discriminated from other transformations by the absence of a measurable isotope effect during microbial reductive dechlorination. The utility of HPLC isolation for compd.-specific d37Cl analyses of environmentally relevant species is also demonstrated. No Cl isotopic fractionation was obsd. for the microbial reductive dechlorination of 2,3,4,5-CB.