Several pentapeptides selected through combinatorial chemistry to selectively bind dioxin derivatives, were synthesized and labeled with fluorescent marker at the C-end. The peptides were incorporated into a polyaniline matrix via glutaraldehyde, or by in-situ chemical polymerization of aniline. The stability of the reagent was evaluated at different pH and leaching was observed only in strongly acidic or basic medium. The peptide binding was tested in organic solution by the fluorescent changes of the ligand in presence of a toxin. Solid state extraction experiments were performed by passing a toxin-spiked water sample through the solid reagent, followed by elution with organic solvent and GC/MS analysis of both the water and organic phases.