

STIR BAR SORPTIVE EXTRACTION FOR THE DETERMINATION OF PBBS AND PBDES IN WATER SAMPLES

PRIETO A., USOBIAGA A., ZULOAGA O., ETXEBARRIA N., FERNÁNDEZ L.A.

Kimika Analitiko Saila, Euskal Herriko Unibertsitatea, 644, P.K., 48080, Bilbao, Basque Country; Tel. +34 94 601 5550; e-mail: qabprsoa@lg.ehu.es

Stir bar sorptive extraction (SBSE) was optimised for the pre-concentration of polybrominated biphenyls (PBB#7, PBB#31, PBB#103, PBB#153) and polybrominated diphenyl ethers (PBDEs, 2,4,4'-tribromodiphenylether (BDE#28); 2,2',4,4'-tetrabromodiphenylether (BDE#47); 2,3',4,4'-tetrabromodiphenylether (BDE#66); 2,2',3,4,4'-pentabromodiphenylether (BDE#85); 2,2',4,4',5-pentabromodiphenylether (BDE#99); 2,2',4,4',6-pentabromodiphenylether (BDE#100), 2,2',3,4,4',5'-hexabromodiphenylether (BDE#138); 2,2',4,4',5,6'-hexabromodiphenylether (BDE#153); 2,2',4,4',5,5'-hexabromodiphenylether (BDE#154)) in water samples. Optimisation was performed using spiked synthetic water samples. The variables studied during the optimisation process were: sample volume (5-100 mL), sodium chloride addition (0%-30%), and methanol addition (0%-20%), desorption time (4-10 min), desorption temperature (250-300°C), desorption flow (50-100 ml min⁻¹), cryo-focusing temperature (-150°C-+40°C) and vent pressure (0-12.8 psi). All those variables were studied using experimental design approaches by means of the Unscrambler® program. In all cases 0.5 mm x 10 mm (fill thickness and length) stir bars were used. Once all those variables were optimised, the extraction time profile (2-24 hours) was studied. The limits of detection, accuracy and within and among day precision were studied.

ACKNOWLEDGMENTS: This work was supported by the Ministry of Science and Technology (MCYT) of the Spanish Government through the Research Project No.: REN2002-01441.