

Supelclean™ Sulfoxide SPE Product Line

Description

Supelclean Sulfoxide SPE consists of a silica-bonded sulfoxide (-SO) phase. The technology was specifically developed for the extraction of polychlorinated biphenyls (PCBs) from transformer, waste and mineral oil. PCB retention is facilitated via interaction between the SPE phase's electrophilic sulfur atom and the π -electron cloud formed from aromatic rings inherent with PCBs. This unique SPE phase offers a simple and efficient sample prep method for identifying PCBs at quantitation limits of 0.5 ppm.

Recommended generic protocol

The generic protocols described below offer users a starting point for method development. Sample prep can be conducted via negative pressure (SPE vacuum manifold) or gravity. When processing SPE samples via gravity, attachment of a disposable PTFE liner (Cat. No. 57059) to the SPE luer tip is recommended for improving flow rate speed and consistency.

For additional technical support, please contact Supelco Technical Service at 800-359-3041 or e-mail: techservice@sial.com

SPE Phase:	Supelclean Sulfoxide Glass SPE, 6 g/20 mL (Cat. No. 55252-U)
1. Sample Pre-Treatment	Dilute oil sample 1:1 (v/v) with hexane and spike sample with internal standard PCB congener – ^{13}C (0.5 ppm) as necessary
2. Condition/Equilibration	Condition SPE phase with 20 mL acetone (to remove residual moisture); and equilibrate with 40 mL of hexane
3. Sample Load	Layer 250 μL of pre-treated sample (from step 1) onto the upper frit of the SPE tube. Rinse the inner wall of the SPE tube 2 x 0.5 mL hexane to further drive the oil sample into the packed bed.
4. Wash	Once the hexane solvent (applied in sample load (step 3)) has cleared the upper frit of the packed bed, wash the SPE tube with 11 mL hexane (for subsequent GC-HRMS) or 12 mL (for subsequent GC-QMS). As the hexane wash solvent passes through the cartridge, PCBs are preferentially retained/retarded on the SPE phase whereas endogenous sample interferences (e.g., long chain hydrocarbons) are eluted from the phase.
5. Elution	Upon complete elution of the hexane wash solvent (step 4), elute PCBs with 25 mL hexane and collect for subsequent analysis.

6. Evaporation/ Reconstitution	Evaporate 25 mL eluate containing PCBs under nitrogen, and adjust to 250 µL with hexane.
7. Analysis	GC-HRMS or GC-QMS

SPE Phase:	Supelclean Sulfoxide PP SPE, 3 g/6 mL (Cat. No. 55253-U)
1. Sample Pre-Treatment	Dilute oil sample 1:1 (v/v) with hexane and spike sample with internal standard PCB congener – ¹³ C (0.5 ppm) as necessary
2. Condition/Equilibration	Condition SPE phase with 10 mL acetone (to remove residual moisture); and equilibrate with 20 mL of hexane
3. Sample Load	Layer 250 µL of pre-treated sample (from step 1) onto the upper frit of the SPE tube. Rinse the inner wall of the SPE tube 2 x 0.5 mL hexane to further drive the oil sample into the packed bed.
4. Wash	Once the hexane solvent applied in sample load (step 3) has cleared the upper frit of the packed bed, wash the SPE tube with 5 mL hexane (for subsequent GC-HRMS) or 5.5 mL (for subsequent GC-QMS). As the hexane wash solvent passes through the cartridge, PCBs are preferentially retained/retarded on the SPE phase whereas endogenous sample interferences (e.g., long chain hydrocarbons) are eluted from the phase.
5. Elution	Upon complete elution of the hexane wash solvent (step 4), elute PCBs with 13 mL hexane and collect for subsequent analysis.
6. Evaporation/ Reconstitution	Evaporate 13 mL eluate containing PCBs under nitrogen, and adjust to 250 µL with hexane.
7. Analysis	GC-HRMS or GC-QMS

Product Information

Description	Qty.	Catalog No.
Supelclean Sulfoxide Glass SPE Tube, 6 g/20 mL	5	55252-U
Supelclean Sulfoxide SPE, 3 g/6 mL	30	55253-U
Supelclean Sulfoxide, Bulk	100 g	55254-U
Empty Glass SPE Tube (17 mm I.D. x 137 mm L) with PE frit, 20 mL, with PE frit, luer cap, and screw-top cap	5	55255-U
Frit Insertion Tool for 20 mL Glass SPE tube	1	55257-U
Disposable PTFE liners	100	57059
Large volume reservoir (25 mL) for 6 mL SPE tubes, PP	30	54258-U
Large volume reservoir (25 mL) for 6 mL SPE tubes, PTFE	3	54259-U