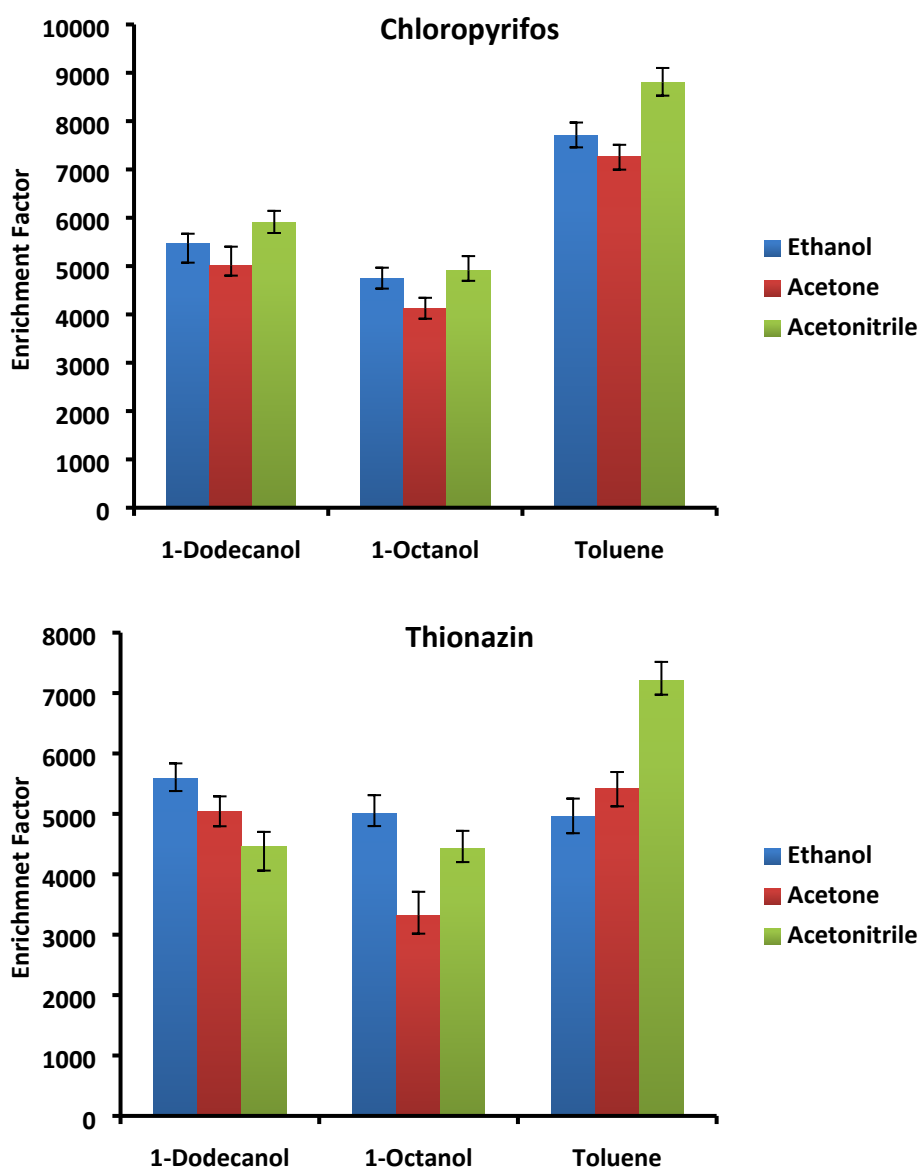**Fig. S1.** Selection of extracting and disperser/eluting solvents in USAL-SPE-LDS-DLLME. USL conditions: soil sample weight, 2 gr; spiked concentration, 50 ng g^{-1}; leaching solvent volume, 2 mL (Methanol); ultrasound radiation time, 2 min; centrifugation time, 5 min; SPE Conditions: water sample volume, 50 mL; flow rate, 15 mL min^{-1}; disperser/eluting solvent volume, 1 mL; LDS-DLLME conditions: extraction solvent volume, 10µL; aqueous solution volume, 5 mL; extraction time, 1 min; centrifugation time, 2 min.
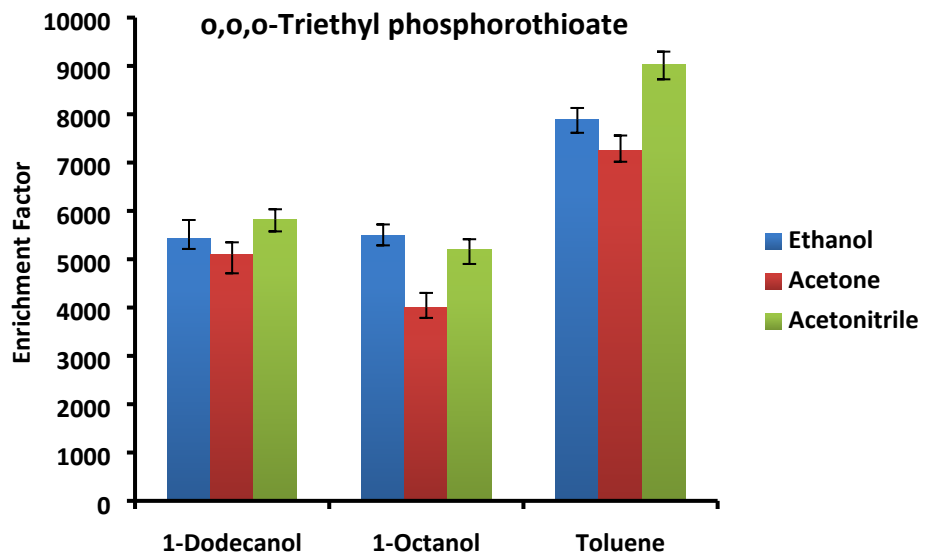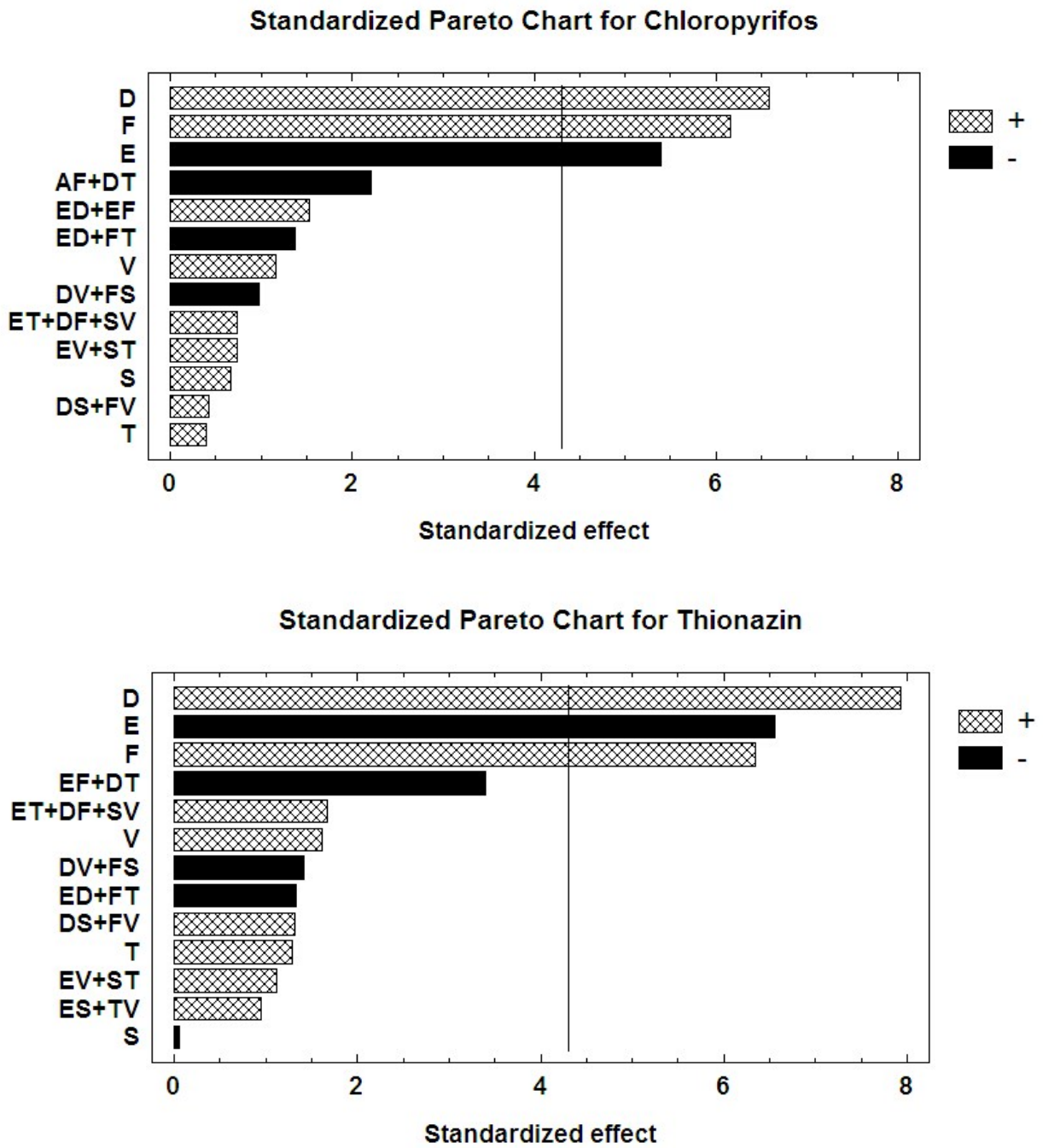
o,o,o-Triethyl phosphorothioate

Legend:
- Ethanol
- Acetone
- Acetonitrile

Y-axis: Enrichment Factor (0 to 10000)

X-axis categories: 1-Dodecanol, 1-Octanol, Toluene

Fig. S2. Standardized (P = 0.05) Pareto chart, representing the estimated effects of parameters and parameter interactions on enrichment factor
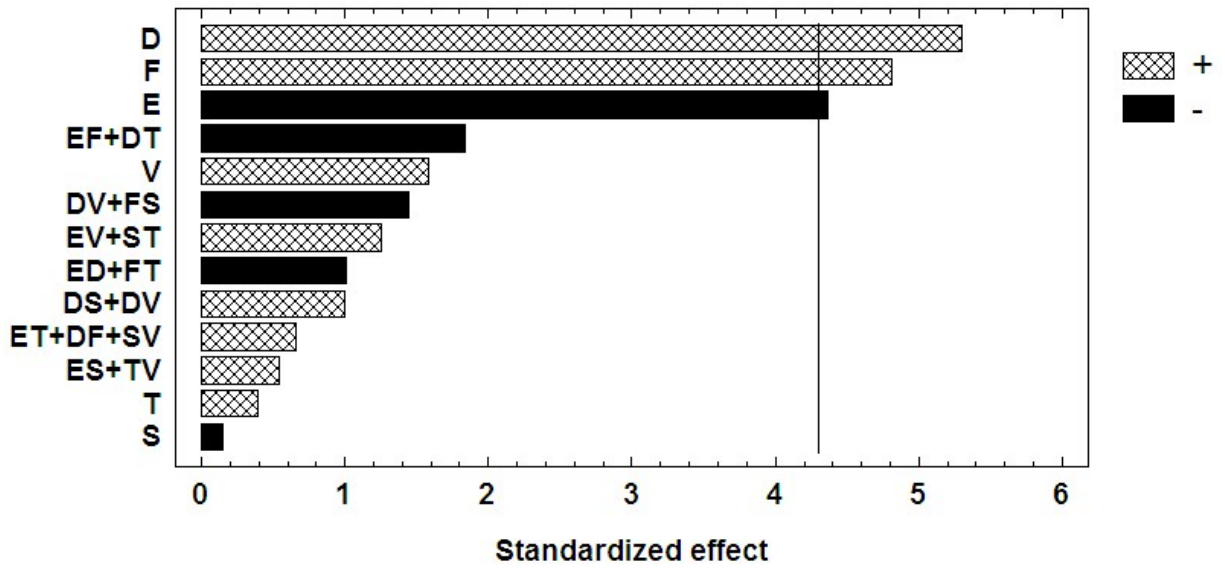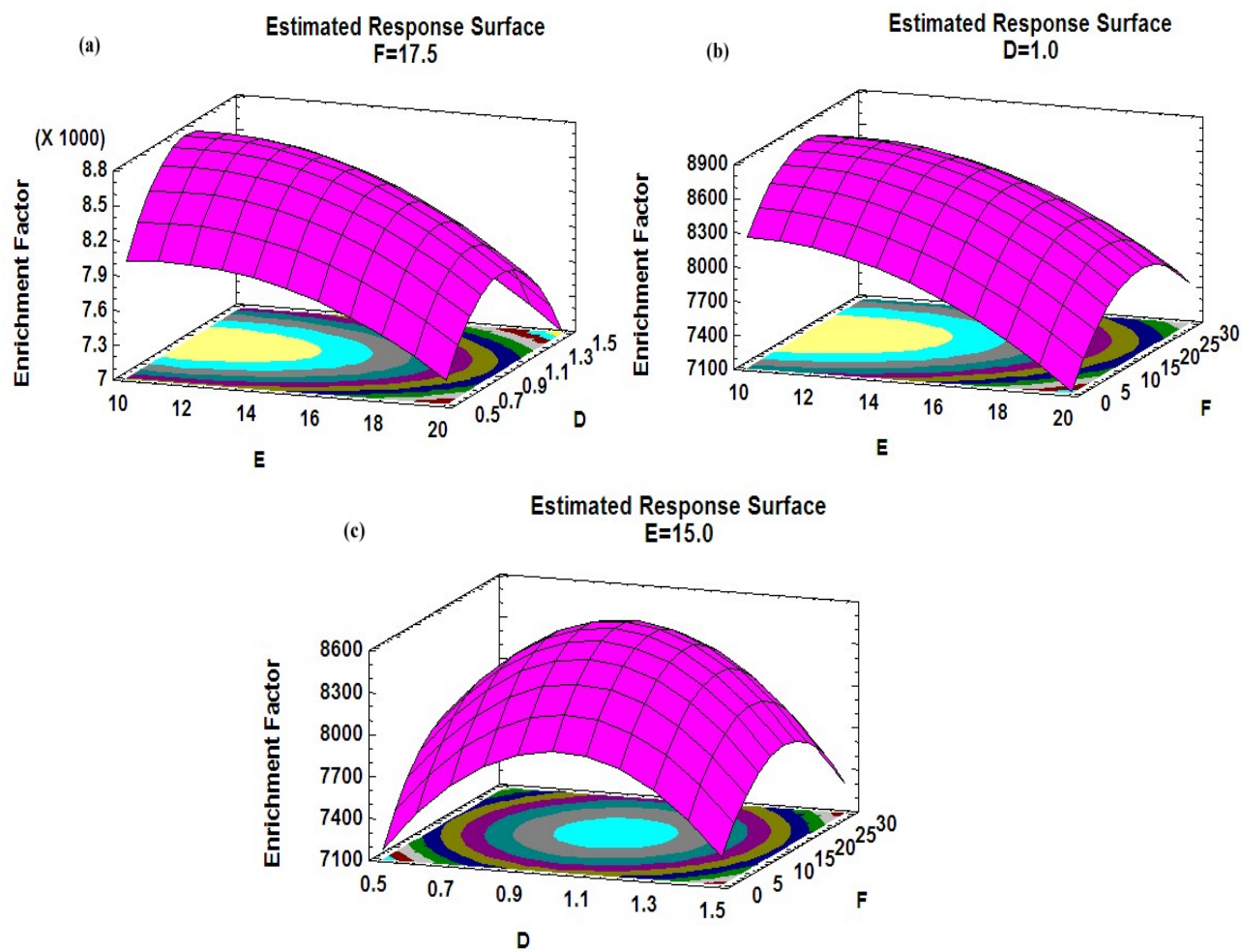

Standardized Pareto Chart for Chloropyrifos

Standardized Pareto Chart for Thionazin

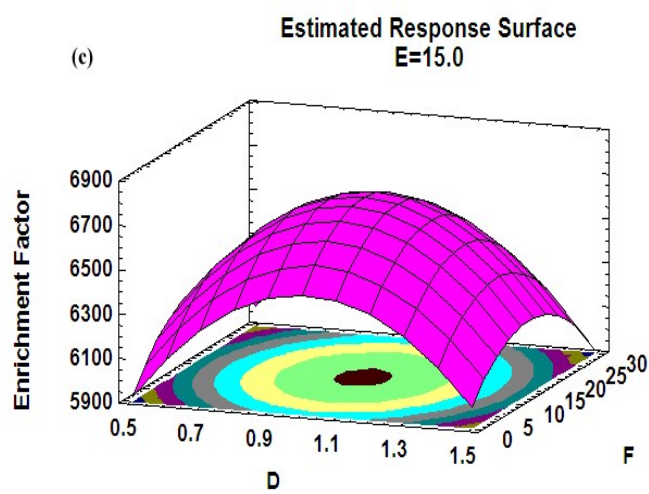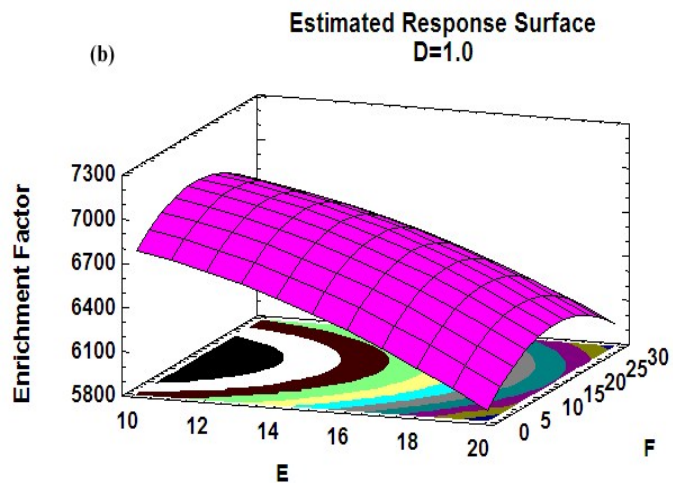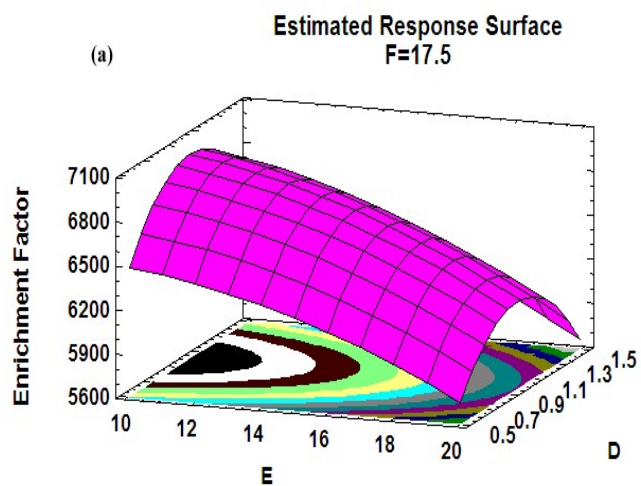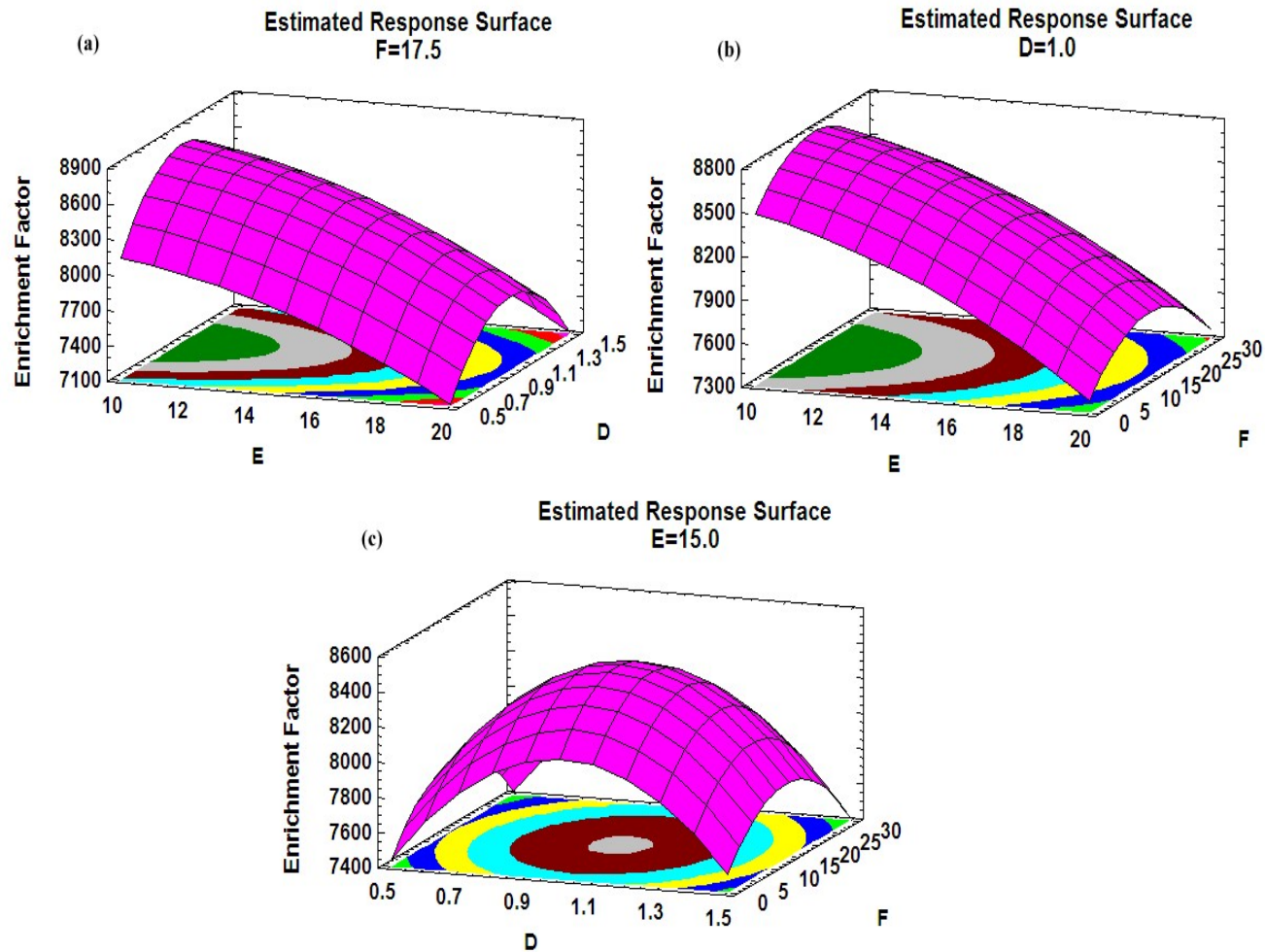## Standardized Pareto Chart for o,o,o-Triethyl phosphorothioate

Fig. S3. Estimated response surfaces with related contours for (A) Chloropyrifos, (B) Thionazin and (C) o,o,o-Triethyl phosphorothioate by plotting enrichment factor versus a: extraction solvent volume (E, μL) and dispersive solvent volume (D, mL); b: extraction solvent volume (E) and Flow rate of sample solution through solid phase (F, mL min^{-1}); c: dispersive solvent volume (D) and Flow rate of sample solution through solid phase (F);

(A) Chloropyrifos

(a) Estimated Response Surface F=17.5

(b) Estimated Response Surface D=1.0

(c) Estimated Response Surface E=15.0

(B) Thionazin

Estimated Response Surface
F=17.5

Estimated Response Surface
D=1.0

Estimated Response Surface
E=15.0

(C) o,o,o-Triethyl phosphorothioate