Supplementary Information (SI) for Analyst.
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Supporting Information

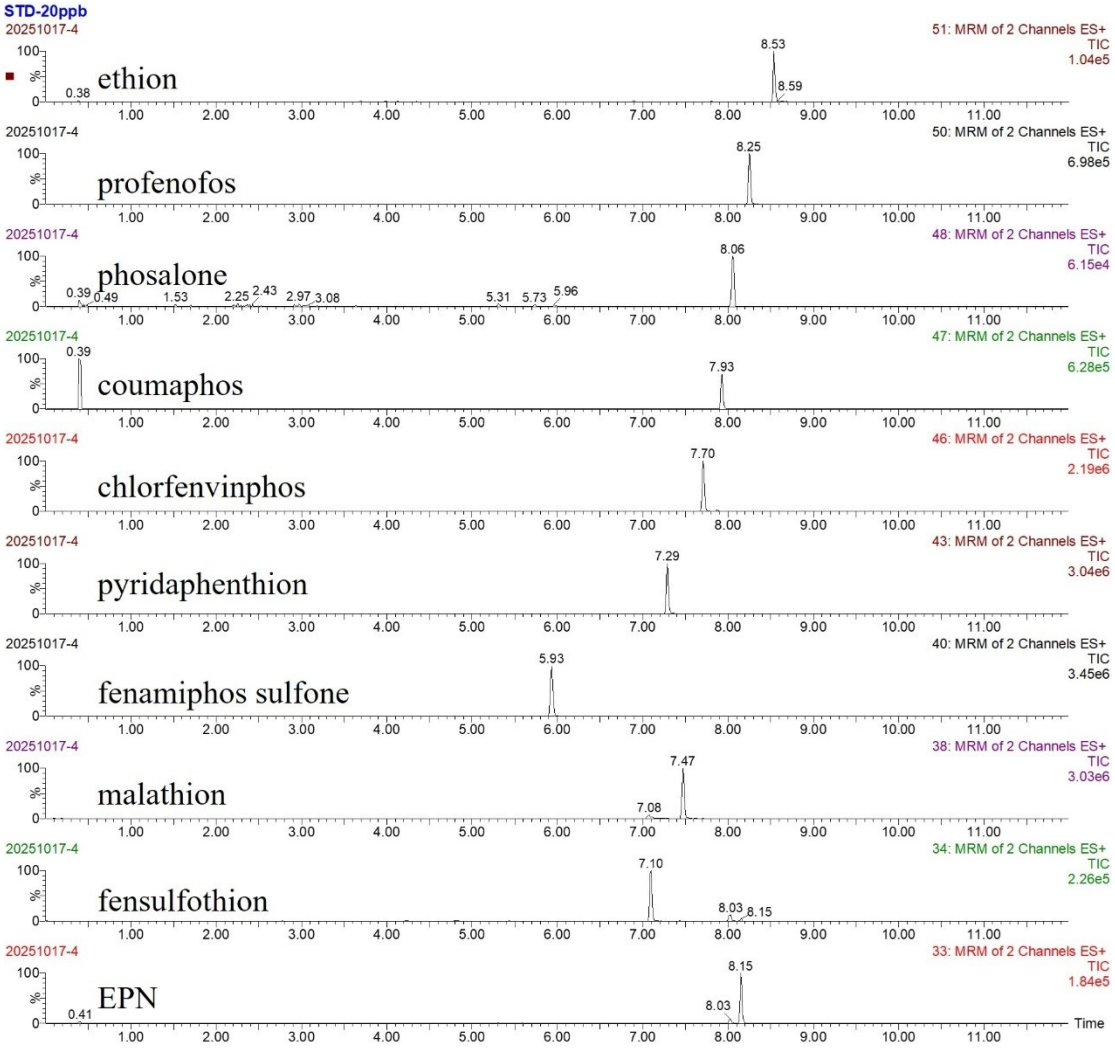
Supplementary methods

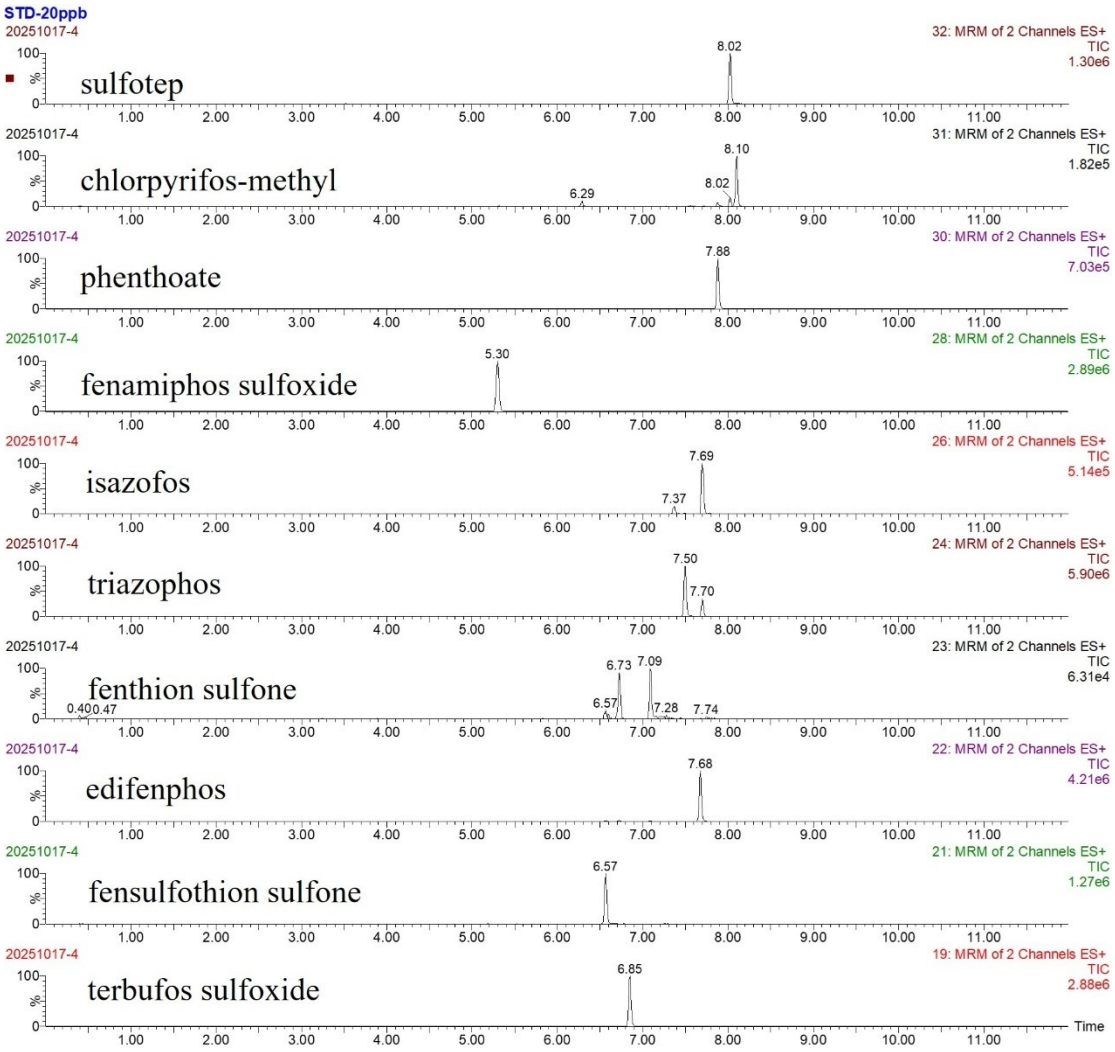
UPLC-MS/MS analysis

We studied the LC-MS/MS detection methods for cadusafos, chlorfenvinphos, chlorpyrifos methyl, coumaphos, disulfoton sulfoxide, edifenphos, EPN, ethion, ethoprophos, fenamiphos sulfone, fenamiphos sulfoxide, fensulfothion, fensulfothion sulfone, fenthion sulfone, fenthion sulfoxide, heptenophos, isazofos, malathion, methiocarb sulfoxide, parathion, phenthoate, phorate sulfoxide, phosalone, phoxim, profenofos, pyridaphenthion, quinalphos, sulfotep, terbufos sulfoxide and triazophos. Figure S1 shows the chromatogram of phenylpyrazoles under simultaneous positive and negative multiple reaction monitoring mode (MRM). Table S1 shows the chromatographic conditions for the detection of organophosphate pesticides.

The mass spectrometer used a Z-spray ESI. In positive mode, the MS parameters were the following: capillary voltage, 3.00 kV; source temperature, 150 ℃; desolvation temperature, 300℃; desolvation gas flow, 650 L/h; cone gas flow, 150 L/h; and collision gas 0.16 mL/min. In negative mode, the MS parameters were the following, capillary voltage, 2.80 kV; source temperature, 150 ℃; desolvation temperature, 300 ℃; desolvation gas flow, 600 L/h; cone gas flow, 150 L/h; and collision gas, 0.15 mL/min.

Supplementary figures




STD-20ppb
20251017-4
32: MRM of 2 Channels ES+
TIC
1.30e6
8.02
sulfotep
20251017-4
31: MRM of 2 Channels ES+
TIC
1.82e5
8.10
8.02
6.29
chlorpyrifos-methyl
20251017-4
30: MRM of 2 Channels ES+
TIC
7.03e5
7.88
phenthoate
20251017-4
28: MRM of 2 Channels ES+
TIC
2.89e6
5.30
fenamiphos sulfoxide
20251017-4
26: MRM of 2 Channels ES+
TIC
5.14e5
7.69
7.37
isazofos
20251017-4
24: MRM of 2 Channels ES+
TIC
5.90e6
7.50
7.70
triazophos
20251017-4
23: MRM of 2 Channels ES+
TIC
6.31e4
6.73
7.09
6.57
7.28
7.74
0.40
0.47
fenthion sulfone
20251017-4
22: MRM of 2 Channels ES+
TIC
4.21e6
7.68
edifenphos
20251017-4
21: MRM of 2 Channels ES+
TIC
1.27e6
6.57
fensulfothion sulfone
20251017-4
19: MRM of 2 Channels ES+
TIC
2.88e6
6.85
terbufos sulfoxide
%
Time
1.00
2.00
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00

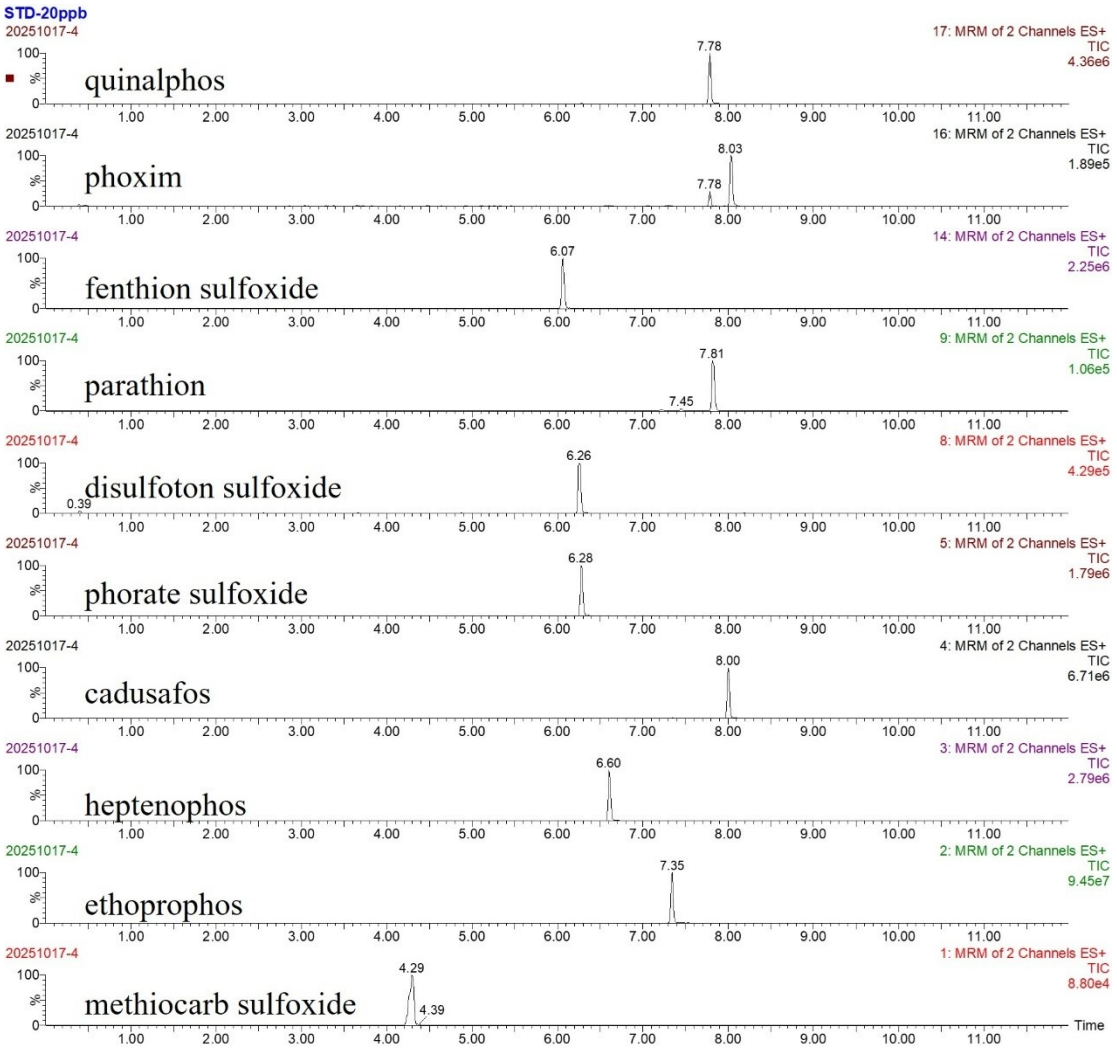
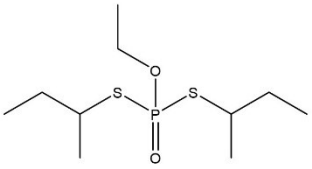
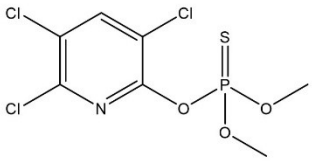


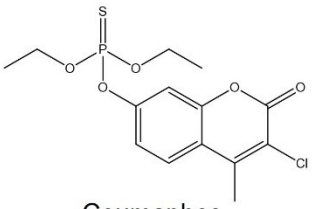
Figure S1. UPLC-MS/MS Chromatograms of MRM Modes for Thirty Organophosphorus Pesticides.



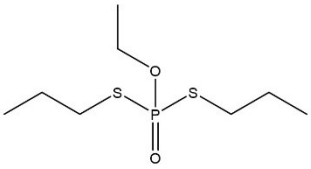
Cadusafos



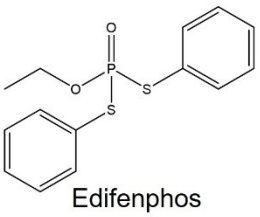
Chlorpyrifos methyl



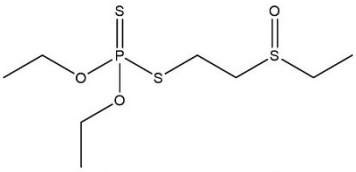
Coumaphos



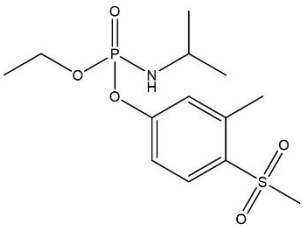
Ethoprophos



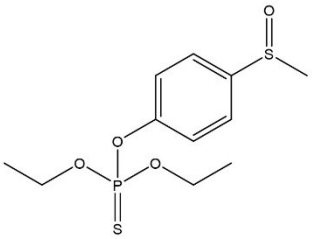
Edifenphos



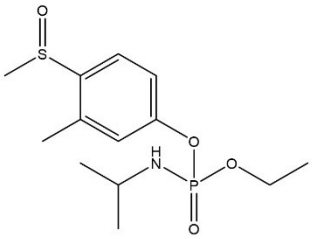
Disulfoton sulfoxide



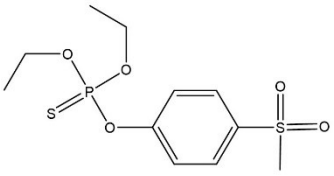
Fenamiphos sulfone



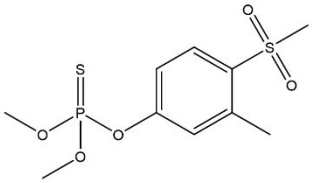
Fensulfothion



Fenamiphos sulfoxide



Fensulfothion sulfone



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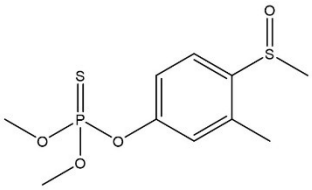
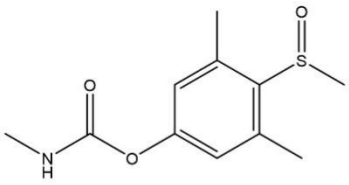
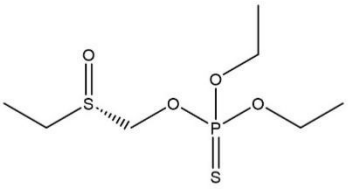


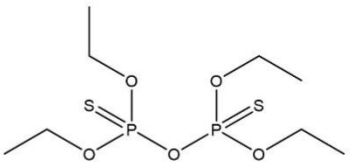
Fenthion sulfoxide



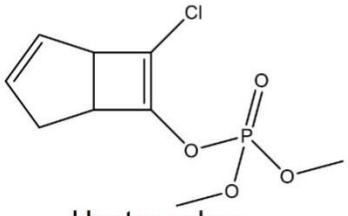
Methiocarb sulfoxide



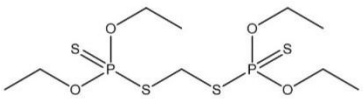
Phorate sulfoxide



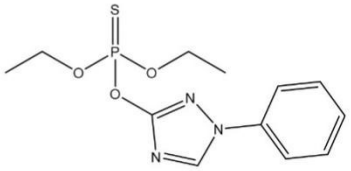
Sulfotep



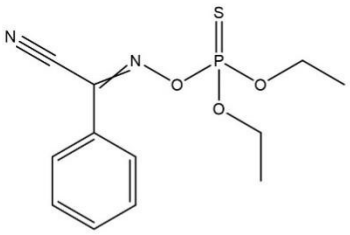
Heptenophos



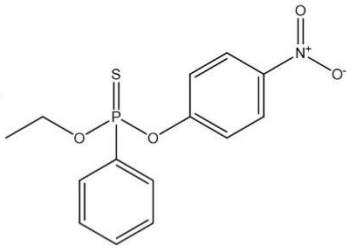
Ethion



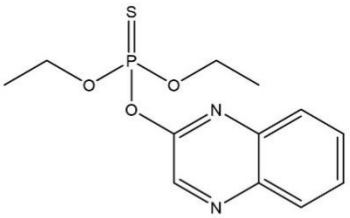
Triazophos



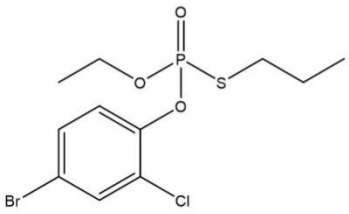
Phoxim



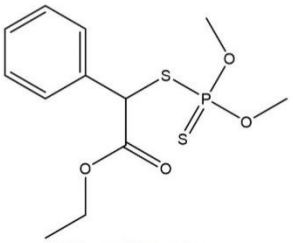
EPN



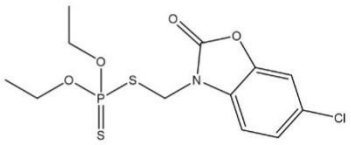
Quinalphos



Profenofos



Phenthoate



Phosalone

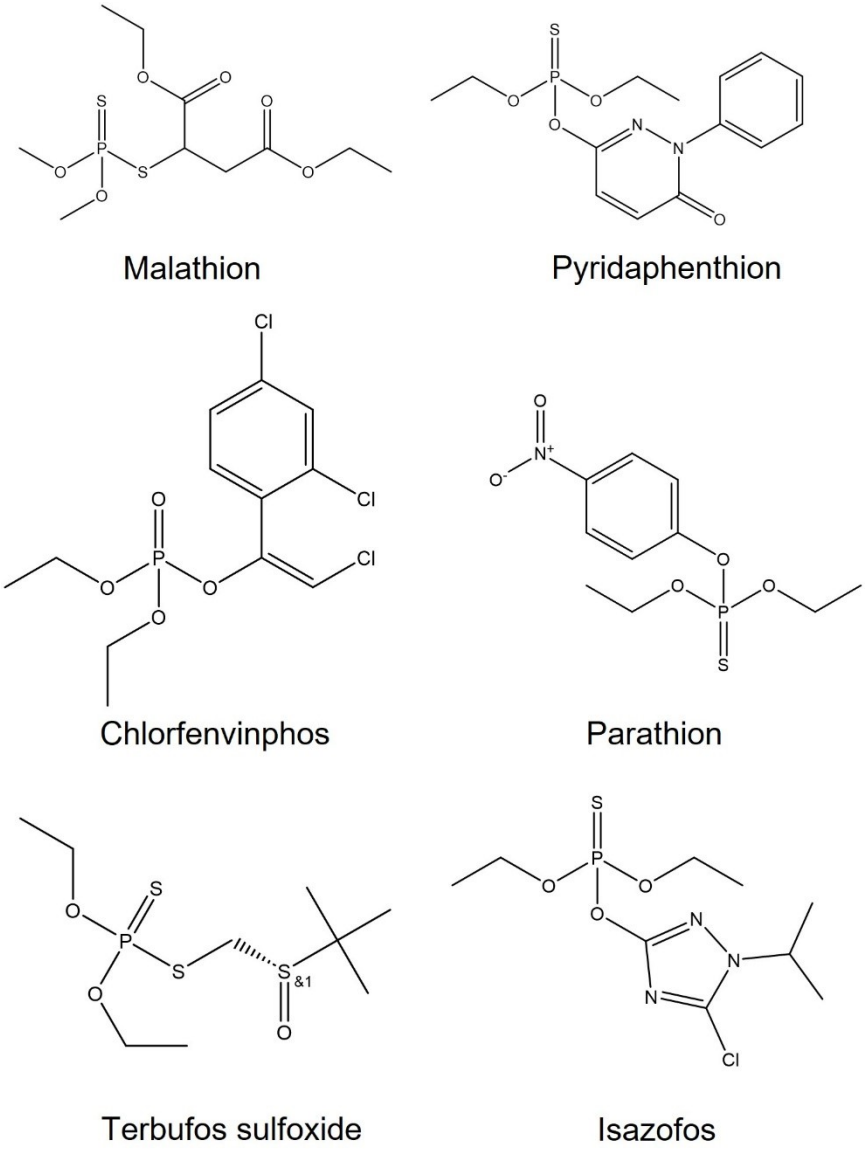


Figure S2. Chemical Structures of Thirty Organophosphorus Pesticides.

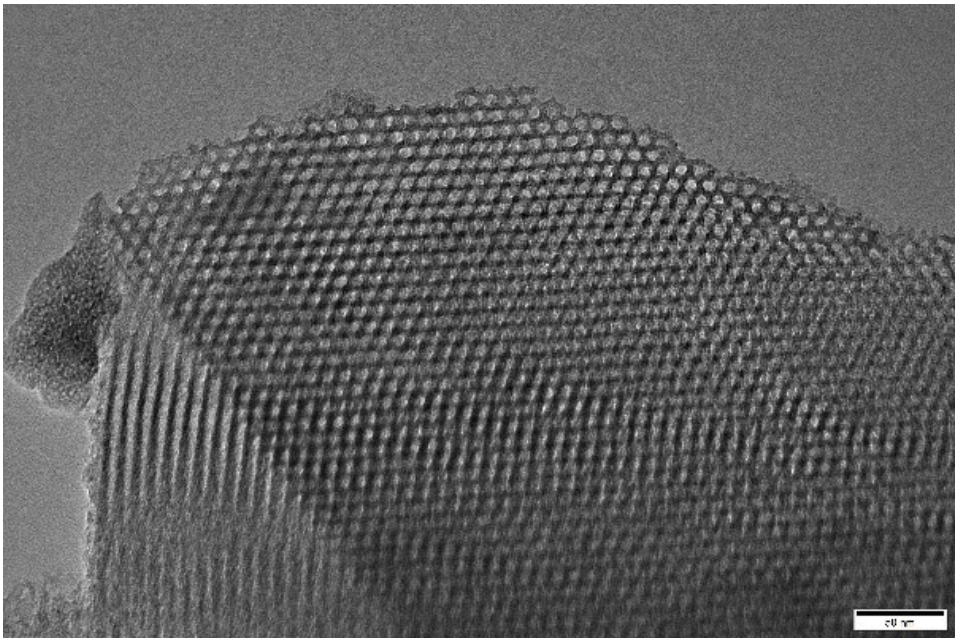
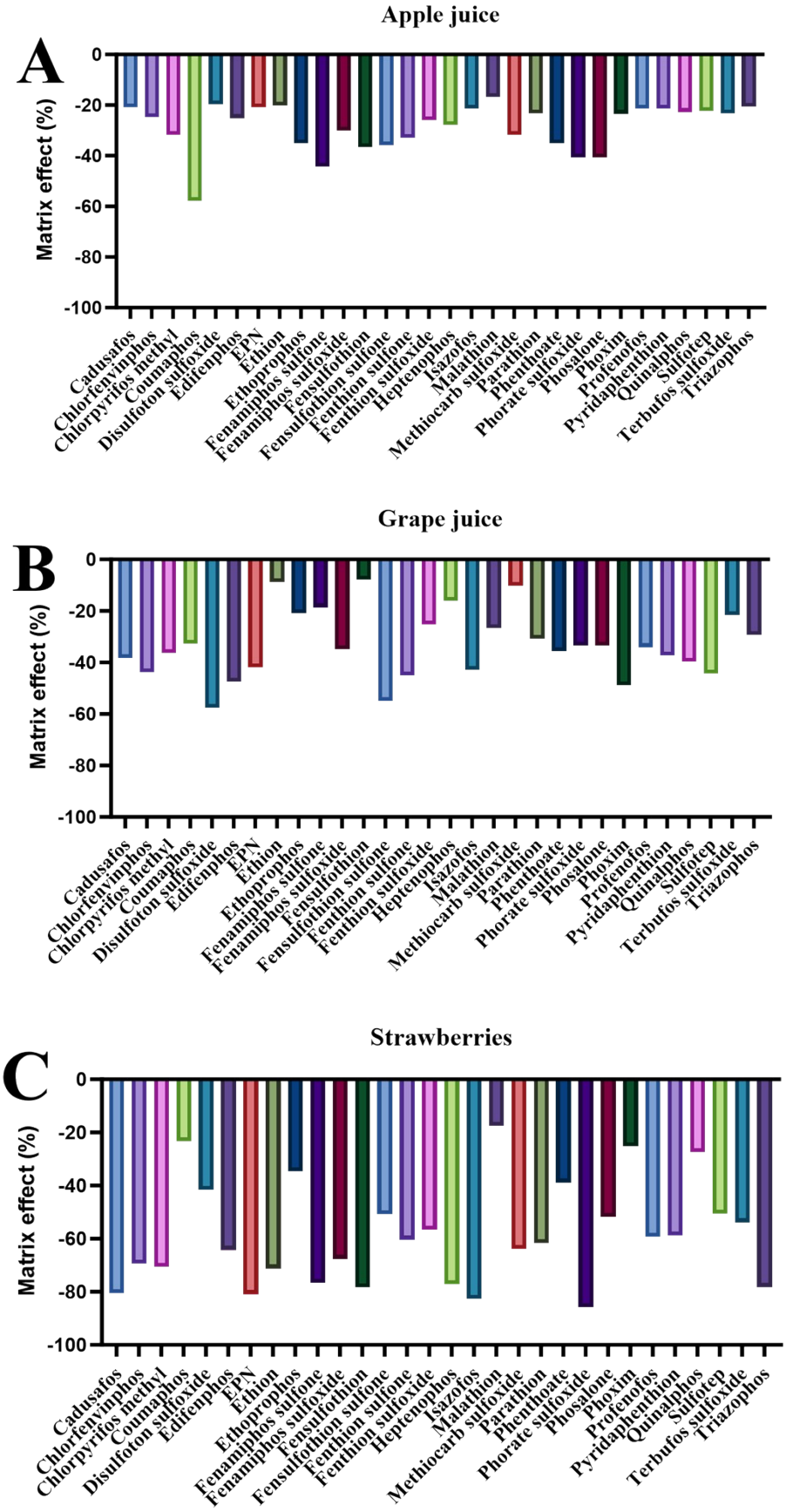


Figure S3. TEM image of SBA-15.


A
Apple juice
Matrix effect (%)
0
-20
-40
-60
-80
-100
Cadusafos
Chlorfenvinphos
Chlorpyrifos methyl
Coumaphos
Disulfoton sulfoxide
Edifenphos
EPN
Ethion
Ethoprophos
Fenamiphos sulfone
Fenamiphos sulfoxide
Fensulfothion
Fensulfothion sulfone
Fenthion sulfone
Fenthion sulfoxide
Heptenophos
Isazofos
Malathion
Methiocarb sulfoxide
Parathion
Phenthoate
Phorate sulfoxide
Phosalone
Phoxim
Profenofos
Pyridaphenthion
Quinalphos
Sulfotep
Terbufos sulfoxide
Triazophos
B
Grape juice
Matrix effect (%)
0
-20
-40
-60
-80
-100
Cadusafos
Chlorfenvinphos
Chlorpyrifos methyl
Coumaphos
Disulfoton sulfoxide
Edifenphos
EPN
Ethion
Ethoprophos
Fenamiphos sulfone
Fenamiphos sulfoxide
Fensulfothion
Fensulfothion sulfone
Fenthion sulfone
Fenthion sulfoxide
Heptenophos
Isazofos
Malathion
Methiocarb sulfoxide
Parathion
Phenthoate
Phorate sulfoxide
Phosalone
Phoxim
Profenofos
Pyridaphenthion
Quinalphos
Sulfotep
Terbufos sulfoxide
Triazophos
C
Strawberries
Matrix effect (%)
0
-20
-40
-60
-80
-100
Cadusafos
Chlorfenvinphos
Chlorpyrifos methyl
Coumaphos
Disulfoton sulfoxide
Edifenphos
EPN
Ethion
Ethoprophos
Fenamiphos sulfone
Fenamiphos sulfoxide
Fensulfothion
Fensulfothion sulfone
Fenthion sulfone
Fenthion sulfoxide
Heptenophos
Isazofos
Malathion
Methiocarb sulfoxide
Parathion
Phenthoate
Phorate sulfoxide
Phosalone
Phoxim
Profenofos
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Quinalphos
Sulfotep
Terbufos sulfoxide
Triazophos

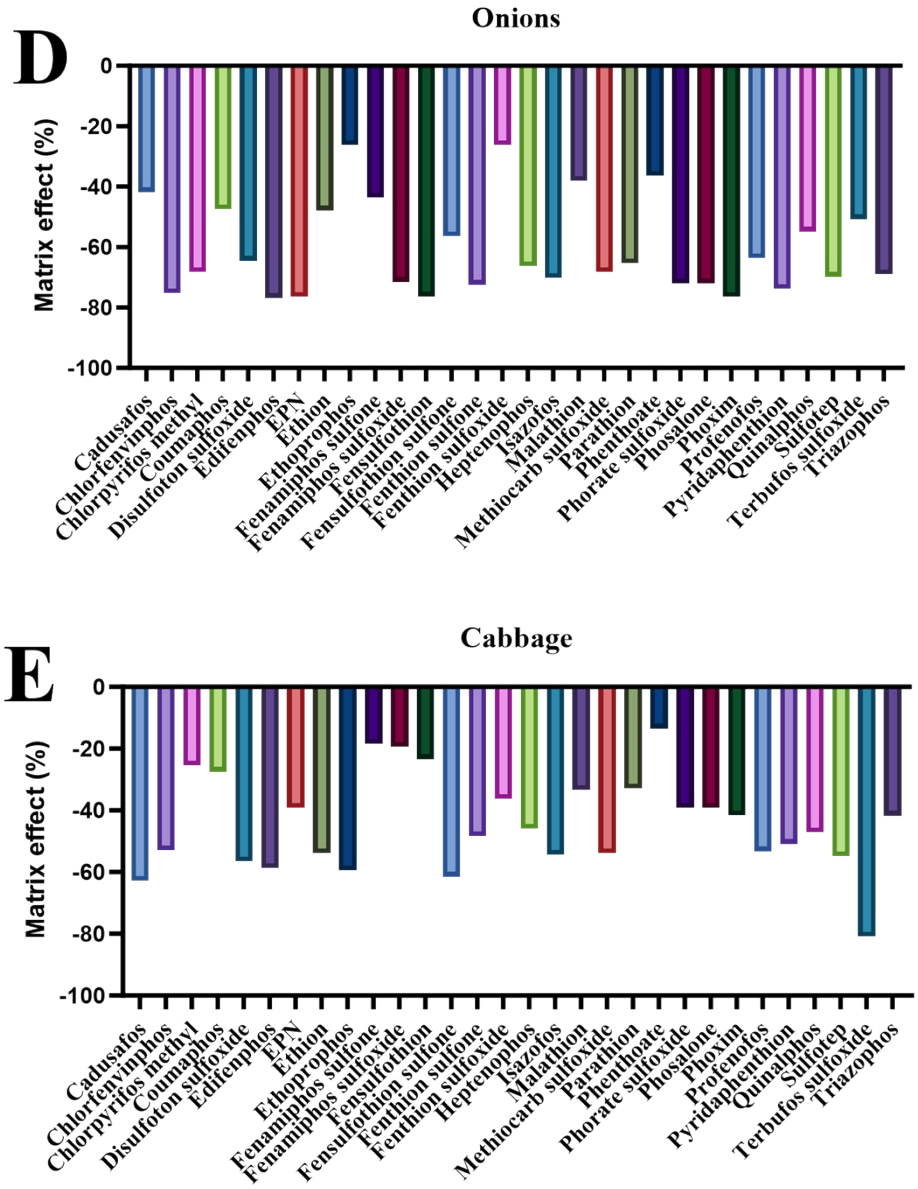

Figure S4. Matrix effects of thirty organophosphate pesticides in five types of food matrices. (A) Apple juice, (B) Grape juice, (C) Strawberries, (D) Onions, (E) Cabbage.

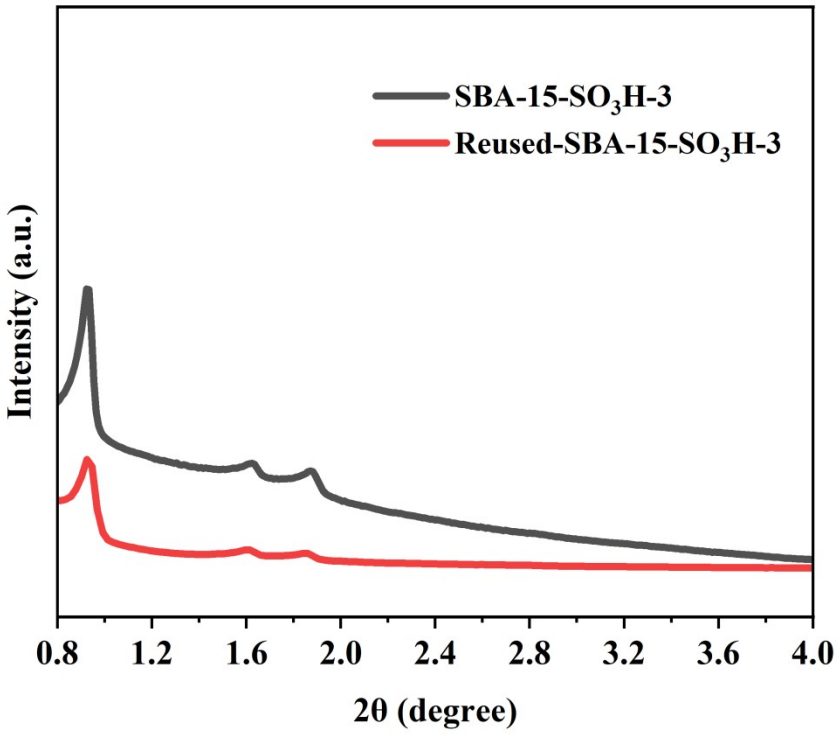

Figure S5. XRD spectra of SBA-15-SO_3H-3 and the Reused-SBA-15-SO_3H-3.

Table S1. Chromatographic conditions for the detection of organophosphorus pesticides.

Instrument conditions			
Chromatographic column	ACQUITY UPLC® BEH C18 column (2.1 mm ×100 mm, 1.7 μm; Waters)		
Column temperature	40℃		
Injection volume	2 μL		
Flow rate	0.3 mL/min		
Mobile phase A	0.1% formic acid aqueous		
Mobile phase B	ACN		
Gradient elution	Time (min)	A (%)	B (%)
	0	95%	5%
	1	95%	5%
	3	24%	76%
	6	1%	99%
	9.1	1%	99%
	9.6	95%	5%
	11.5	95%	5%

Table S2. The BET surface areas, pore volume and pore diameter of SBA-15, SBA-15-SO_3H-1, SBA-15-SO_3H-2 and SBA-15-SO_3H-3.

Sample	Surface area (m^2/g)	Pore volume (cm^3/g)	Pore diameter (nm)
SBA-15	643.06	0.95	5.79
SBA-15-SO_3H-1	147.90	0.23	5.29
SBA-15-SO_3H-2	100.08	0.15	5.07
SBA-15-SO_3H-3	259.93	0.34	5.08

Table S3. Analyze the contamination levels (μg/kg) of thirty organophosphate pesticides in food samples.

Sample	No.	Malathion	Profenofos	Triazophos	Phoxim	Others
Apple juice	1	ND	ND	ND	ND	ND
	2	ND	ND	ND	ND	ND
	3	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND
	5	ND	ND	ND	ND	ND
Grape juice	1	ND	ND	ND	ND	ND
	2	ND	ND	ND	ND	ND
	3	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND
	5	ND	ND	ND	ND	ND
Strawberries	1	ND	14.47	ND	ND	ND
	2	34.65	ND	1.67	ND	ND
	3	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND
	5	44.21	10.03	2.11	ND	ND
Onions	1	ND	ND	ND	ND	ND
	2	45.23	ND	ND	ND	ND
	3	ND	9.91	4.21	ND	ND
	4	49.11	8.85	3.23	ND	ND
	5	ND	ND	ND	ND	ND
Cabbage	1	ND	ND	ND	9.75	ND
	2	ND	ND	21.1	9.08	ND
	3	55.24	ND	ND	8.86	ND
	4	ND	19.28	ND	5.70	ND
	5	ND	ND	ND	ND	ND

ND, not detected.