Electronic Supplementary Material (ESI) for Analytical Methods.

- ELECTRONIC SUPPLEMENTARY INFORMATION -

Ammonium salting out extraction with analyte preconcentration for sub-part per billion quantitative analysis in surface, ground and drinking water by flow injection tandem mass spectrometry

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Table S-1. Guidance for the preparation of matrix-matched standard solutions by serial dilution. All solutions described below should be prepared by diluting with control matrix solvent.

Standard concentration	Aliquot to be taken from	Aliquot size	Diluent volume**	Final volume	Glassware
1,000 ng/mL intermediate std.	10 μg/mL mixed stock std. (containing all analytes)	40 μL	360 μL	0.40 mL	HPLC vial
100 ng/mL intermediate std.	1,000 ng/mL intermediate std.	40 μL	360 μL	0.40 mL	HPLC vial
10 ng/mL calibration std.	100 ng/mL intermediate std.	100 μL	900 μL	1.0 mL	HPLC vial
5.0 ng/mL calibration std.	100 ng/mL intermediate std.	50 μL	950 μL	1.0 mL	HPLC vial
1.0 ng/mL calibration std.	10 ng/mL calibration std.	100 μL	900 μL	1.0 mL	HPLC vial
0.5 ng/mL calibration std.	10 ng/mL calibration std.	50 μL	950 μL	1.0 mL	HPLC vial
0.25 ng/mL calibration std.	5 ng/mL calibration std.	50 μL	950 μL	1.0 mL	HPLC vial
0.15 ng/mL calibration std.	1 ng/mL calibration std.	150 μL	850 μL	1.0 mL	HPLC vial
0.07 ng/mL calibration std	1 ng/mL calibration std.	70 μL	930 μL	1.0 mL	HPLC vial

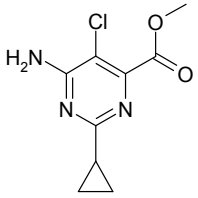
**Diluted control matrix solvent was used for preparation of the intermediate standard and calibration standards. The diluted control matrix solvents were prepared as indicated below.

Diluted control matrix solvent for analysis of water

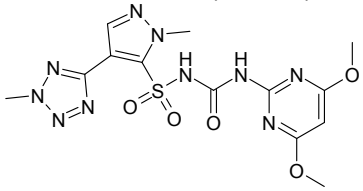
Mix 2.0 mL of pooled acetonitrile-rich phase (top layer) of control water samples with 6.0 mL of acetonitrile; centrifuge if necessary. This yields 8.0 mL of diluted control matrix solvent, which should be enough to prepare the matrix-matched standards described above.

Figure S-1. Pesticide active ingredients used as representative small organic molecules (analytes) to develop and test the ammonium chloride salting out extraction method for water analysis.

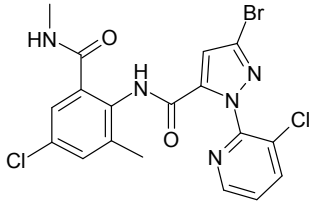
Aminocyclopyrachlor methyl (227 Da)



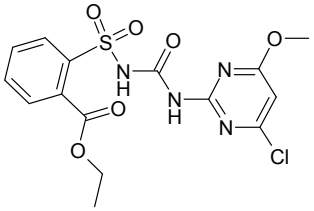
Azimsulfuron (424 Da)



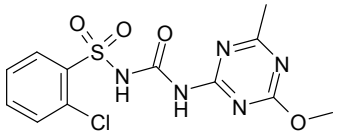
Chlorantraniliprole (482 Da)



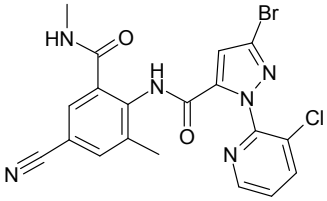
Chlorimuron ethyl (414 Da)



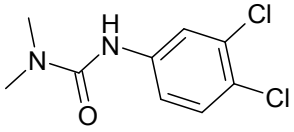
Chlorsulfuron (357 Da)



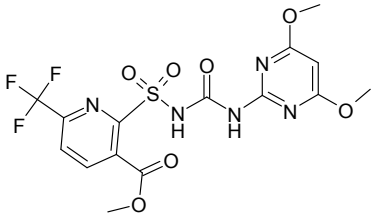
Cyantraniliprole (473 Da)



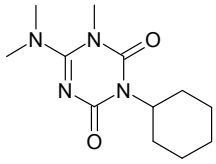
Diuron (232 Da)



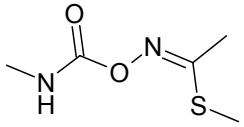
Flupyrsulfuron methyl (465 Da)



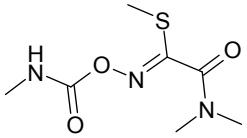
Hexazinone (252 Da)



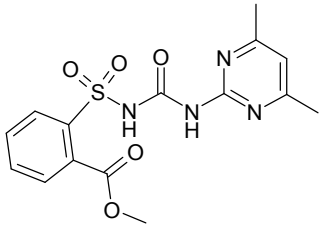
Methomyl (162 Da)



Oxamyl (219 Da)



Sulfometuron methyl (364 Da)



Triflusulfuron methyl (492 Da)

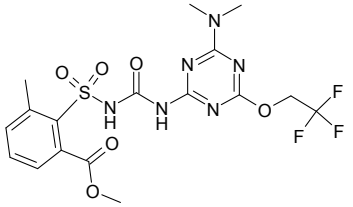


Figure S-2. Representative FI/MS/MS chronograms (a through h) obtained for diuron, m/z 233 → 72 m/z, in matrix-matched calibration standards prepared using tap water extracts. The 0.07 ng/mL to 10 ng/mL concentration range in the calibration standards corresponds to analyte concentrations in water ranging from 0.056 µg/L to 8.0 µg/L. The resulting calibration curve is shown in panel (i).