

SUPPLEMENTARY INFORMATION

Organic Contaminants in Bio-based Fertilizer Treated Soil: Target and Suspect Screening Approaches

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Figure S 1 Google Earth (V. 9.166.0.1) view showing the field experiment locations: Seville, Spain (left) and Jokioinen, Finland (right).

Table S 1 Physical and chemical properties of Soil A and Soil B.

Characteristics	Value (Soil A)	Value (Soil B)
% clay	61.7	48.8
% silt	25.9	26.1
% sand	10.6	20.4
Organic matter (%)	1.9	4.8
pH	7.66	5.74

Table S 2 Name, uses, supplier, CAS number and molecular formula of the reference standards.

Name	Uses	Supplier	CAS number
1-Hydroxyibuprofen	Anti-inflammatory drug metabolite	Sigma Aldrich	53949-53-4
2,4,5-Trichlorophenoxyacetic acid	Herbicide	Sigma Aldrich	93-76-5
2,4-DB neg / 4-(2,4-dichlorophenoxy)butyric acid	Herbicide	Sigma Aldrich	94-82-6
2,4-DP neg (Dichlorprop)	Herbicide	Sigma Aldrich	120-36-5
2,4-Dichlorophenoxyacetic acid	Herbicide	Sigma Aldrich	94-75-7
3-Hydroxycarbamazepine	Anti-convulsant drug metabolite	Sigma Aldrich	68011-67-6
Azinophos-ethyl	Insecticide	Sigma Aldrich	220-147-6
Aldicarb	Insecticide	Sigma Aldrich	116-06-3
Bezafibrate	Lipid-lowering drug	Sigma Aldrich	41859-67-0
Chlorbromuron	Herbicide	Sigma Aldrich	13360-45-7
Clofibric acid	Lipid-lowering drug metabolite	Sigma Aldrich	882-09-7
Diclofenac	Anti-inflammatory drug	Sigma Aldrich	15307-79-6
Diuron	Herbicide	Sigma Aldrich	330-54-1
Ethidimuron	Herbicide	Sigma Aldrich	30043-49-3
Fluometuron	Herbicide	Sigma Aldrich	2164-17-2
Fluroxypyr	Herbicide	Sigma Aldrich	69377-81-7
Gemfibrozil	lipid-lowering drug	Sigma Aldrich	25812-30-0
Heptenophos	Insecticide	Sigma Aldrich	23560-59-0
Ibuprofen	Anti-inflammatory drug	Sigma Aldrich	15687-27-1
Ketoprofen	Anti-inflammatory drug	Sigma Aldrich	22071-15-4
Lenacil	Herbicide	Sigma Aldrich	2164-08-1
MCPA	Herbicide	Sigma Aldrich	94-74-6

MCPB	Herbicide	Sigma Aldrich	94-81-5
MCPPP-p	Herbicide	Sigma Aldrich	93-65-2
Methabenzthiazuron	Herbicide	Sigma Aldrich	18691-97-9
Metolachlor-s	Herbicide	Sigma Aldrich	87392-12-9
Metoprolol	Beta-blocker	Sigma Aldrich	37350-58-6
Monolinuron	Herbicide	Sigma Aldrich	1746-81-2
N4-Acetylsulfamethoxazole	Antibiotic	Sigma Aldrich	21312-10-7
Prosulfocarb (SPV A066)	Herbicide	Sigma Aldrich	52888-80-9
Siduron	Herbicide	Sigma Aldrich	1982-49-6
Simetryne	Herbicide	Sigma Aldrich	1014-70-6
Sotalol	Beta-blocker	Sigma Aldrich	3930-20-9
Triazophos	Acaricides/Nematicides	Sigma Aldrich	24017-47-8
Trietazine	Herbicide	Sigma Aldrich	1912-26-1

Table S 3 Monitored quantifier and qualifier ions, ion ratios and retention time

Compound Name	Formula	Quantifier m/z (Q)	Qualifier m/z (q)	q/Q	ESI Mode	Rt	Internal Standard
1-Hydroxyibuprofen	C13H18O3	221.1183	159.1174	0.72	-	12.3	Ibuprofen-d3
2,4,5-Trichlorophenoxyacetic acid	C8H5Cl3O3	252.9232	158.941	0.63	-	13	n/a
2,4-DB neg / 4-(2,4-dichlorophenoxy)butyric acid	C10H10Cl2O3	246.9934	159.9719	0.65	-	14.58	n/a
2,4-DP neg (Dichlorprop)	C9H8Cl2O3	232.9778	162.9532	0.7	-	13.1	n/a
2,4-Dichlorophenoxyacetic acid	C7H5Cl2NO	189.9821	172.9557	0.91	-	9.35	n/a
3-Hydroxycarbamazepine	C15H12N2O2	251.0826	207.0687	0.82	-	12.2	Carbamazepine 13C6
Aldicarb	C7H14N2O2S	213.0668	116.0533	0.54	+	11.4	n/a
Azinophos-ethyl	C12H16N3O3PS2	346.0443	368.0263	1.06	+	15.9	n/a
Bezafibrate	C19H20ClNO4	362.1154	138.9944	0.38	+	13.68	n/a
Chlorbromuron	C9H10BrClN2O2	292.9687	203.9212	0.7	+	13.64	n/a
Clofibric acid	C10H11ClO3	213.0324	153.1279	0.72	-	13.04	n/a
Diclofenac	C14H11Cl2NO2	294.0094	250.0187	0.85	-	15.42	Diclofenac-13C6
Diuron	C9H10Cl2N2O	233.0243	218.0981	0.94	+	12.61	Diuron-d6
Ethidimuron	C7H12N4O3S2	265.0424	208.0213	0.78	+	10.6	n/a
Fluometuron	C10H11F3N2O	233.0896	72.0444	0.31	+	11.93	n/a
Fluroxypyr	C7H5Cl2FN2O3	252.9588	174.9466	0.69	-	10.96	n/a
Gemfibrozil	C15H22O3	249.1496	121.0656	0.49	-	15.6	n/a
Heptenophos	C9H12ClO4P	251.0234	127.015	0.51	+	13.45	n/a
Ibuprofen	C13H18O2	205.1234	161.133	0.79	-	14.8	Ibuprofen-d3
Ketoprofen	C16H14O3	255.1016	209.0961	0.82	+	13.91	n/a
Lenacil	C16H14O3	255.1016	209.0961	0.82	-	13.91	n/a
MCPA	C9H9ClO3	199.0167	144.03	0.72	-	12.6	MCPA-d3

MCPB	C11H13ClO3	227.048	143.008	0.63	-	14.6	MCPA-d3
MCPP-p	C10H11ClO3	213.0324	141.0111	0.66	-	13.3	MCPA-d3
Methabenzthiazuron	C10H11N3OS	222.0696	165.048	0.74	+	13.45	n/a
Metolachlor-s	C15H22ClNO2	284.1412	252.1153	0.89	+	15.09	n/a
Metoprolol	C15H25NO3	268.1907	191.1056	0.71	+	10.72	Metoprolol-d7
Monolinuron	C9H11ClN2O2	215.0582	148.0628	0.69	+	12.6	n/a
N4-Acetylsulfamethoxazole	C12H13N3O4S	294.0554	198.0226	0.67	-	11	n/a
Prosulfocarb	C14H21NOS	252.1417	274.1236	1.09	+	15.85	n/a
Siduron	C14H20N2O	233.1648	255.1468	1.09	+	13.4	n/a
Simetryne	C8H15N5S	214.1121	236.094	1.1	+	12.72	n/a
Sotalol	C12H20N2O3S	273.1267	213.0697	0.78	+	7	n/a
Triazophos	C12H16N3O3PS	314.0723	167.0978	0.53	+	15.2	n/a
Trietazine	C9H16ClN5	230.1167	189.0767	0.82	+	14.25	Trietazine-d5

Table S 4 Average recoveries, matrix effect (ME), linearity, Limit of Detection (LoD) and Limit of Quantitation (LoQ) for targeted organic contaminants in Soil A.

Compound Name	Chemical Classification	Linearity	Soil A				% ME	LoD (ng/g)	LoQ (ng/g)
			10 ng/g		50 ng/g				
			% Recovery	%RSD	% Recovery	%RSD			
Azinophos-ethyl	Insecticide	0,992	64	18	91	3	-51	1,2	3,5
Diuron	Herbicide	0,99727	102	10	103	3	-40	1,3	3,8
Ethidimuron	Herbicide	0,99723	87	2	133	11	-46	0,4	1,3
Ethofumesate	Herbicide	0,9981	90	18	86	4	-60	2,2	6,6
Fluometuron	Herbicide	0,99017	129	2	88	13	-27	1,6	4,7
Heptenophos	Insecticide	0,99872	117	21	129	1	-40	0,9	2,7
Metolachlor-s	Herbicide	0,99515	121	13	109	16	-24	0,8	2,5
Metoprolol	Beta-blocker	0,99909	65	11	111	1	-15	1,6	4,8
Monolinuron	Herbicide	0,9909	115	13	115	6	-42	1,5	4,6
Prosulfocarb	Herbicide	0,99828	117	16	67	1	-55	0,2	0,5
Siduron	Herbicide	0,99754	129	12	104	17	-37		0,0
Simetryne	Herbicide	0,99927	52	11	58	20	-29	0,0	0,0
Sotalol	Beta-blocker	0,99675	42	16	89	9	-50	0,5	1,5
Triazophos	Acaricides/Nematicides	0,99734	116	13	106	6	-45	0,5	1,4
1-Hydroxyibuprofen	Anti-inflammatory drug metabolite	0,99738	115	1	118	6	-20	0,4	1,3

2,4,5-Trichlorophenoxyacetic acid	Herbicide	0,99764	75	1	72	1	-17	0,5	1,4
2,4-DB neg / 4-(2,4-dichlorophenoxy)butyric acid	Herbicide	0,99168	121	6	115	1	-11	0,4	1,3
2,4-DP neg (Dichlorprop)	Herbicide	0,99433	96	7	91	3	-18	1,3	4,0
2,4-Dichlorphenoxyacetic acid	Herbicide	0,99928	67	7	69	1	-8	0,2	0,7
3-Hydroxycarbamazepine	Anti-convulsant drug metabolite	0,9966	78	6	100	8	-21	0,3	1,0
Clofibric acid	Lipid-lowering drug metabolite	0,99321	111	4	110	5	-9	0,3	1,0
Diclofenac	Anti-inflammatory drug	0,99956	81	12	83	7	-40	0,4	1,4
Fluroxypyr	Herbicide	0,99825	69	1	72	3	-11	0,4	1,3
Gemfibrozil	lipid-lowering drug	0,99475	72	13	66	10	-58	0,5	1,4
Ibuprofen	Anti-inflammatory drug	0,99809	106	22	89	12	-41	0,3	1,0
Lenacil	Herbicide	0,99051	117	13	105	1	-28	1,2	3,5
MCPA	Herbicide	0,99219	61	9	66	1	-15	0,2	0,7
MCPB	Herbicide	0,9983	113	10	97	2	-26	0,4	1,2
MCPP-p	Herbicide	0,9963	102	8	94	2	-16	0,4	1,2
Methoxyfenozone	Insecticide	0,99153	76	18	81	4	-55	1,2	3,8
Neburon	Herbicide	0,97699	85	18	79	2	-45	1,1	3,3
N4-Acetylsulfamethoxazole	Antibiotic	0,9983	156	3	158	5	-13	0,4	1,3

Aldicarb	Insecticide	0,99224	144	6	167	5	-8	1,9	5,8
Bezafibrate	Lipid-lowering drug	0,99252	54	5	59	11	-45	1,5	4,4
Chlorbromuron	Herbicide	0,98424	56	28	86	5	-58	2,1	6,3
Ketoprofen	Anti-inflammatory drug	0,99009	50	5	47	2	-65	1,5	4,6
Methabenzthiazuron	Herbicide	0,99785	129	4	52	27	-30	2,3	7,0
Tetrachlorovinphos	Insecticide	0,9941	68	26	67	2	-66	1,8	5,4
Trietazine	Herbicide	0,98	114	26	55	1	-19	2,8	8,5
		Mean	93,8	11,1	92,1	6,2	-33,5	1,0	2,8
		Median	96,1	10,9	89,5	4,3	-30,0	0,7	1,5
		Max	155,56	28,00	167,34	27,41	-8,25	2,8	8,5

Table S 5 Average recoveries, matrix effect (ME), linearity, Limit of Detection (LoD) and Limit of Quantitation (LoQ) for targeted organic contaminants in Soil B.

Compound Name	Chemical Classification	Linearity	Soil B				%ME	LoD (ng/g)	LoQ (ng/g)
			10 ng/g		50 ng/g				
			% Recovery	%RSD	% Recovery	%RSD			
Azinophos-ethyl	Insecticide	0,992	43	5	75	18	-71,9	3,8	11,5
Diuron	Herbicide	0,997	86	20	102	6	-45,1	3,8	11,5
Ethidimuron	Herbicide	0,997	99	19	137	10	-30,7	0,8	2,5
Fluometuron	Herbicide	0,990	126	2	99	17	-38,3	0,6	1,7
Heptenophos	Insecticide	0,999	117	4	134	5	-42,8	1,1	3,2
Metolachlor-s	Herbicide	0,995	130	5	124	6	-38,8	0,6	1,9
Metoprolol	Beta-blocker	0,999	84	25	51	11	-6,5	6,4	19,3
Monolinuron	Herbicide	0,991	100	16	122	3	-43,5	0,9	2,8
Prosulfocarb	Herbicide	0,998	73	5	61	18	-66,5	0,9	2,8
Siduron	Herbicide	0,998	98	11	95	3	-45,2	0,9	2,7
Simetryne	Herbicide	0,999	104	5	61	13	-63,3		
Sotalol	Beta-blocker	0,997	56	5	99	22	-42,8	4,9	14,7
Triazophos	Acaricides/ Nematicides	0,997	75	20	80	8	-49,2	1,9	5,8
1-Hydroxyibuprofen	Anti-inflammatory drug metabolite	0,997	41	22	105	3	-17,9	0,5	1,6
2,4,5-Trichlorophenoxyacetic acid	Herbicide	0,998	64	8	68	1	-23,9	0,5	1,5
2,4-DB neg / 4-(2,4-dichlorophenoxy)butyric acid	Herbicide	0,992	100	7	83	5	-17,2	0,5	1,6
2,4-DP neg (Dichlorprop)	Herbicide	0,994	80	4	95	1	-22,4	0,7	2,2
2,4-Dichlorophenoxyacetic acid	Herbicide	0,999	62	0	64	2	-17,0	0,2	0,7
3-Hydroxycarbamazepine	Anti-convulsant drug metabolite	0,997	89	11	104	3	-33,7	0,3	0,8
Clofibric acid	Lipid-lowering drug metabolite	0,993	108	8	98	6	-18,6	0,2	0,7
Diclofenac	Anti-inflammatory drug	1,000	44	11	42	4	-61,1	0,4	1,4
Fluroxypyr	Herbicide	0,998	72	6	70	1	-12,8	0,5	1,5
Gemfibrozil	lipid-lowering drug	0,995	36	13	30	6	-74,5	0,5	1,4

Ibuprofen	Anti-inflammatory drug	0,998	94	22	69	7	-43,3	0,3	1,0
Lenacil	Herbicide	0,991	96	21	91	2	-43,4	0,8	2,5
MCPA	Herbicide	0,992	67	8	68	1	-21,4	0,2	0,6
MCPB	Herbicide	0,998	82	11	71	2	-37,2	0,5	1,4
MCPP-p	Herbicide	0,996	105	4	89	1	-23,9	0,2	0,7
N4-Acetylsulfamethoxazole	Antibiotic	0,998	139	7	144	6	-24,5	0,4	1,3
Aldicarb	Insecticide	0,992	149	7	160	9	-12,1	1,6	5,0
Bezafibrate	Lipid-lowering drug	0,993	28	15	53	18	-60,2	3,8	11,5
Chlorbromuron	Herbicide	0,984	42	5	75	17	-79,0	3,8	11,5
Ketoprofen	Anti-inflammatory drug	0,990	48	0	42	15	-77,6	0,9	2,7
Methabenzthiazuron	Herbicide	0,998	104	3	87	16	-36,2	2,0	6,0
Trietazine	Herbicide	0,98	137	1	63	18	-24,4	6,0	18,3
		Mean	85	10	86	8	-39,1	1,5	4,6
		Median	86	7	83	6	-38,3	0,8	2,4
		Max	149	25	160	22	-6,5	6,4	19,3

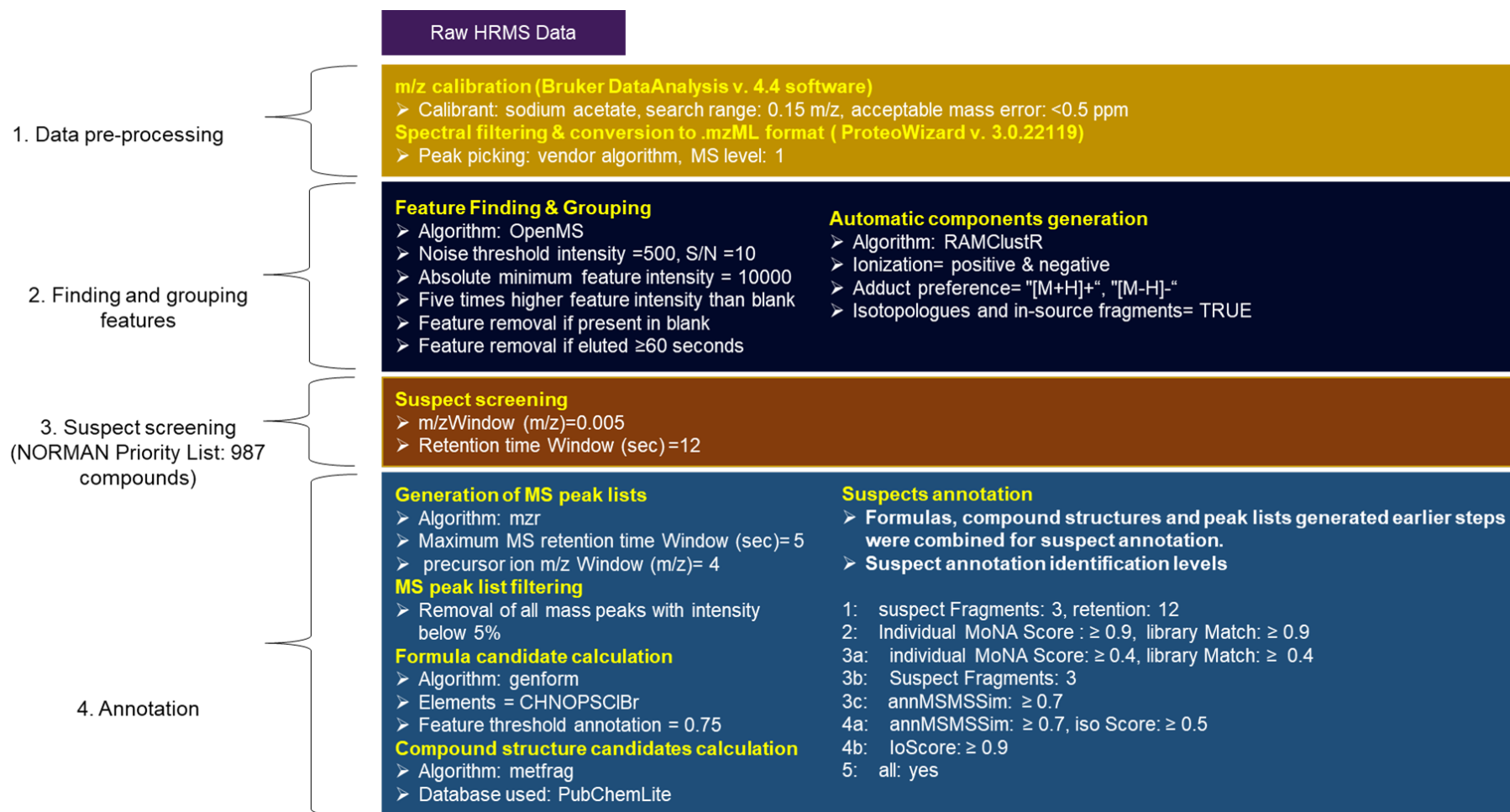


Figure S 2 Suspect screening workflow

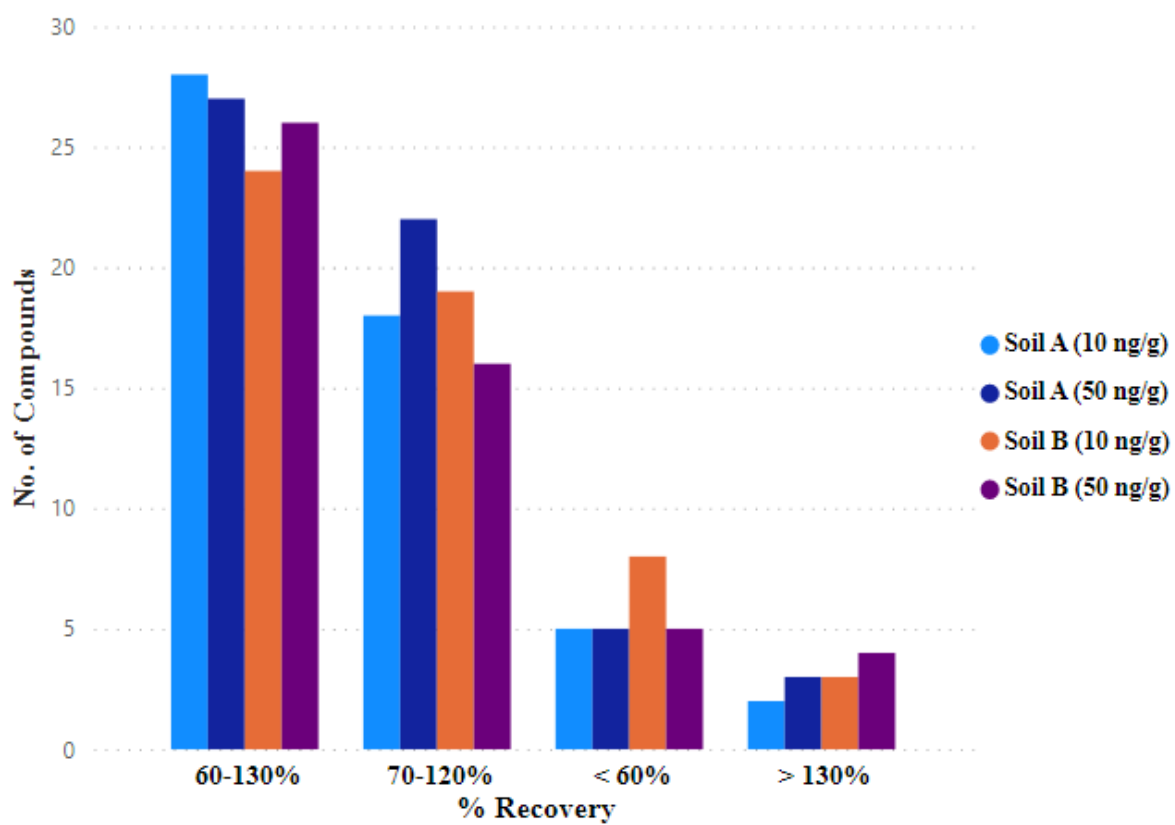


Figure S 3 Method recovery rates for Soil A and Soil B at 10 ng g⁻¹ and 50 ng g⁻¹ concentration levels

Table S 6 Concentration of target compounds found in the before soil sample and after N treatment (n=1).

N Treatment	Concentration (ng g ⁻¹)			
	Soil A		Soil B	
	Ibuprofen	1-Hydroxyibuprofen	1-Hydroxyibuprofen	Lenacil
Before Soil	0.5 ^a	0.6 ^a	ND	ND
BA6	0.5 ^a	0.6 ^a	1.1 ^a	14.1
PAL	ND	0.5 ^a	1.6	1.7 ^a
FEK	0.4 ^a	0.7 ^a	2.2	ND
MO13	0.5 ^a	0.6 ^a	1.0 ^a	ND
ECO	0.4 ^a	1.2 ^a	1.0 ^a	ND
OG2	0.4 ^a	0.8 ^a	1.0 ^a	ND
BIO	0.5 ^a	0.6 ^a	1.2 ^a	ND

ND: concentrations less than (<) their estimated detection limits.

^a: Analyte concentration was < limit of quantification (LoQ) but > limit of detection (LoD)

Table S 7 Concentration of target compounds found in the before soil sample and after P treatment (n=1).

P Treatment	Concentration (ng g ⁻¹)			
	Soil A		Soil B	
	Ibuprofen	1-Hydroxyibuprofen	1-Hydroxyibuprofen	Lenacil
Before Soil	0.5 ^a	1.4	ND	ND
BA1	0.6 ^a	0.8 ^a	1.6	28.7
MO14	ND	1a	2	ND
MB1	ND	0.7 ^a	ND	ND
CGO	0.6 ^a	1.3	0.9 ^a	ND
ADC	0.6 ^a	1a	0.8 ^a	ND
OPU	ND	1.5	1.2 ^a	ND
EPH	0.6 ^a	1.5	1.5	ND
PLA	ND	1.1	ND	ND

ND: concentrations less than (<) their estimated detection limits.

^a: Analyte concentration was < limit of quantification (LoQ) but > limit of detection (LoD)