

Table S1. GC-MS method parameters.

Injection	Volume	1 µL
	Plunger speed	Fast plunger
Inlet	Mode	Split (1:1)
	Liner	Split/splitless straight liner with deactivated fused silica wool (4 mm × 6.3 mm × 78.5 mm)
	Temperature	240 °C
Column	Type	Restek Hydroguard 2m x 0.32mm precolumn Restek Rxi®-624Sil MS column (60 m × 0.32 mm ID × 1.8 µm film thickness)
Flow rate	Initial flow rate	4.0 mL/min held for 1.5 min
	Ramp 1 flow rate	To 2.0 mL/min at 10 mL/min ²
Oven temperature program	Initial temperature	45 °C for 1 min
	Ramp 1	14 °C to 150 °C
	Ramp 2	5 °C to 240 °C
	Final temperature hold time	5 min
MS detector	Transfer line temperature	240 °C
	Ion source temperature	230 °C
	Ionization energy	70 eV
	Mass analyzer temperature	150 °C
	Acquisition mode	SIM (Selected ion monitoring)
	SIM fragmentation ions	Summarized in Table S2 .

Table S2. Retention times, quantifier, and qualifier ions of target analytes

Compound	Quantifier ion (m/z)	Qualifier ion 1 (m/z)	Qualifier ion 2 (m/z)	Retention time (min)
Benzene	78	77	N/A	7.1
Toluene-d8	100	98	N/A	8.7
Toluene	92	91	N/A	8.8
Ethylbenzene	91	106	N/A	10.4
m/p-Xylene	91	106	N/A	10.5
o-Xylene	91	106	N/A	11.0
Sulfolane-d8	46	128	N/A	20.9
Sulfolane	41	56	120	21.0

Table S3. Plackett-Burman design for the evaluation of method ruggedness.

Factor	Description	-1 (Low Level)	1 (High Level)
A	Sample temperature	4 °C	30 °C
B	Discarding the first portion of drawn water	No	Yes
C	Time between opening the sample vial and adding water to dichloromethane	15 sec	90 sec
D	Volume of dichloromethane	450 µL	500 µL
E	Vortex shaking applied	No	Yes
F	Use of already pierced septa	No	Yes
G	Methanol wash only (dichloromethane replaced with methanol in the autosampler)	No	Yes
H	Injection performed 24 h after sample preparation	No	Yes
J	Tool used for sampling water	Syringe	Automatic pipette
K	Dummy factor (for experimental design)	N/A	N/A
L	Order of addition	Water sample to dichloromethane	Dichloromethane to water sample

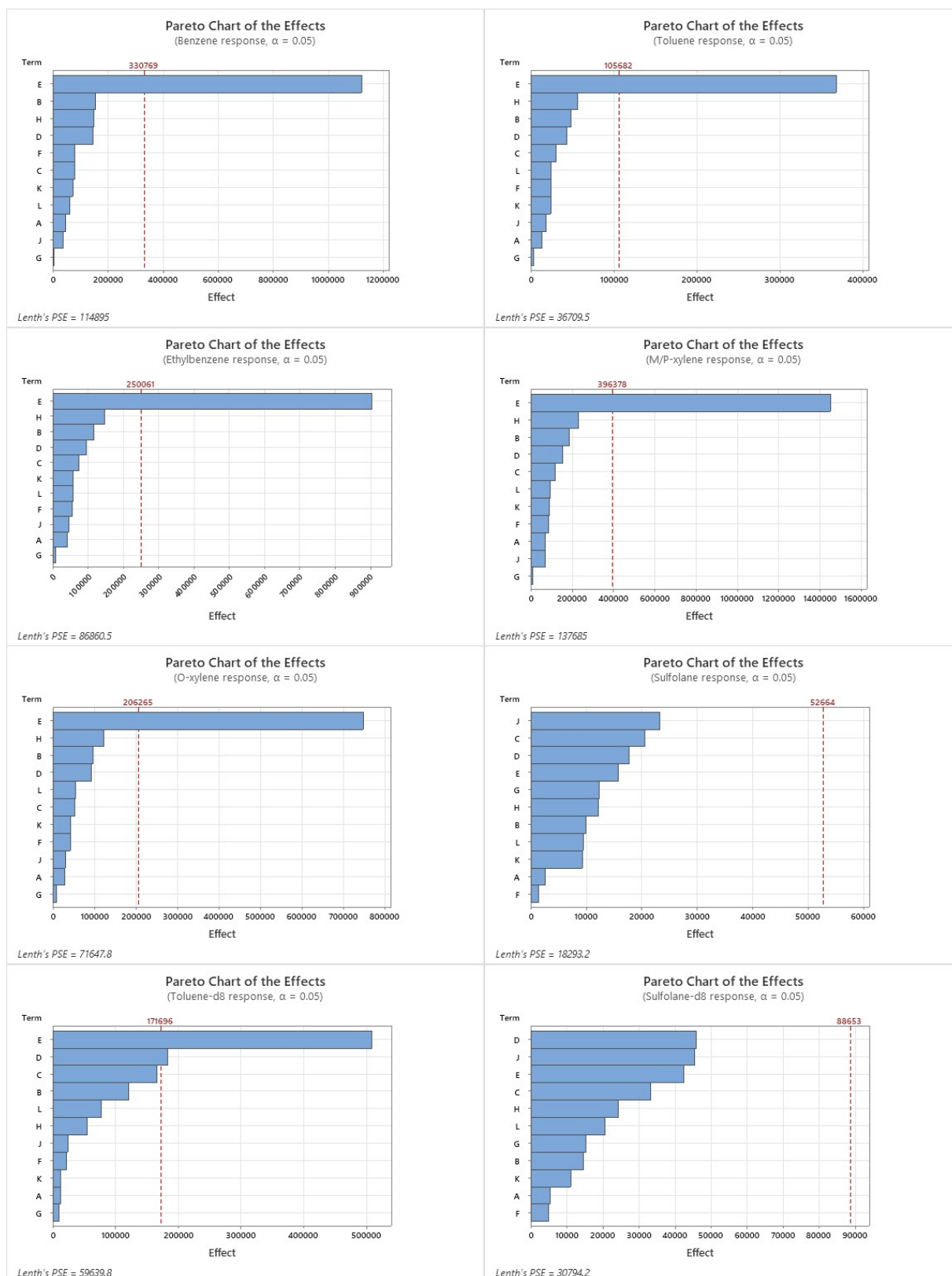


Figure S1. Pareto charts of effects of different factors on the response of the instrument for the target analytes. Factors are described in **Table S2**.

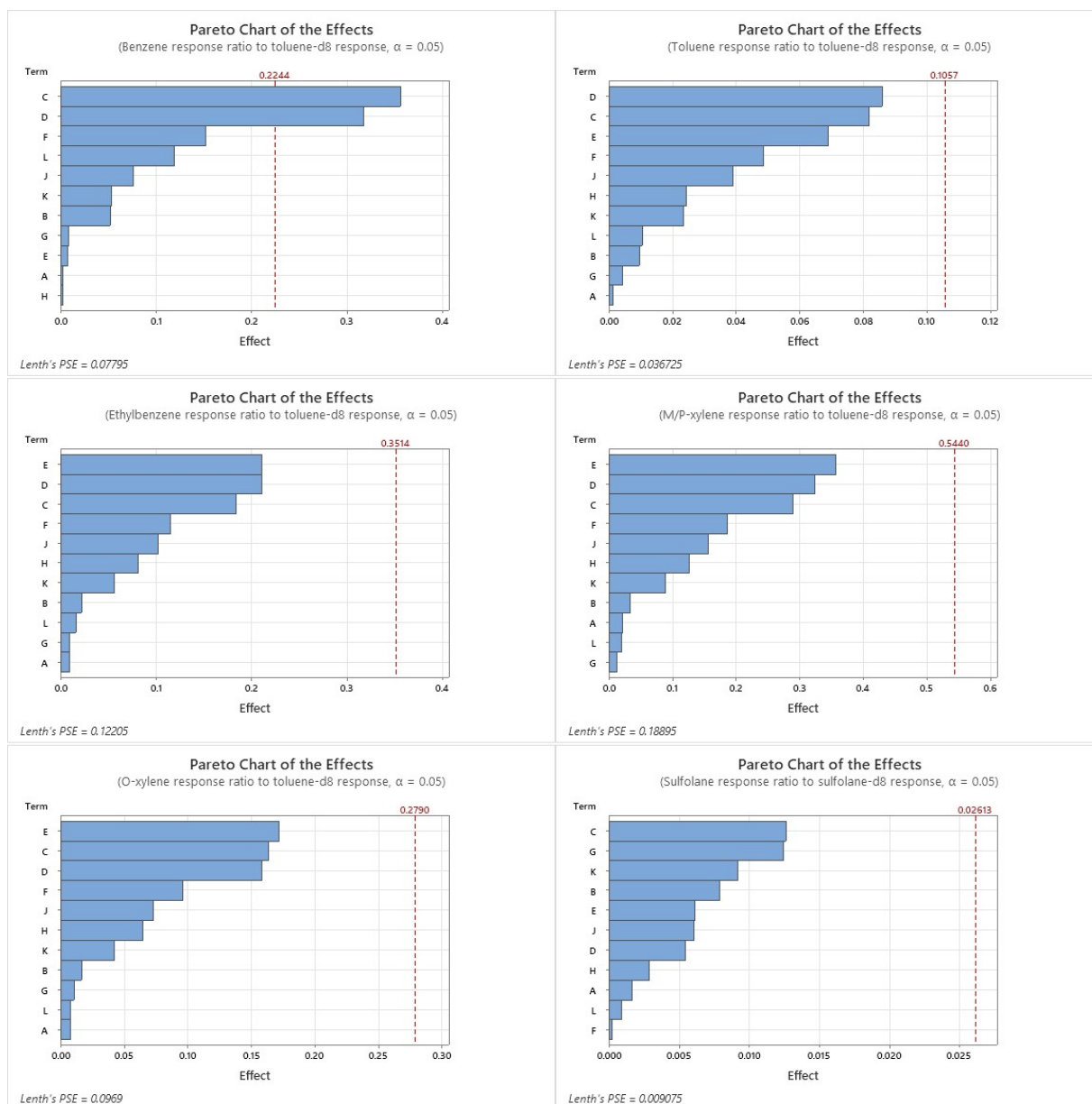


Figure S2. Pareto charts of effects of different factors on the ratio of the response of each target analyte to their surrogate standard. Factors are described in **Table S2**.

Table S4. Results of stability of target analytes in samples kept at room temperature

Day	Statistic	Toluene-d8	Sulfolane-d8	Benzene	Toluene	Ethylbenzene	m/p-Xylene	o-Xylene	Sulfolane
0	N	3	3	3	3	3	3	3	3
	Mean	1441613	726457	2.25	0.66	1.66	2.64	1.38	0.55
	SD	101057	16728	0.14	0.04	0.10	0.16	0.08	0.02
	RSD	7%	2%	6%	6%	6%	6%	6%	3%
3	N	3	3	3	3	3	3	3	3
	Mean	1143753	577020	2.30	0.66	1.60	2.51	1.35	0.50
	SD	67812	16330	0.05	0.02	0.04	0.07	0.03	0.01
	RSD	6%	3%	2%	3%	3%	3%	2%	3%
8	N	3	3	3	3	3	3	3	3
	Mean	1320023	609492	1.94	0.54	1.27	1.96	1.12	0.46
	SD	32754	29046	0.08	0.03	0.07	0.12	0.04	0.02
	RSD	2%	5%	4%	5%	6%	6%	4%	4%
14	N	3	3	3	3	3	3	3	3
	Mean	1167060	435533	1.77	0.48	1.06	1.64	1.00	0.51
	SD	67691	9500	0.06	0.02	0.03	0.05	0.03	0.02
	RSD	6%	2%	3%	3%	3%	3%	3%	4%
23	N	3	3	3	3	3	3	3	3
	Mean	1175603	440954	1.64	0.42	0.85	1.32	0.89	0.48
	SD	47252	36560	0.23	0.07	0.14	0.22	0.12	0.01
	RSD	4%	8%	14%	16%	17%	17%	13%	3%

Table S5. Results of stability of target analytes in samples kept at 4 °C

Day	Statistic	Toluene-d8	Sulfolane-d8	Benzene	Toluene	Ethylbenzene	m/p-Xylene	o-Xylene	Sulfolane
0	N	3	3	3	3	3	3	3	3
	Mean	1723903	358646	1.64	0.86	1.33	2.15	1.13	0.91
	SD	32454	5076	0.11	0.06	0.10	0.15	0.08	0.03
	RSD	2%	1%	7%	7%	7%	7%	7%	3%
3	N	3	3	3	3	3	3	3	3
	Mean	1647095	344945	1.72	0.89	1.36	2.20	1.16	0.94
	SD	86780	16258	0.13	0.07	0.11	0.17	0.08	0.05
	RSD	5%	5%	7%	8%	8%	8%	7%	6%
8	N	3	3	3	3	3	3	3	3
	Mean	1688260	361630	1.60	0.83	1.26	2.02	1.08	0.89
	SD	74115	19287	0.08	0.05	0.08	0.12	0.05	0.05
	RSD	4%	5%	5%	6%	6%	6%	5%	5%
14	N	3	3	3	3	3	3	3	3
	Mean	1361130	321169	1.92	0.97	1.38	2.29	1.26	0.99
	SD	196047	23679	0.30	0.15	0.24	0.34	0.19	0.09
	RSD	14%	7%	15%	15%	17%	15%	15%	9%
23	N	3	3	3	3	3	3	3	3
	Mean	1618825	383752	1.61	0.80	1.19	1.89	1.04	0.86
	SD	57163	13176	0.12	0.06	0.11	0.16	0.08	0.01
	RSD	4%	3%	8%	8%	9%	9%	7%	2%

Table S6. Results of analysis of water samples

Sample ID	Sample type	Toluene-d8 (area)	Sulfolane-d8 (area)	Benzene (µg/L)	Toluene (µg/L)	Ethylbenzene (µg/L)	m/p-Xylene (µg/L)	o-Xylene (µg/L)	Sulfolane (µg/L)
FS1	Surface water	2.43E+06	5.61E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS1D	Surface water	2.35E+06	5.91E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS2	Ground water	2.74E+06	5.99E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS2D	Ground water	2.41E+06	5.98E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS3	Ground water	2.65E+06	5.86E+05	N.D.	2.59E+01	N.D.	N.D.	N.D.	5.20E+02
FS3D	Ground water	2.54E+06	6.19E+05	N.D.	6.65E+00	N.D.	N.D.	N.D.	4.40E+02
FS4	Ground water	2.22E+06	5.46E+05	N.D.	N.D.	N.D.	N.D.	N.D.	1.02E+03
FS4D	Ground water	2.43E+06	6.41E+05	N.D.	N.D.	N.D.	N.D.	N.D.	9.62E+02
FS5	Ground water	1.70E+06	4.35E+05	N.D.	N.D.	N.D.	N.D.	N.D.	3.19E+01
FS6	Ground water	2.62E+06	5.38E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS7	Ground water	2.62E+06	5.61E+05	N.D.	N.D.	N.D.	N.D.	N.D.	8.38E+01
FS7D	Ground water	2.49E+06	6.24E+05	N.D.	N.D.	N.D.	N.D.	N.D.	8.57E+01
FS8	Ground water	2.45E+06	5.47E+05	N.D.	N.D.	N.D.	N.D.	N.D.	9.69E+00
FS9	Ground water	2.32E+06	5.50E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS9D	Ground water	2.18E+06	6.13E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS10	Ground water	2.25E+06	5.30E+05	N.D.	N.D.	N.D.	N.D.	N.D.	2.41E+02
FS10D	Ground water	2.59E+06	6.07E+05	N.D.	N.D.	N.D.	N.D.	N.D.	2.30E+02
FS11	Ground water	2.39E+06	5.53E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS11D	Ground water	2.78E+06	6.38E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS12	Ground water	2.66E+06	5.62E+05	N.D.	N.D.	N.D.	N.D.	N.D.	6.15E+01
FS12D	Ground water	2.75E+06	6.43E+05	N.D.	N.D.	N.D.	N.D.	N.D.	5.91E+01
FS13	Ground water	2.53E+06	5.61E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS13D	Ground water	2.38E+06	6.22E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS14	Ground water	2.25E+06	5.36E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS14D	Ground water	2.12E+06	5.99E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS15	Ground water	2.13E+06	5.34E+05	N.D.	N.D.	N.D.	9.35E+00	N.D.	3.86E+02
FS15D	Ground water	2.39E+06	6.21E+05	N.D.	N.D.	N.D.	positive	N.D.	3.72E+02
FS16	Ground water	2.23E+06	5.38E+05	N.D.	N.D.	N.D.	N.D.	N.D.	6.35E+02
FS16D	Ground water	2.61E+06	6.43E+05	N.D.	N.D.	N.D.	N.D.	N.D.	6.27E+02

FS17	Ground water	2.35E+06	5.40E+05	N.D.	N.D.	N.D.	N.D.	N.D.	positive
FS17D	Ground water	2.60E+06	6.35E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS18	Ground water	2.48E+06	5.18E+05	N.D.	N.D.	N.D.	N.D.	N.D.	3.71E+02
FS18D	Ground water	2.63E+06	6.36E+05	N.D.	N.D.	N.D.	N.D.	N.D.	3.20E+02
FS19	Ground water	2.52E+06	5.72E+05	N.D.	N.D.	N.D.	N.D.	N.D.	2.21E+03
FS19D	Ground water	2.65E+06	6.43E+05	N.D.	N.D.	N.D.	N.D.	N.D.	2.07E+03
FS20	Ground water	2.27E+06	5.49E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS20D	Ground water	2.43E+06	5.82E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS21	Ground water	2.32E+06	5.49E+05	N.D.	N.D.	N.D.	N.D.	N.D.	1.59E+03
FS21D	Ground water	2.63E+06	6.35E+05	N.D.	N.D.	N.D.	N.D.	N.D.	1.50E+03
FS22	Surface water	2.32E+06	5.24E+05	N.D.	N.D.	N.D.	N.D.	N.D.	positive
FS22D	Surface water	1.96E+06	5.91E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS23	Ground water	2.50E+06	6.09E+05	N.D.	N.D.	N.D.	N.D.	N.D.	5.65E+02
FS23D	Ground water	2.34E+06	5.38E+05	N.D.	N.D.	N.D.	N.D.	N.D.	6.06E+02
FS24	Ground water	2.55E+06	6.17E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS24D	Ground water	2.09E+06	5.37E+05	N.D.	2.55E+0 1	N.D.	N.D.	N.D.	N.D.
FS25	Ground water	2.38E+06	5.73E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS25D	Ground water	2.52E+06	6.24E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS26	Surface water	2.47E+06	5.76E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS26D	Surface water	2.93E+06	6.27E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS27	Ground water	2.28E+06	5.68E+05	N.D.	N.D.	N.D.	N.D.	N.D.	positive
FS27D	Ground water	2.56E+06	6.34E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS28	Ground water	2.65E+06	5.85E+05	N.D.	N.D.	N.D.	N.D.	N.D.	9.88E+02
FS28D	Ground water	2.65E+06	6.52E+05	N.D.	N.D.	N.D.	N.D.	N.D.	8.12E+02
FS29	Surface water	2.73E+06	6.02E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS29D	Surface water	2.63E+06	6.13E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS30	Surface water	2.41E+06	5.61E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS30D	Surface water	2.30E+06	5.86E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS31	Surface water	2.29E+06	5.56E+05	N.D.	N.D.	N.D.	N.D.	N.D.	8.89E+00
FS31D	Surface water	2.28E+06	6.17E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS32	Surface water	2.18E+06	5.48E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS32D	Surface water	2.74E+06	6.23E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

FS33	Ground water	2.55E+06	5.92E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS33D	Ground water	2.32E+06	6.00E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS34	Ground water	2.46E+06	6.03E+05	N.D.	N.D.	N.D.	N.D.	N.D.	7.79E+02
FS34D	Ground water	2.53E+06	6.49E+05	N.D.	N.D.	N.D.	N.D.	N.D.	4.20E+01
FS35	Surface water	2.44E+06	5.41E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS35D	Surface water	2.41E+06	6.53E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS36	Ground water	2.52E+06	5.67E+05	N.D.	1.87E+0 1	N.D.	N.D.	N.D.	N.D.
FS36D	Ground water	2.18E+06	6.26E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS37	Ground water	2.60E+06	5.71E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS37D	Ground water	2.41E+06	6.39E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS38	Surface water	2.65E+06	5.72E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS38D	Surface water	2.76E+06	6.27E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS39	Ground water	2.54E+06	5.76E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS39D	Ground water	2.45E+06	5.89E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS40	Ground water	2.58E+06	5.53E+05	N.D.	9.97E+0 1	N.D.	N.D.	N.D.	N.D.
FS40D	Ground water	2.66E+06	6.47E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS41	Surface water	2.58E+06	5.56E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS41D	Surface water	2.77E+06	6.52E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS42	Ground water	2.62E+06	5.65E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS42D	Ground water	2.05E+06	6.10E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS43	Ground water	2.64E+06	5.73E+05	N.D.	1.35E+0 1	N.D.	N.D.	N.D.	N.D.
FS43D	Ground water	2.50E+06	6.61E+05	N.D.	6.01E+0 0	N.D.	N.D.	N.D.	N.D.
FS44	Surface water	2.47E+06	5.99E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS44D	Surface water	2.75E+06	6.57E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS45	Ground water	2.87E+06	6.07E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS45D	Ground water	2.51E+06	6.21E+05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
FS46	Ground water	2.64E+06	6.09E+05	N.D.	2.03E+0 2	N.D.	8.36E+00	N.D.	9.34E+02
FS46D	Ground water	2.62E+06	6.35E+05	N.D.	1.93E+0 2	N.D.	6.85E+00	N.D.	7.61E+02
FS47	Surface water	2.67E+06	6.05E+05	N.D.	N.D.	N.D.	N.D.	N.D.	1.12E+03
FS47D	Surface water	2.48E+06	6.16E+05	N.D.	N.D.	N.D.	N.D.	N.D.	1.07E+03
FS48	Ground water	2.63E+06	5.92E+05	N.D.	N.D.	N.D.	N.D.	N.D.	3.90E+02
FS48D	Ground water	2.73E+06	6.43E+05	N.D.	N.D.	N.D.	N.D.	N.D.	4.61E+02

FS49	Ground water	2.53E+06	5.97E+05	N.D.	5.86E+0 1	N.D.	N.D.	N.D.	1.52E+03
FS49D	Ground water	2.48E+06	6.19E+05	N.D.	1.64E+0 1	N.D.	N.D.	N.D.	8.64E+02
FS50	Surface water	9.43E+05	5.68E+05	N.D.	N.D.	N.D.	N.D.	N.D.	positive
FS50D	Surface water	2.69E+06	6.03E+05	N.D.	N.D.	N.D.	N.D.	N.D.	positive

*Samples marked with 'D' were collected from the same location, but were not always true duplicates (see the paper).