Supplementary Information

A Cyanoalkyl Silicone GC Stationary-Phase Polymer as an Extractant for Dispersive Liquid–Liquid Microextraction

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1. The general DLLME procedure:

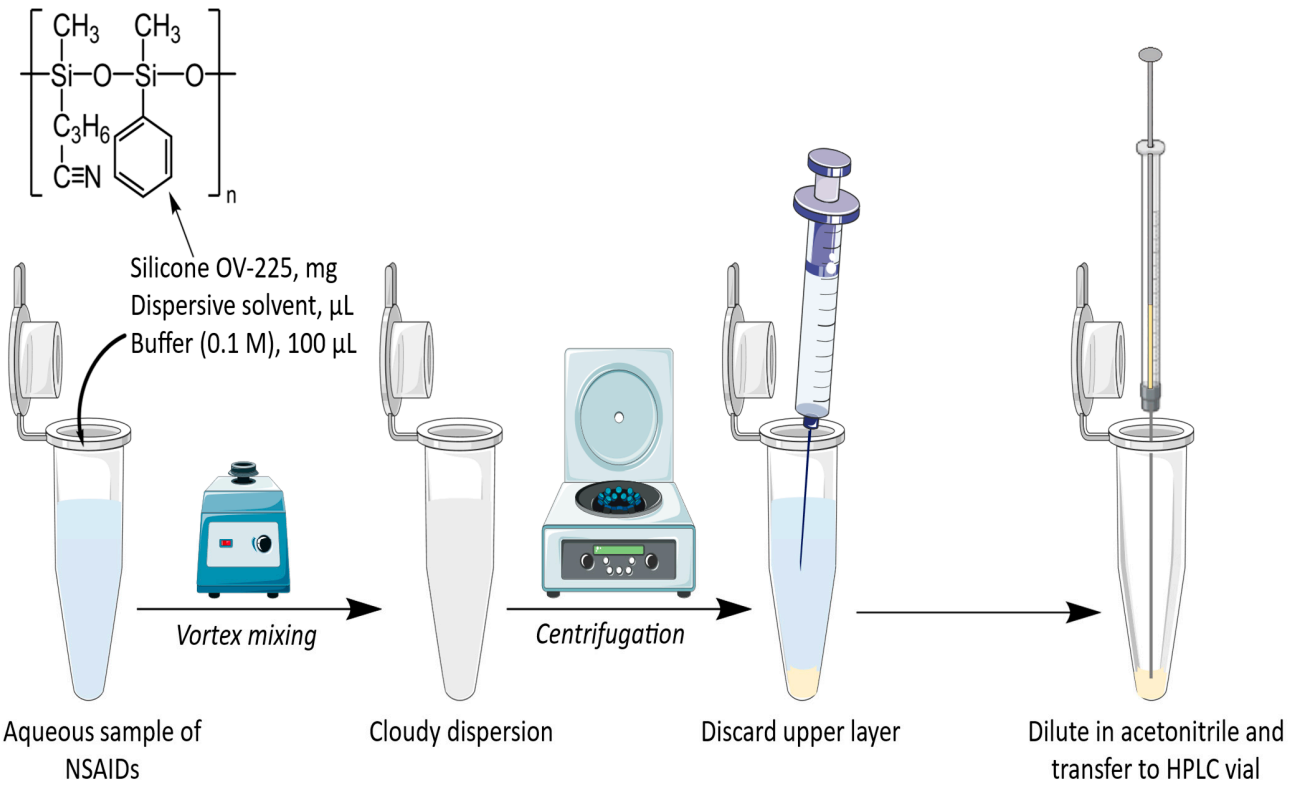

Figure S1. A general schematic overview of the dispersive liquid-liquid micro-extraction procedure.

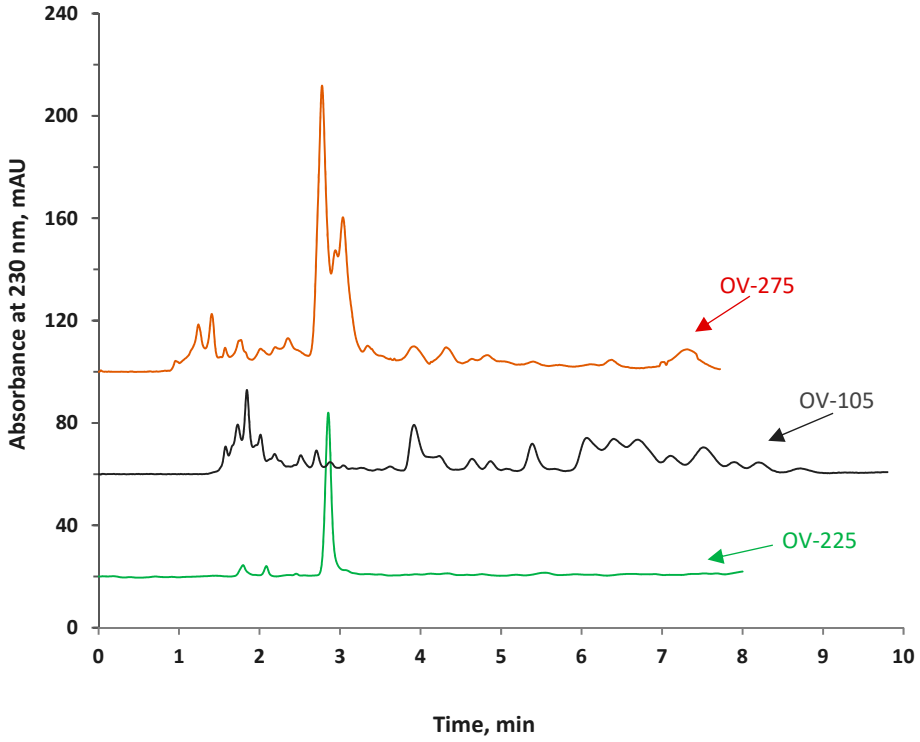

Figure S2. Dispersive liquid-liquid micro-extraction of blank ultrapure water samples using the investigated cyanopropyl polysiloxanes: OV-275, OV-105, and OV-225.

2. *The 2^4 full factorial design:*

Table S1: The 2^4-FFD experiment of independent DLLME variables and their corresponding responses.

Std Order	Run Order	Independent DLLME variables				Responses (Enrichment Factor)			
		Extractant mass, mg	Disperser solvent volume, µL	pH	Extraction time, sec	NAP	DIF	IND	IBU
5	1	20	50	8	10	0.16	0.61	0.31	0.45
27	2	20	150	2.2	120	10.73	12.60	13.80	13.69
9	3	20	50	2.2	120	9.69	13.60	11.89	10.49
11	4	20	150	2.2	120	*	*	*	*
12	5	50	150	2.2	120	9.22	11.45	10.55	10.28
1	6	20	50	2.2	10	9.82	13.74	12.32	10.90
8	7	50	150	8	10	0.06	0.11	0.37	0.46
25	8	20	50	2.2	120	*	*	*	*
6	9	50	50	8	10	0.22	0.20	0.48	0.28
2	10	50	50	2.2	10	5.68	8.24	7.88	7.52
22	11	50	50	8	10	0.22	0.16	0.49	0.33
4	12	50	150	2.2	10	7.02	9.70	8.79	8.37
13	13	20	50	8	120	0.06	0.21	0.03	0.73
19	14	20	150	2.2	10	9.33	14.31	11.60	10.67
29	15	20	50	8	120	0.02	0.22	0.03	0.73
24	16	50	150	8	10	0.02	0.05	0.44	0.36
28	17	50	150	2.2	120	8.98	10.90	10.06	9.92
16	18	50	150	8	120	0.10	0.11	0.42	0.53
7	19	20	150	8	10	0.00	0.01	0.10	0.07
23	20	20	150	8	10	0.09	0.23	0.21	0.29
20	21	50	150	2.2	10	7.44	9.94	9.17	8.91
17	22	20	50	2.2	10	*	*	*	*
15	23	20	150	8	120	0.24	0.20	0.38	0.33
32	24	50	150	8	120	0.08	0.17	0.21	0.42
10	25	50	50	2.2	120	8.45	10.10	10.05	10.23
3	26	20	150	2.2	10	8.25	12.54	10.44	9.46
14	27	50	50	8	120	0.17	0.14	0.55	0.30
31	28	20	150	8	120	0.24	0.22	0.34	0.44
18	29	50	50	2.2	10	4.49	6.65	6.47	6.32
30	30	50	50	8	120	0.15	0.13	0.28	0.17
21	31	20	50	8	10	0.16	0.61	0.31	0.45
26	32	50	50	2.2	120	10.54	11.01	10.41	9.44

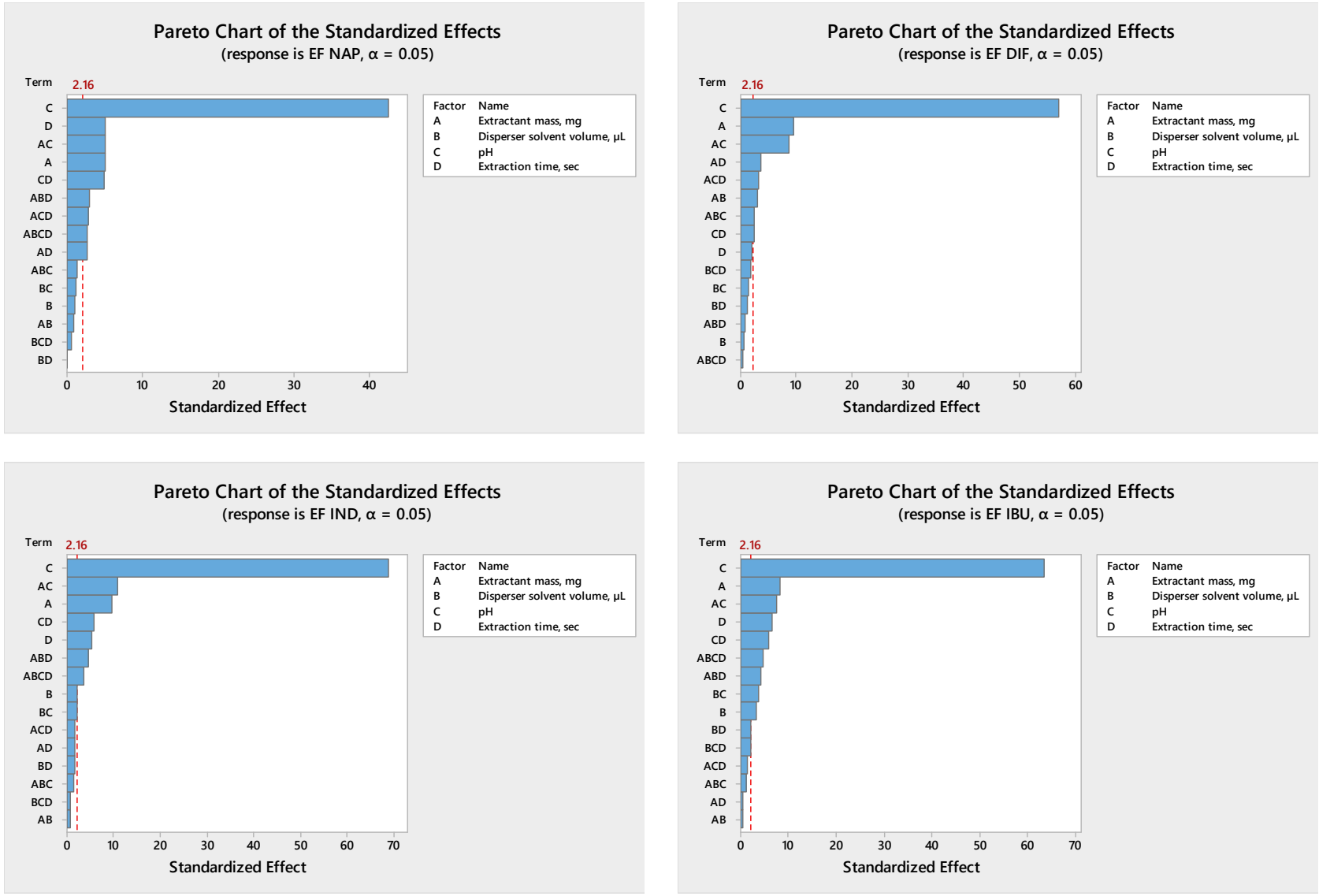

Figure S3: Pareto charts from the 2^4-FFD showing the main and interaction effects of the investigated experimental variables on the preconcentration of the studied NSAIDs. The dotted red line represents Bonferroni's statistical limit of significance.

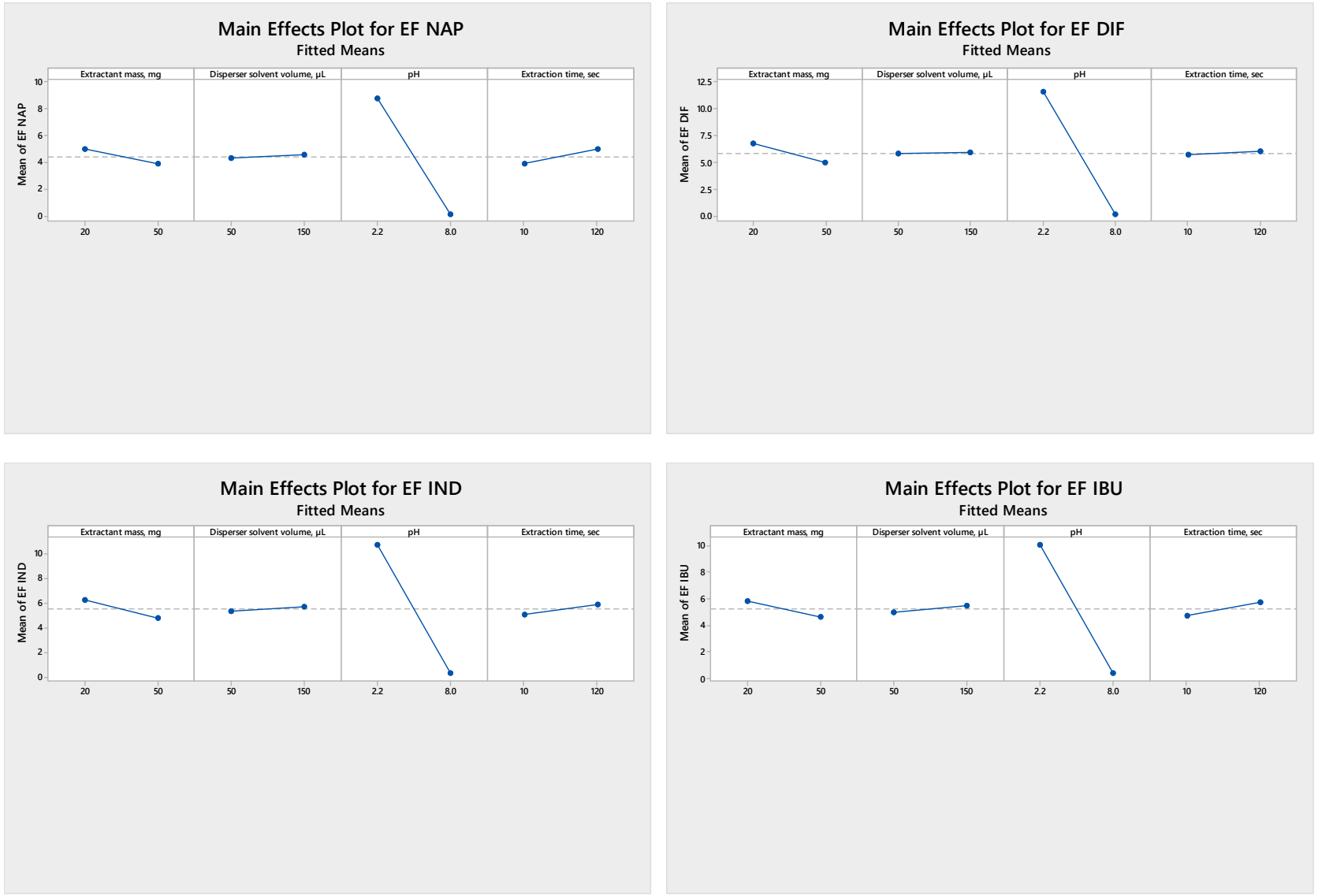

Figure S4: Main effects plots from the 2^4-FFD showing the main effects of the investigated experimental variables on the pre-concentration of the studied NSAIDs.

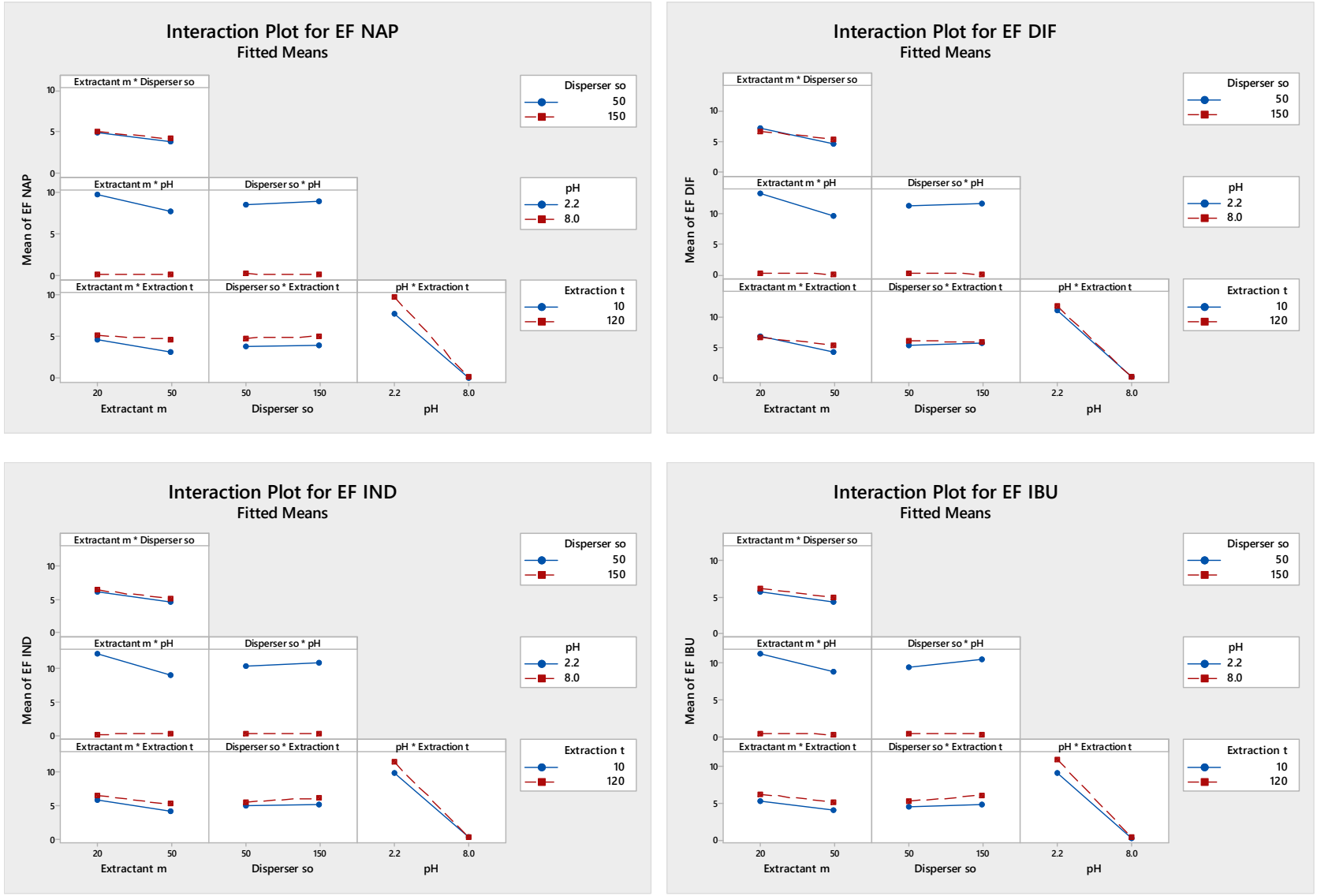

Figure S5: Interaction plots from the 2^4-FFD showing the interaction effects of the investigated experimental variables on the pre-concentration of the studied NSAIDs.

3. The Face-centered central composite design:

Table S2: The FCCD experiment of independent DLLME variables and their corresponding responses.

Run Order	Pt. Type	Blocks	Independent DLLME variables			Responses (Enrichment Factor)			
			Extractant mass (mg)	pH	Extraction time (sec)	NAP	DIF	IND	IBU
1	1 Cube points	1	10	2.2	60	21.026	29.46	31.457	27.179
2	1 Cube points	1	30	3.4	60	16.392	18.243	22.556	19.625
3	1 Cube points	1	30	2.2	180	20.922	24.835	25.984	23.661
4	1 Cube points	1	10	3.4	180	26.889	29.856	38.279	34.204
5	0 Center points in cube	1	20	2.8	120	25.608	30.611	32.511	30.261
6	0 Center points in cube	1	20	2.8	120	23.229	26.941	30.347	27.465
7	1 Cube points	2	30	2.2	60	20.317	23.625	23.401	22.838
8	1 Cube points	2	10	3.4	60	18.72	17.621	29.157	26.15
9	1 Cube points	2	10	2.2	180	24.894	34.084	34.724	31.713
10	1 Cube points	2	30	3.4	180	16.467	16.402	18.503	18.177
11	0 Center points in cube	2	20	2.8	120	22.889	27.087	30.114	27.122
12	0 Center points in cube	2	20	2.8	120	22.531	20.867	29.621	27.336
13	-1 Axial points, α = 1	3	10	2.8	120	21.907	27.026	32.29	26.301
14	-1 Axial points, α = 1	3	30	2.8	120	14.972	15.891	18.875	17.003
15	-1 Axial points, α = 1	3	20	2.2	120	17.999	21.573	22.945	20.841
16	-1 Axial points, α = 1	3	20	3.4	120	18.099	18.902	23.063	20.963
17	-1 Axial points, α =1	3	20	2.8	60	22.99	27.795	31.654	27.796
18	-1 Axial points, α =1	3	20	2.8	180	*	*	*	*
19	1 Cube points	1	10	2.2	60	27.774	37.334	38.698	34.006
20	1 Cube points	1	30	3.4	60	18.592	20.313	24.554	21.798
21	1 Cube points	1	30	2.2	180	20.784	24.567	25.617	23.308
22	1 Cube points	1	10	3.4	180	27.517	29.666	36.854	33.744
23	0 Center points in cube	1	20	2.8	120	22.328	29.01	30.818	27.122
24	0 Center points in cube	1	20	2.8	120	25.175	30.347	33.615	29.982
25	1 Cube points	2	30	2.2	60	17.125	22.392	22.852	20.113
26	1 Cube points	2	10	3.4	60	23.598	25.299	34.497	30.313
27	1 Cube points	2	10	2.2	180	24.471	32.998	34.811	30.563
28	1 Cube points	2	30	3.4	180	16.314	16.721	20.682	18.843
29	0 Center points in cube	2	20	2.8	120	23.962	28.016	30.202	28.284
30	0 Center points in cube	2	20	2.8	120	22.758	27.286	29.59	27.344
31	-1 Axial points, α = 1	3	10	2.8	120	26.41	31.874	37.415	32.55
32	-1 Axial points, α = 1	3	30	2.8	120	17.129	18.477	20.473	18.849
33	-1 Axial points, α = 1	3	20	2.2	120	19.73	23.815	24.975	22.606
34	-1 Axial points, α = 1	3	20	3.4	120	18.618	19.419	23.087	21.144
35	-1 Axial points, α = 1	3	20	2.8	60	22.609	25.669	29.532	26.322
36	-1 Axial points, α = 1	3	20	2.8	180	20.985	23.55	25.974	23.752

Table S3: Mathematical model fitting for each response.

Response	*p*-value (lack-of-fit)	R^2_{adj}
$EF_{(IND)}$	0.140	0.8640
$EF_{(IBU)}$	0.363	0.8365
$EF_{(NAP)}$	0.420	0.7177
$EF_{(DIF)}$	0.309	0.7689

4. The salting-out effect on enrichment factors:

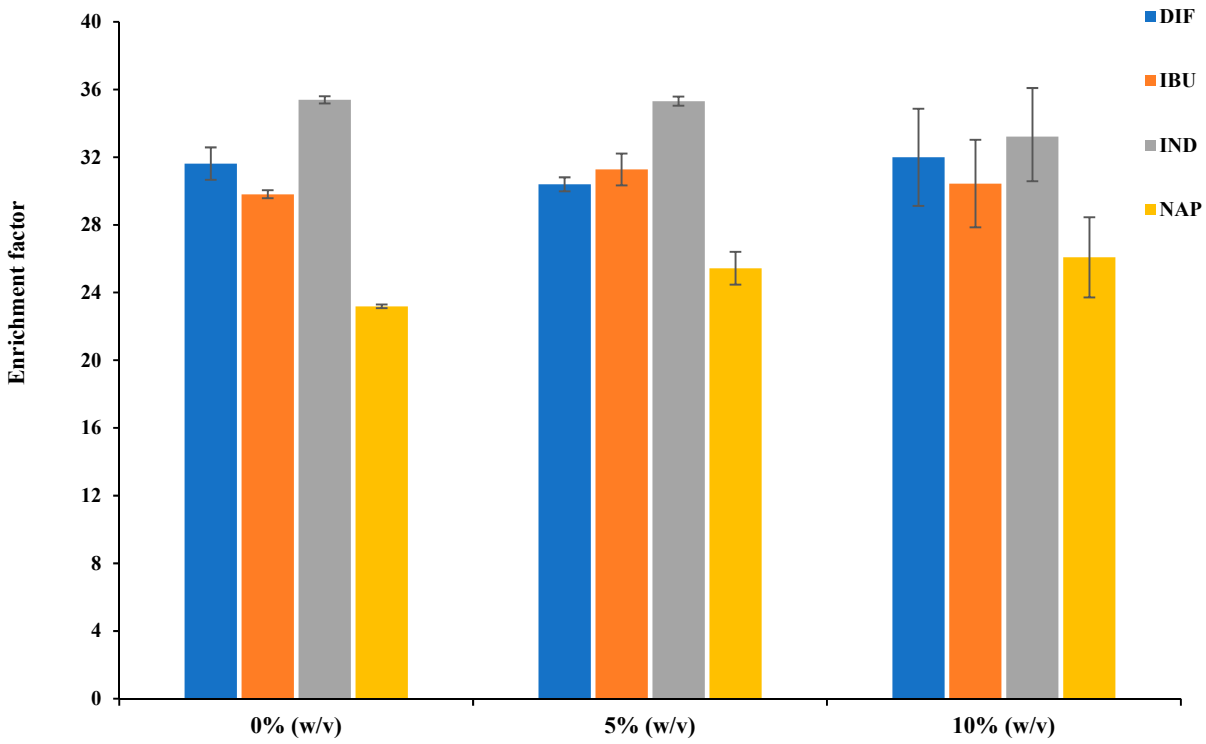

Figure S6. Salting-out effect on enrichment factors of the studied analytes. Note: the *x*-axis is the NaCl concentration in the sample solution before extraction. n=2.

Table S4: Comparison with other reported liquid chromatographic methods.
SPE: solid-phase extraction; MISPE: molecularly imprinted SPE; MSPE: magnetic SPE; IL-DLLME: ionic liquid dispersive liquid-liquid microextraction; FLD: fluorescence detection; LOQ: limit of quantitation; RSD: relative standard deviation. NAP: naproxen sodium; DIF: diflunisal; IBU: ibuprofen; IND: indomethacin.

Extraction method	Instrument	Matrix	Sample volume (mL)	Extraction time (min)	Analyte	LOQ (ng/mL)	RSD (%)	Reference
SPE	HPLC-UV	Water, urine	-	10	Aspirin, naproxen, ibuprofen	<1.3	<3.5	[14]
MISPE	HPLC-UV	Tap water	10.0	30	Ibuprofen	75	-	[15]
MISPE	HPLC-UV	River water	50.0	160	Naproxen, ibuprofen, diclofenac	0.5 (NAP) 3.33	<10.3	[16]
MSPE	HPLC/UV	Tap, river, and wastewater	10.0	Extraction 2.4;	Naproxen, diclofenac, mefenamic	2.9 (NAP)	<3.8	[17]
MSPE	UHPLC-MS/MS	Lake water, wastewater	50.0	Extraction 45.0;	Naproxen, meloxicam, carprofen, diclofenac,	0.3 (NAP)	7.2	[18]
IL-DLLME	LC-MS/MS	Wastewater	10.0	5	Naproxen, ibuprofen, ketoprofen	0.142 (NAP)	<10	[19]
IL-DLLME	HPLC/UV/FLD	Tap and river water	5.0	0.5	ibuprofen, diclofenac, ketoprofen	137 (IBU)	<2	[20]
(OV-225 polymer)-DLLME	HPLC-UV	Tap water	1.5	3	Naproxen	5.0	0.7	This work
					Diflunisal	10.0	6.7	
					Indomethacin	25.0	0.4	
					Ibuprofen	75	1.9	

Bibliometric Analysis Search Key

Chlorinated hydrocarbons

	Solvent	Search key	Results
1.	Trichloromethane	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS (chloroform) OR ABS (trichloromethane)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	294
2.	Tetrachloromethane	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("carbon tetrachloride") OR ABS (tetrachloromethane)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	183
3.	Dichloromethane	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("methylene chloride") OR ABS (dichloromethane)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	78
4.	Tetrachloroethane	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("ethylene tetrachloride") OR ABS (tetrachloroethane)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	32
5.	Tetrachloroethene	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("tetrachloroethylene") OR ABS (tetrachloroethene) OR ABS (perchloroethylene)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	23
6.	Dichloroethane	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("ethylene dichloride") OR ABS (dichloroethane)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	18
7.	Trichloroethane	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("ethylene trichloride") OR ABS (trichloroethane)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	25
8.	Trichloroethene	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("trichloroethylene") OR ABS (trichloroethene)) AND ABS (extract*) AND NOT	15

		TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	
9.	**Dichloroethene**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("dichloroethylene") OR ABS (dichloroethene)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	2
10.	**Chlorobenzene and dichlorobenzenes**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("chlorobenzene") OR ABS (monochlorobenzene) OR ABS (dichlorobenzene)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	78

Alkanes

	Solvent	Search key	Results
1.	**Pentane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("pentane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	2
2.	**Hexane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("hexane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	25
3.	**Heptane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("heptane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	4
4.	**Octane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("octane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	4
5.	**Isooctane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("isooctane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	2
6.	**Dodecane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("dodecane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	5
7.	**Hexadecane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("hexadecane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	6
8.	**cyclohexane**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("cyclohexane" W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	6

Alcohols

	Solvent	Search key	Results
1.	**Hexanol including 2-ethyl-1-hexanol and 5-methyl-1-hexanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (hexanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	9
2.	**Heptanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (heptanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	7
3.	**Octanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (octanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	67
4.	**Nonanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (nonanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	2
5.	**Decanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (decanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	9
6.	**Undecanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (undecanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	80
7.	**Dodecanol**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS (dodecanol W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	58

Other Solvents

	Solvent	Search key	Results
1.	**Ionic liquids**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("ionic liquid") AND ABS (extract*) AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))	455
2.	**Deep eutectic solvents**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("deep eutectic") AND ABS (extract*)) AND (LIMIT-TO (DOCTYPE , "ar"))	213
3.	**Acetates**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("acetate") AND ABS (extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	53
4.	**Ether**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("ether") AND ABS (extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND NOT (determination W/5 ether) AND (LIMIT-TO (DOCTYPE , "ar"))	8
5.	**Ketone**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND ABS ("ketone") AND ABS (extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND NOT (determination W/5 ketone) AND (LIMIT-TO (DOCTYPE , "ar"))	5
6.	**Carbon disulfide**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS ("carbon disulfide") OR ABS ("carbon disulphide")) AND ABS (extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND (LIMIT-TO (DOCTYPE , "ar"))	5
7.	**Toluene**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS (toluene W/10 extract*) OR ABS (methylbenzene W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND NOT ABS (toluene W/6 determination)) AND (LIMIT-TO (DOCTYPE , "ar"))	46
8.	**xylene**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS (xylene W/10 extract*) OR ABS (dimethylbenzene W/10 extract*) OR ABS (methyltoluene W/10 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY (eutectic) AND NOT ABS (xylene W/6 determination)) AND (LIMIT-TO (DOCTYPE , "ar"))	6
9.	**Brominated solvents**	((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS (*bromo* W/8 extract*)) AND NOT TITLE-ABS-KEY ("ionic liquid")	23

		AND NOT TITLE-ABS-KEY (eutectic) AND NOT ABS (*bromo* W/6 determination)) AND (LIMIT-TO (DOCTYPE , "ar"))	

Polymer

((TITLE-ABS-KEY ("dispersive liquid-liquid microextraction") OR TITLE-ABS-KEY (dllme)) AND (ABS (poly*)) AND ABS (extract*) AND NOT TITLE-ABS-KEY ("ionic liquid") AND NOT TITLE-ABS-KEY ("polycyclic") AND NOT TITLE-ABS-KEY (eutectic)) AND (LIMIT-TO (DOCTYPE , "ar"))