

The diagram illustrates a five-step sample preparation workflow for HPLC analysis:

- vortexed mixture**: 40 mL sample, 1.2 mL 2-propanol, 67 mg C18, 33 mg SAX, pH 4.2.
- extraction**: 30 min fast shaking.
- settling**: 30 min twitching.
- sample removal**: Centrifugation Decantation, followed by the addition of +0.75 mL acetonitrile.
- desorption**: 30 min ultrasound bath.

The final step is **extract filtration & analysis**, which results in a chromatogram showing four distinct peaks labeled 1, 2, 3, and 4.

Chemical structures of the four drugs are shown below:

- Moxifloxacin (MOX)**: A fluoroquinolone antibiotic structure.
- Rosuvastatin (ROS)**: A statin structure featuring a pyrimidine ring and a carboxylic acid group.
- Simvastatin (SIM)**: A statin structure featuring a naphthalene ring system and a carboxylic acid group.
- Telmisartan (TEL)**: An angiotensin II receptor antagonist structure featuring a benzimidazole ring system and a carboxylic acid group.

sum of recoveries (%)

MOX, ROS, SIM, TEL

C18, hydra C18, or C8

SDB - Styrene-divinylbenzene

CRS - Chromasorb W-AW DCMS

Mix C18/strogr anion exchanger (SAX)

sorbent mixture (m = 60 mg), pH

Sorbent Mixture <th>MOX (%)</th> <th>ROS (%)</th> <th>SIM (%)</th> <th>TEL (%)</th> <th>Total (%)</th>	MOX (%)	ROS (%)	SIM (%)	TEL (%)	Total (%)
C18_pH7	25	65	45	65	200
C18_pH2	10	15	60	55	130
C8_pH7	25	75	45	65	210
C8_pH2	15	10	70	55	150
C18OH_pH7	10	55	25	50	140
C18OH_pH2	10	55	30	50	145
CRS_pH7	0	0	80	60	140
CRS_pH2	0	0	40	40	80
SDB_pH7	20	10	20	50	100
SDB_pH2	15	5	45	35	100
C18+SAX_pH7	35	50	40	25	150
C18+SAX_pH2	10	60	50	30	150
C18+SAX_pH4	25	65	100	50	240
C8+SAX_pH7	20	55	50	55	180
C8+SAX_pH2	5	65	65	55	190
C8+SAX_pH4	15	55	100	50	220

Sorbent Load (XX mg C18 + YY mg SAX)	MOX (%)	ROS (%)	SIM (%)	TEL (%)
27+13	55	28	20	54
40+20	63	48	27	60
53+27	74	45	54	63
67+33	68	47	70	55

Figure 10: Recovery (%) for MOX, ROS, SIM and TEL under various Ext/Twi/Des (min) conditions. The chart shows that recovery is generally higher for MOX and SIM compared to ROS and TEL. The 30/30/30 condition (green line) shows a significant drop in recovery for TEL compared to the other conditions.

Condition	Ext/Twi/Des (min)	Recovery (%)
MOX	30/30/15	95
	30/15/30	98
	30/15/15	85
	15/15/30	99
	15/30/15	83
	15/30/30	110
ROS	30/30/15	72
	30/15/30	75
	30/15/15	68
	15/15/30	75
	15/30/15	68
	15/30/30	68
SIM	30/30/15	62
	30/15/30	78
	30/15/15	67
	15/15/30	78
	15/30/15	70
	15/30/30	77
TEL	30/30/15	49
	30/15/30	65
	30/15/15	53
	15/15/30	53
	15/30/15	49
	15/30/30	55

Figure 10: Recovery of chemical mixtures. The chart displays the recovery percentage for eight different chemical mixtures across four target compounds: MOX, ROS, SIM, and TEL. The y-axis represents recovery in percent, ranging from 0 to 140. The x-axis lists the target compounds. The legend identifies the mixtures by color: methanol (blue), methanol/formic acid (orange), methanol/acetonitrile (grey), acetonitrile/methanol/formic acid (yellow), acetonitrile (light blue), acetonitrile/water (green), acetonitrile/acetone (dark blue), and acetonitrile/formic acid (light green line with circles). The acetonitrile/formic acid mixture shows the highest recovery for MOX and ROS, while the methanol/formic acid mixture shows the highest recovery for MOX. For SIM and TEL, the recovery is generally lower, with the acetonitrile/water mixture showing the highest recovery for SIM and the acetonitrile/acetone mixture showing the highest recovery for TEL.

Target Compound	methanol	methanol/formic acid	methanol/acetonitrile	acetonitrile/methanol/formic acid	acetonitrile	acetonitrile/water	acetonitrile/acetone	acetonitrile/formic acid
MOX	65	115	40	100	30	35	38	98
ROS	42	68	50	52	72	40	62	75
SIM	45	58	52	55	60	53	54	78
TEL	32	34	32	30	40	40	50	65

Figure 1 is a combined bar and line chart. The x-axis lists four analytes: MOX, ROS, SIM, and TEL. The left y-axis represents 'recovery (%)' ranging from 0 to 120. The right y-axis represents 'matrix effect (%)' ranging from 0 to 180. For each analyte, there are three bars representing different water matrices: ultrapur water (orange), seawater (yellow), and river water (green). Additionally, there are two lines with markers representing the matrix effect (ME) for seawater (dashed line with solid circles) and river water (dotted line with solid circles). Error bars are present on the recovery bars. The chart shows that recovery is generally higher in ultrapur water and lower in seawater and river water, with the most significant difference seen for MOX. The matrix effect is highest for MOX in seawater and decreases for the other analytes.

Analyte	Ultrapur water (%)	Seawater (%)	River water (%)	ME (seawater) (%)	ME (river water) (%)
MOX	~84	~66	~73	~104	~31
ROS	~64	~108	~71	~10	~10
SIM	~66	~92	~80	~1	~8
TEL	~52	~32	~29	~4	~10

Optimized method had recoveries between 50 and 80% reaching > 25 preconcentration factors. Recoveries obtained in seawater/river water were different compared to ultrapure water suggesting the impact of the ionic strength. Repeatability was < 5% RSD. Matrix effects were below 20% except for MOX (the most polar & first-eluting analyte) that suffered up to 100% signal enhancement in seawater.

DMSPE-HPLC-UV is a promising & simple procedure with high multiplexing capacity. It is applicable for fast extraction & preconcentration of analytes in water analysis.