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Microextraction of fluoroquinolones from biological fluids using supramolecular solvents based on di-(2-ethylhexyl)-phosphoric acid salt

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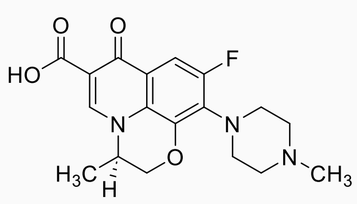
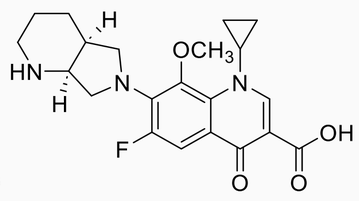
Relevance



Ofloxacin



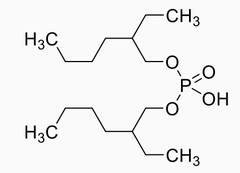
Moxifloxacin

Fluoroquinolones are used in the treatment of infectious and inflammatory diseases, meningitis, gonorrhea, sinusitis, tuberculosis, pneumonia, chronic bronchitis, chlamydia, etc.

In the context of personalized medicine, the monitoring of the content of fluoroquinolones in biological fluids is required to take into account the individual characteristics of the human body during pharmacotherapy and to increase its safety and effectiveness, thereby reducing the likelihood of serious side effects

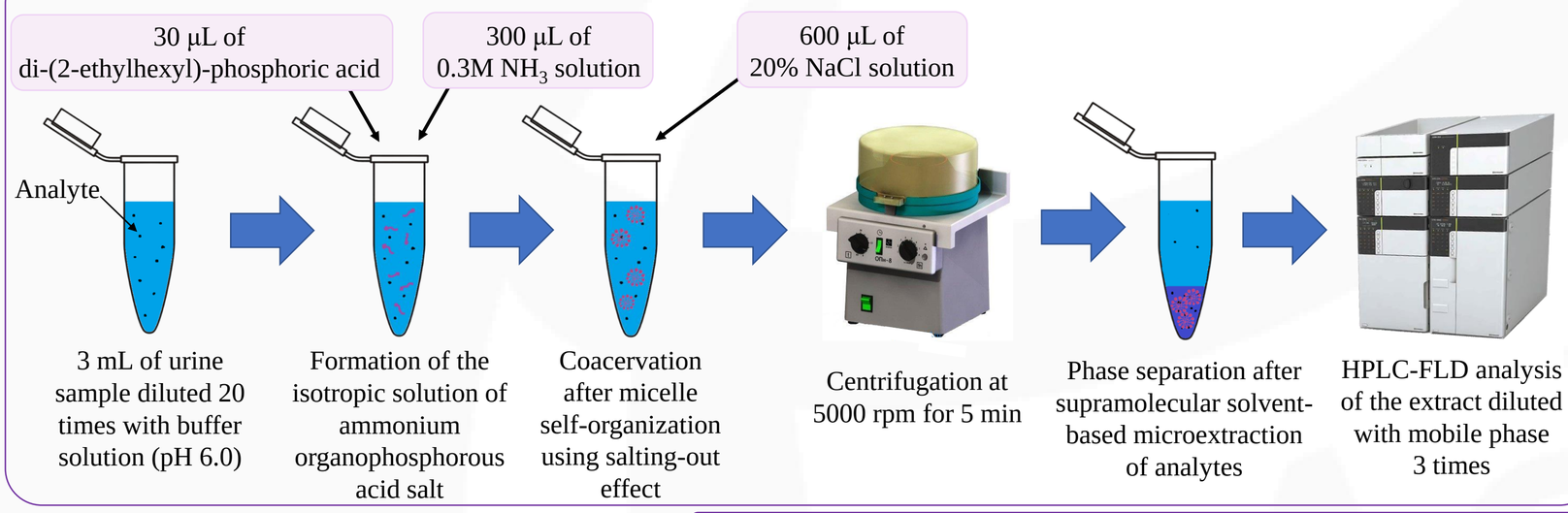
Aim:

To develop a new approach for the extraction and preconcentration of two fluoroquinolones (ofloxacin and moxifloxacin) from biological fluid by supramolecular solvent-based microextraction using di-(2-ethylhexyl)-phosphoric acid ammonium salt as an amphiphile



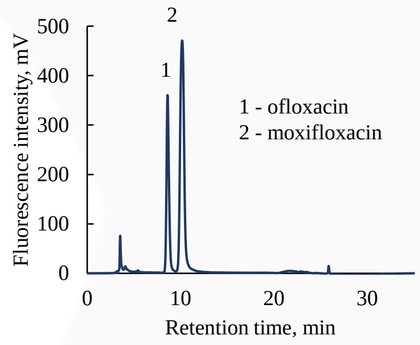
di-(2-ethylhexyl)-phosphoric acid

Graphical representation of microextraction procedure

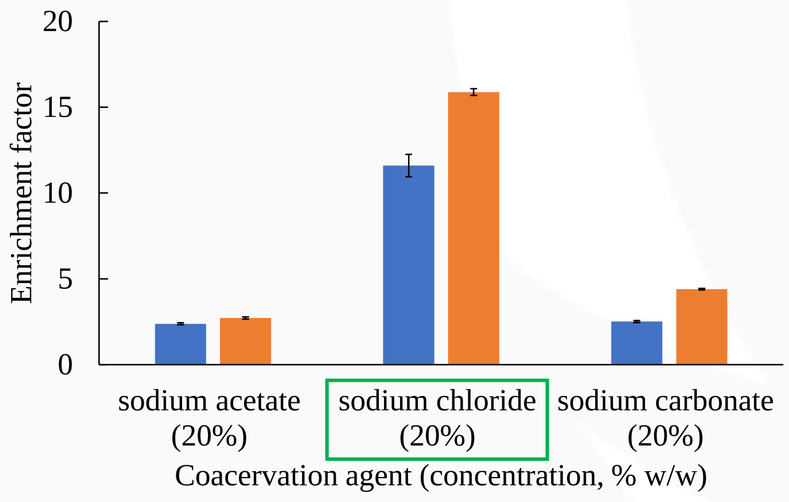


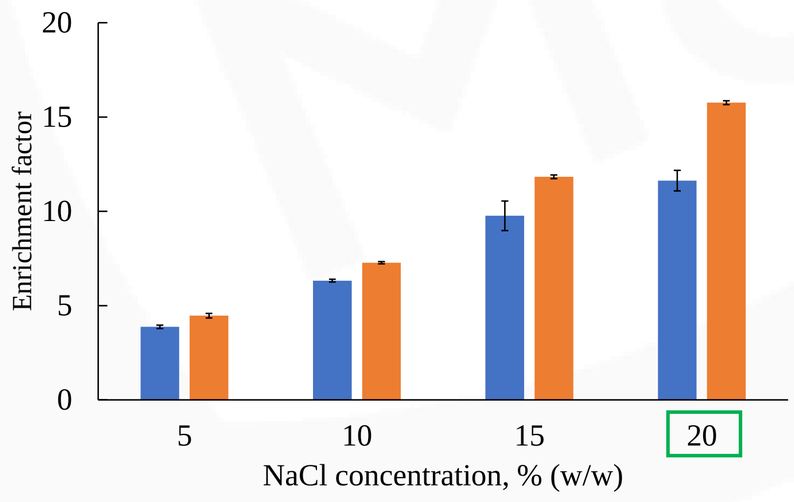
HPLC-FLD analysis conditions

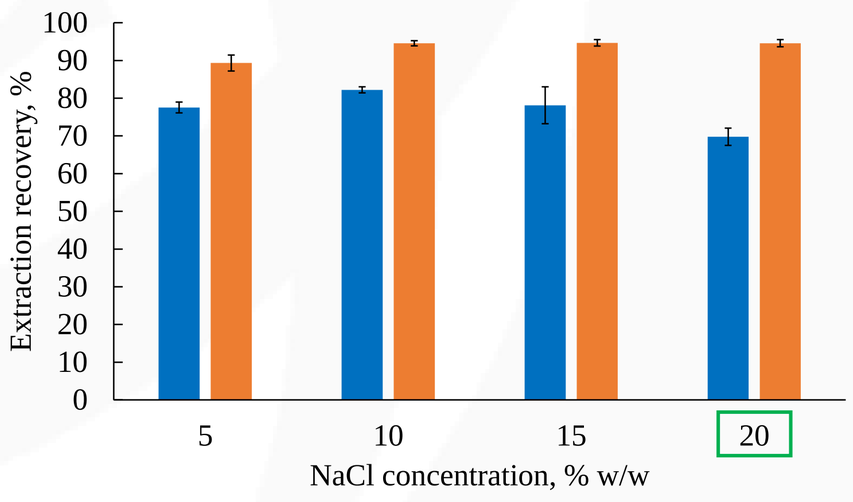
- Supelco C18 column (250 mm×4.6 mm, 5 μm);
- mobile phase: 50 mM phosphate buffer solution with 5 mM of Na_2EDTA (pH 6.5) (A) and CH_3OH (B);
- flow rate: 0.8 mL min^{-1};
- oven temperature: 40 °C;
- gradient (B): 50% → 80%;
- fluorescence excitation and emission wavelengths: 293 and 502 nm;
- duration of the chromatographic analysis: 35 min.

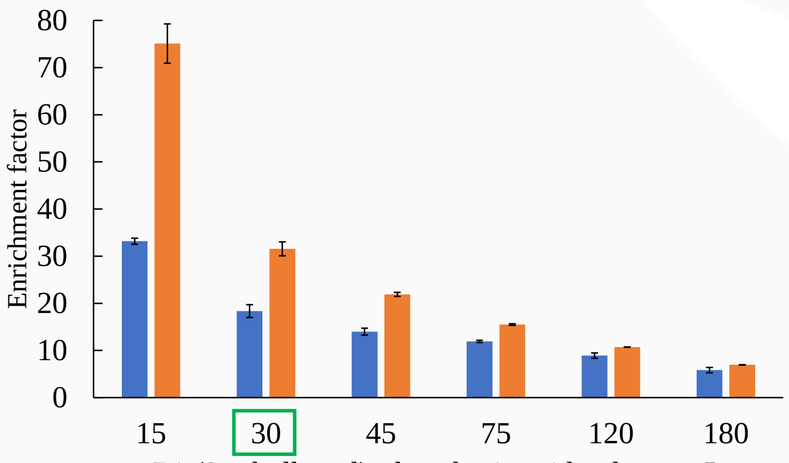


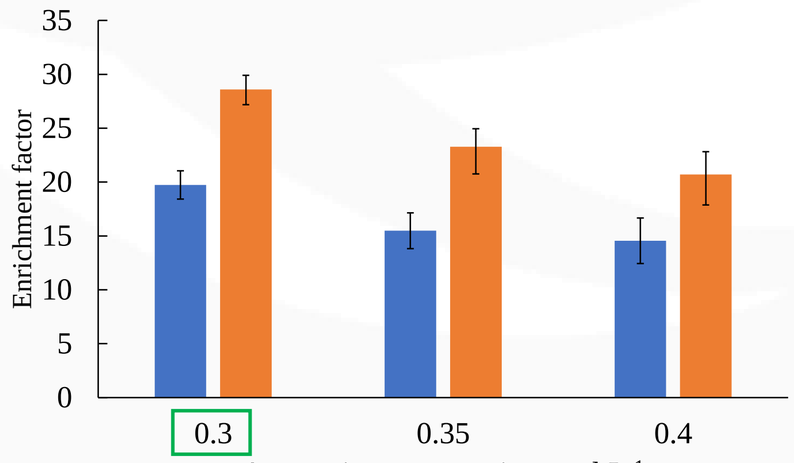
Optimization of extraction conditions and characterization of the extraction system

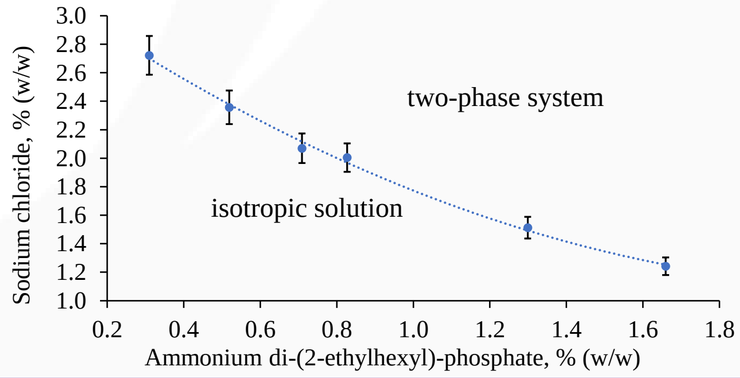












Physico-chemical properties of the supramolecular solvent

Phase location	Water concentration, % (w/w)	Volume, μL	Density, kg m^{-3}	Dynamic viscosity, mPa·s	pH
Bottom	52.89±0.08	52±10	1046±19	44.7±1.2	5.47±0.10

Validation of the proposed procedure

Parameter	Value for each analyte	
	Ofloxacin	Moxifloxacin
Linear range, μg L^{-1}	20-4000	10-2000
Determination coefficient (R^2)	0.9998	0.9979
Limit of detection (3σ), μg L^{-1}	4	2
Limit of quantification (10σ), μg L^{-1}	20	10
Enrichment factor (n=3)	18±1	40±3
Extraction recovery (n=3), %	44±3	87±1
Relative standard deviation, % (n=3)	7-9	5-10

Sample	Moxifloxacin			Ofloxacin		
	Added, μg L^{-1}	Found, μg L^{-1}	Relative bias, %	Added, μg L^{-1}	Found, μg L^{-1}	Relative bias, %
Urine A	20	20.9±1.3	4	40	39±4	-2
	1500	1567±98	4	3000	2775±394	-7
Urine B	20	20.83±0.20	4	40	43±10	8
	1500	1790±337	19	3000	2833±275	-6

n = 3, P = 0.95

Conclusions:

- A new approach for the microextraction and preconcentration of ofloxacin and moxifloxacin from urine using supramolecular solvent based on di-(2-ethylhexyl)-phosphoric acid salt was developed;
- The conditions for the formation of the supramolecular solvent with phase separation were revealed;
- Optimal conditions for microextraction of analytes were established;
- The developed procedure was validated and showed good analytical performance for real urine samples analysis.

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