

Development of a methodology for quantification of pharmaceuticals and personal care products in hydric media

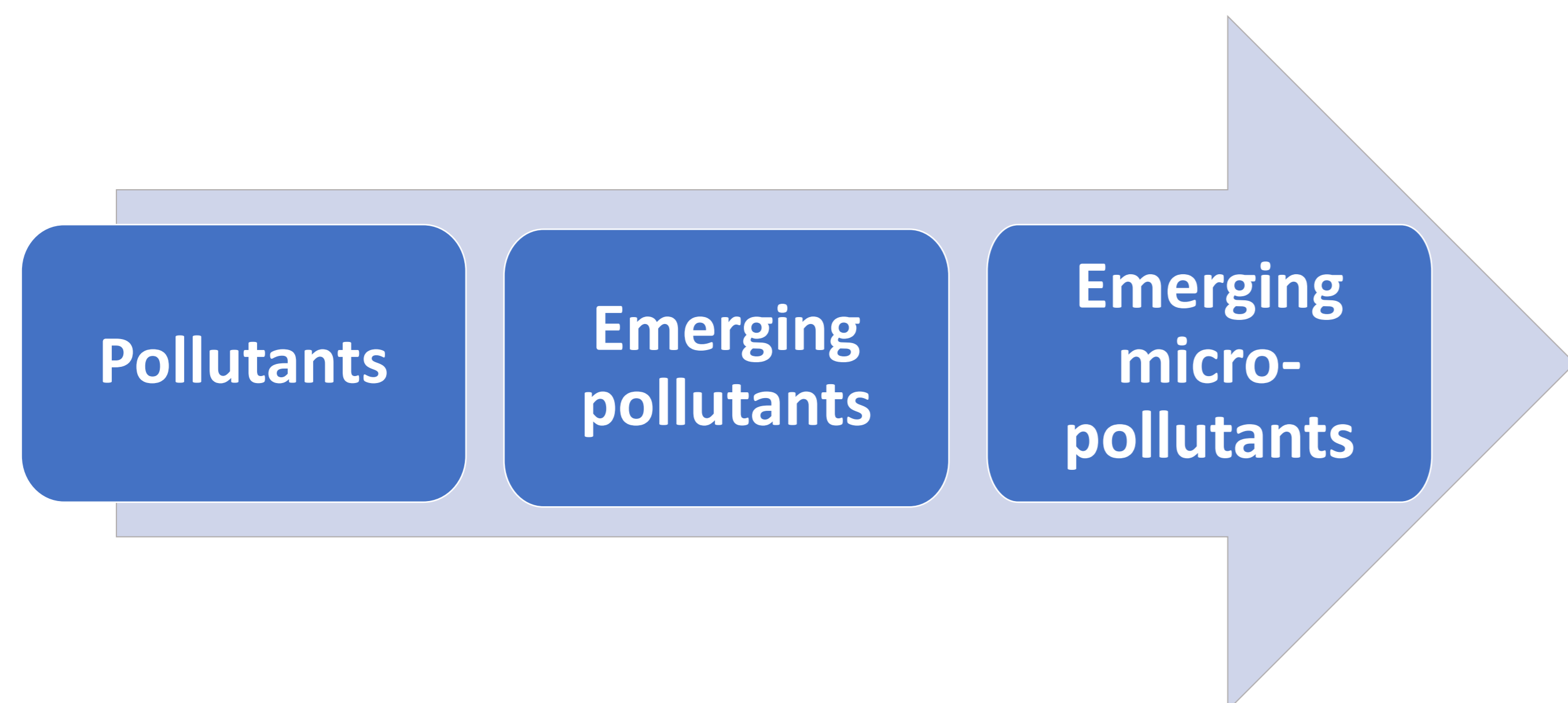
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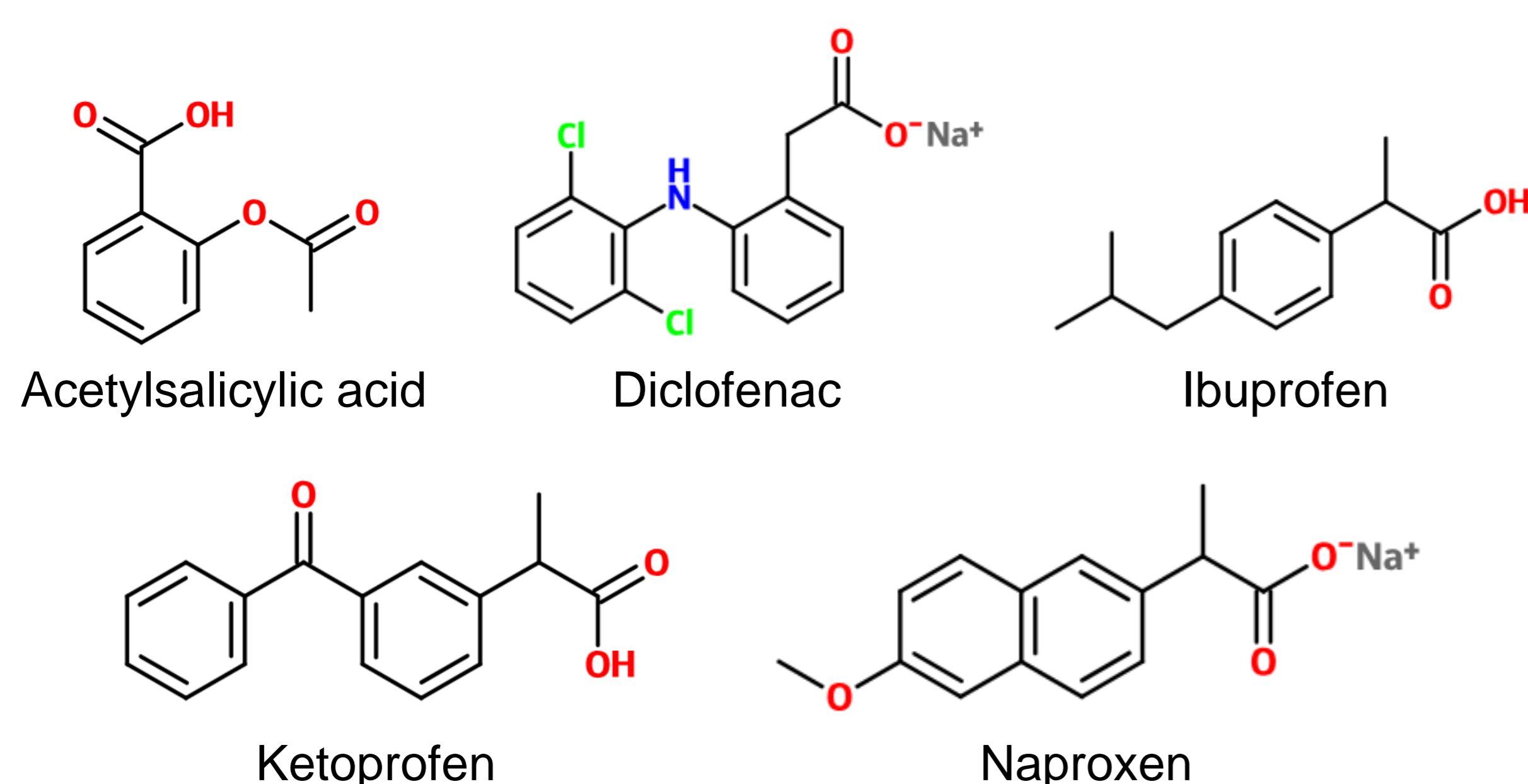
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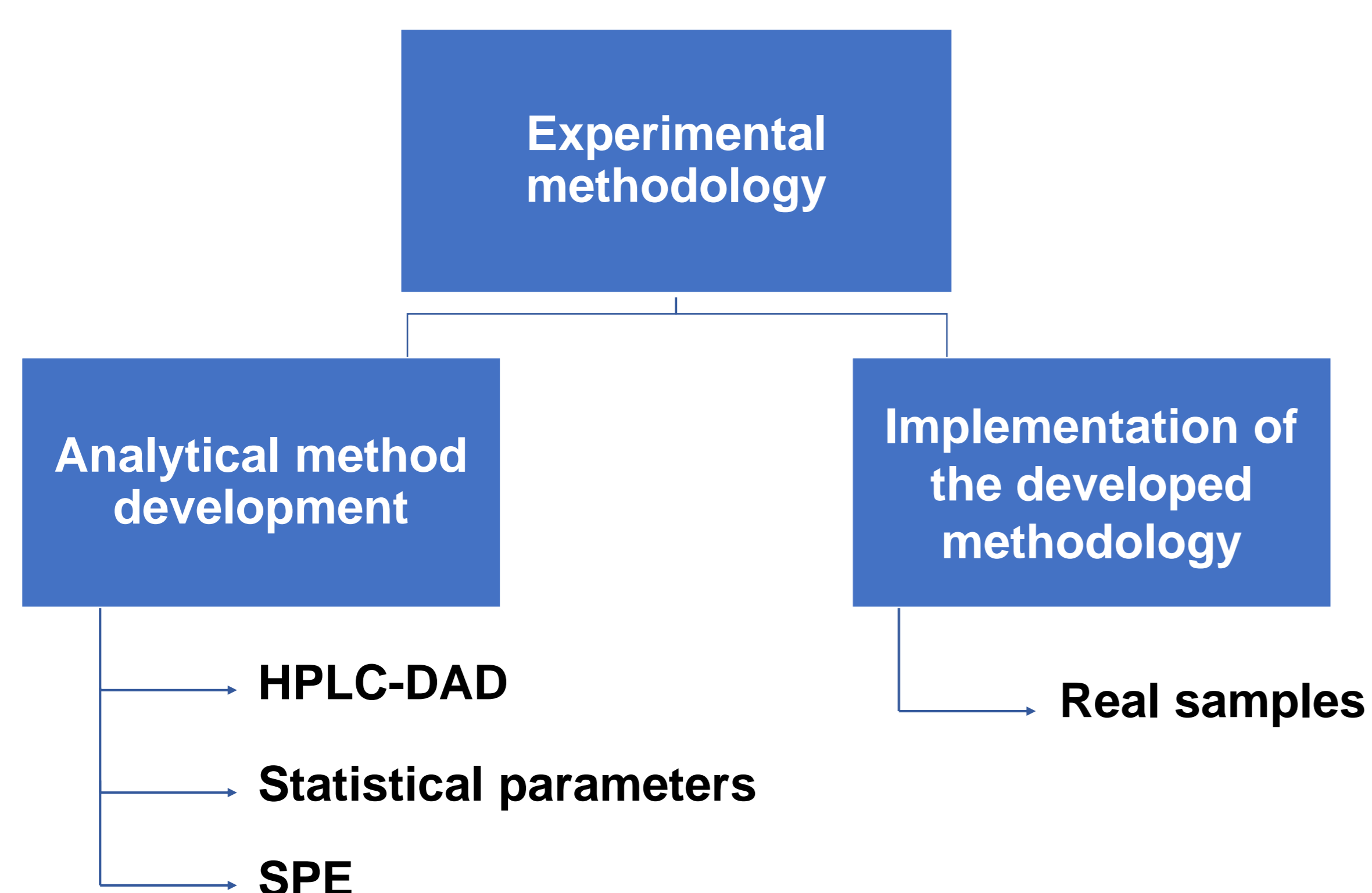
EMERGING MICROPOLLUTANTS



Emerging micropollutants are chemical substances present in different matrices at very low concentrations, ranging from nanograms to micrograms per liter. **Non-Steroidal Anti-Inflammatory Drugs (NSAIDs)** are some of the most prescribed drugs worldwide and several studies report the presence of these substances in various hydric media including drinking water, surface water, sewage water, among others¹. Since they are present in **very low concentrations**, their identification, quantification and removal are not easy tasks².



EXPERIMENTAL METHODOLOGY



The HPLC-DAD method uses a **C18 column**, a mobile phase composition of **60acetonitrile:40water:0.01 trifluoroacetic acid (V/V/V)** with a flow-rate of **1.2 mL/min**. The loop injection volume was **20 µL** and the monitored wavelengths were **219, 224, 254 and 275 nm**.

The optimized **Solid Phase Extraction (SPE)** procedure uses a cartridge Oasis HBL (N-vinylpyrrolidone-divinylbenzene copolymer). The flow-rates for each step ranged from **0.2 to 0.9 mL/min**.

Table 1 – SPE operating conditions.

Step	Solvent	Volume (mL)	Flow-rate (mL/min)
Conditioning A	Methanol	5	0.22
Conditioning B	Water	5	0.51
Loading	Aqueous sample	10	0.53
Washing	Water	5	0.87
Elution	Methanol	8	0.18

RESULTS

The optimized methodology led to **Limits of Detection (LOD)** and **Limits of Quantification (LOQ)** that ranged from 2.2 ppb to 74.0 ppb and 7.5 ppb to 246.7 ppb, respectively. The **Coefficients of Variation** for the **Repeatability** studies were between 0.25% and 4.76%. The **Intermediate Precision** ranged from 0.43% to 9.2%. The **recovery** values for the SPE procedure were above 91.4% (m/m) and 114.3% (C/C).

For the **Calibration Curves** 6 concentration levels were used for each compound, with exception of acetylsalicylic acid, that used 8 levels. Ketoprofen and naproxen presented the lowest linear ranges (5 to 50 ppb) and acetylsalicylic acid the highest one (40 to 5000 ppb). The **Coefficients of Determination (R²)** were above 0.98, indicating good linearity.

Six samples were analyzed, one from tap water (**S1**), one from groundwater (**S2**), three from surface water (2 locations in River A, **S3** and **S4**, and 1 location in River B, **S5**) and one from pool water (**S6**).

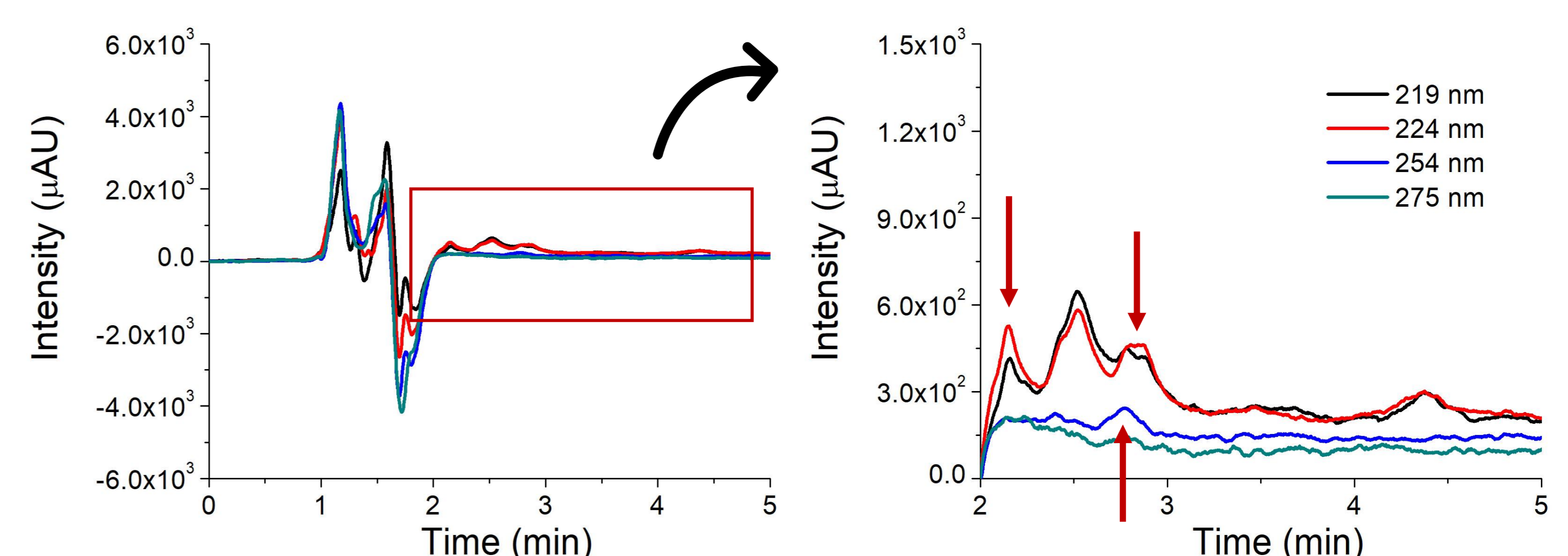


Figure 1 – Surface water from river A (location 1) results.

Table 2 – Contaminated samples results.

Sample	Compound	Concentration (ppb)
S2	Ketoprofen	<LOQ
S3	Acetylsalicylic acid	<LOD
	Ketoprofen	11.23 ± 0.77
S6	Naproxen	<LOQ
	Acetylsalicylic acid	<LOD

REFERENCES

- [1] Patel P. N.; Samanthula G.; Shrigod V.; Modh S. C.; Chaudhari J. R., "RP-HPLC Method for Determination of Several NSAIDs and Their Combination Drugs", *Chromatography Research International*, 2013, Volume 2013, 1-13.
 [2] Sousa J. C. G.; Ribeiro A. R.; Barbosa M. O.; Pereira M. F. R.; Silva A. M. T., "A review on environmental monitoring of water organic pollutants identified by EU guidelines", *Journal of Hazardous Materials*, 2018, Volume 344, 146-162.