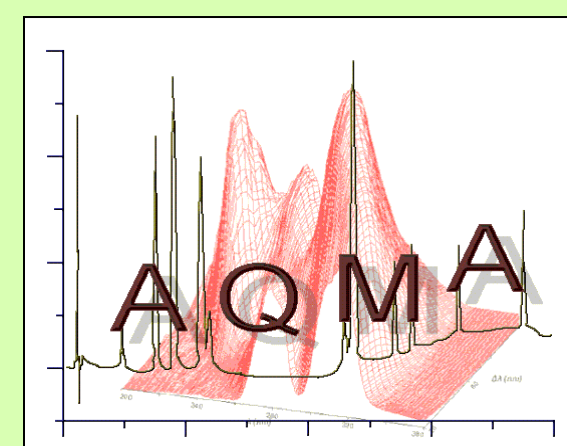


# DETERMINATION OF PHARMACEUTICAL RESIDUES IN WATERS FROM GRAN CANARIA ISLAND USING A SPE-HPLC-ESI/MS/MS METHOD

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## INTRODUCTION

Pharmaceutical compounds are widely used and they can reach the WWTPs, which are unable to remove them completely and they have a strongly contaminating effect on the natural aquatic systems (Martinez et al, 2009). We optimise a method to the determination of atenolol (B-blocker), metamizol (analgesic), fluoxetine (antidepressant), caffeine (stimulant), and its metabolite paraxanthine, nicotine and permethrin in different environmental water samples, including an extraction and preconcentration step using solid-phase extraction (SPE) prior to their determination by liquid chromatography-electrospray ionization tandem mass spectrometry (HPLC-ESI-MS/MS), which has been very efficient (Moldovan, 2006) because their environmental concentrations are very low. This optimised procedure was applied to different groundwater samples (wells, probes and gallery waters) taken in a hydrogeological basin of Gran Canaria island that may be affected for infiltration process from a golf course irrigated with depurated waters. A preliminary sampling was carried out around of the basin and then five significant samples were selected for a temporal monitoring of the concentrations of pharmaceutical compounds.

## METHOD

### Extraction



- ✓ Cartridge: Oasis HLB
- ✓ Flow rate: 10 mL/min
- ✓ Volume sample: 200 mL
- ✓ pH: 8
- ✓ Elution: 2 mL MeOH
- ✓ Reconstitution: 2 mL water

### Detection

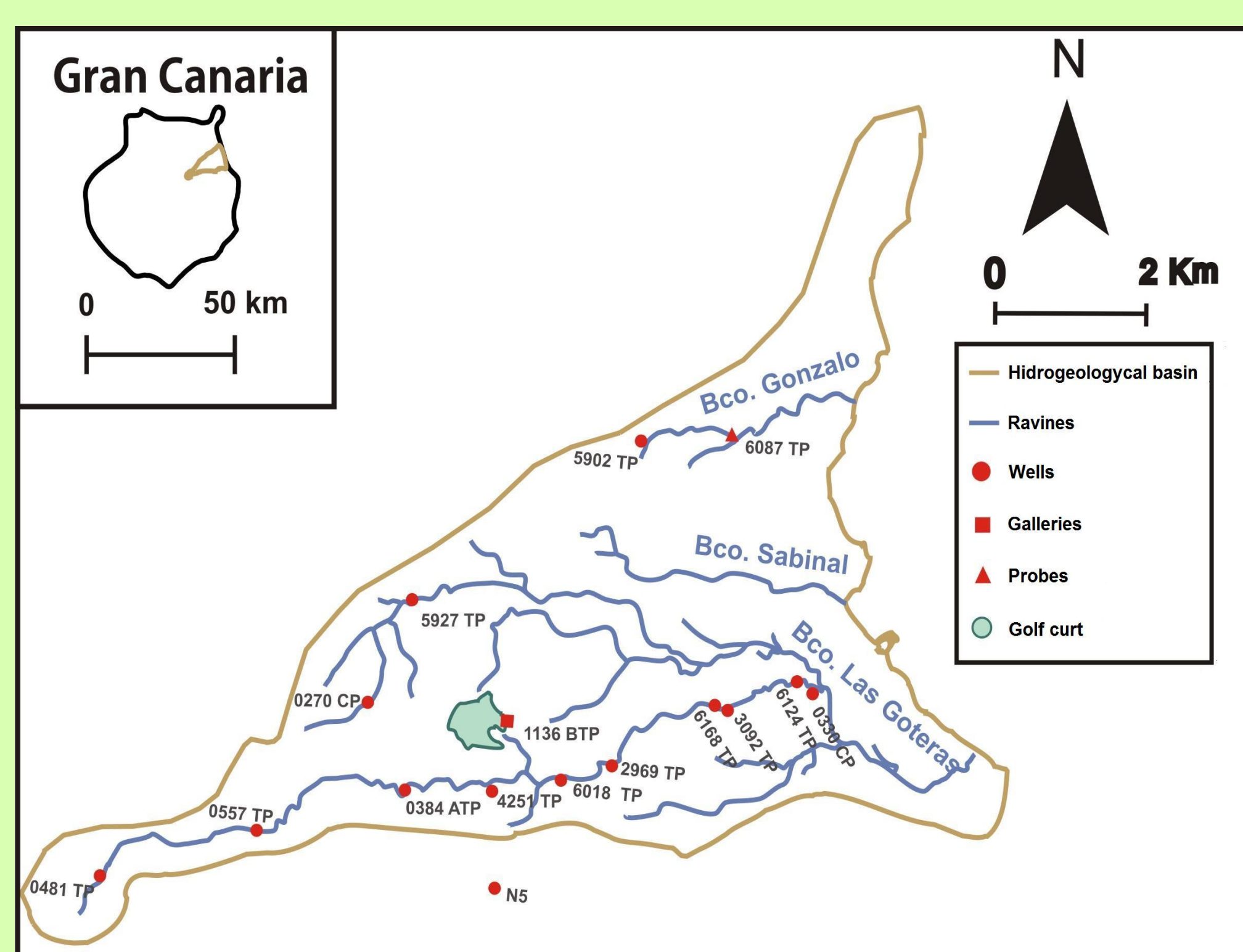
Compound	Retention time (min)	Precursor ion (m/z)	Cone (V)	Fragment ions
Nicotine	2.0	163.2	30	83.9, 79.8
Atenolol	5.0	267.3	56	145, 190
Metamizole	7.1	218.1	40	56.1, 97
Paraxanthine	9.2	181.2	52	123.9
Caffeine	11.3	195.1	30	138
Fluoxetine	17.4	310.3	30	44.1, 148
Permethrin	29.1	310.3	36	236.1

## RESULTS AND DISCUSSION

		LOD <sup>a</sup> (ng·L <sup>-1</sup> )	LOQ <sup>b</sup> (ng·L <sup>-1</sup> )	RSD <sup>c</sup> (%)
1	NICOTINE	39.39	131.30	6.8
2	ATENOLOL	12.39	41.29	8.45
3	METAMIZOLE	5.93	19.77	4.37
4	PARAXANTHINE	21.56	71.88	8.41
5	CAFFEINE	2.86	9.53	5.90
6	FLUOXETINE	28.80	96.01	12.72
7	PERMETHRIN	31.92	95.76	16.10

<sup>a</sup> signal to noise ratio of 3 times;  
<sup>b</sup> signal to noise ratio of 10 times;  
<sup>c</sup> n=6

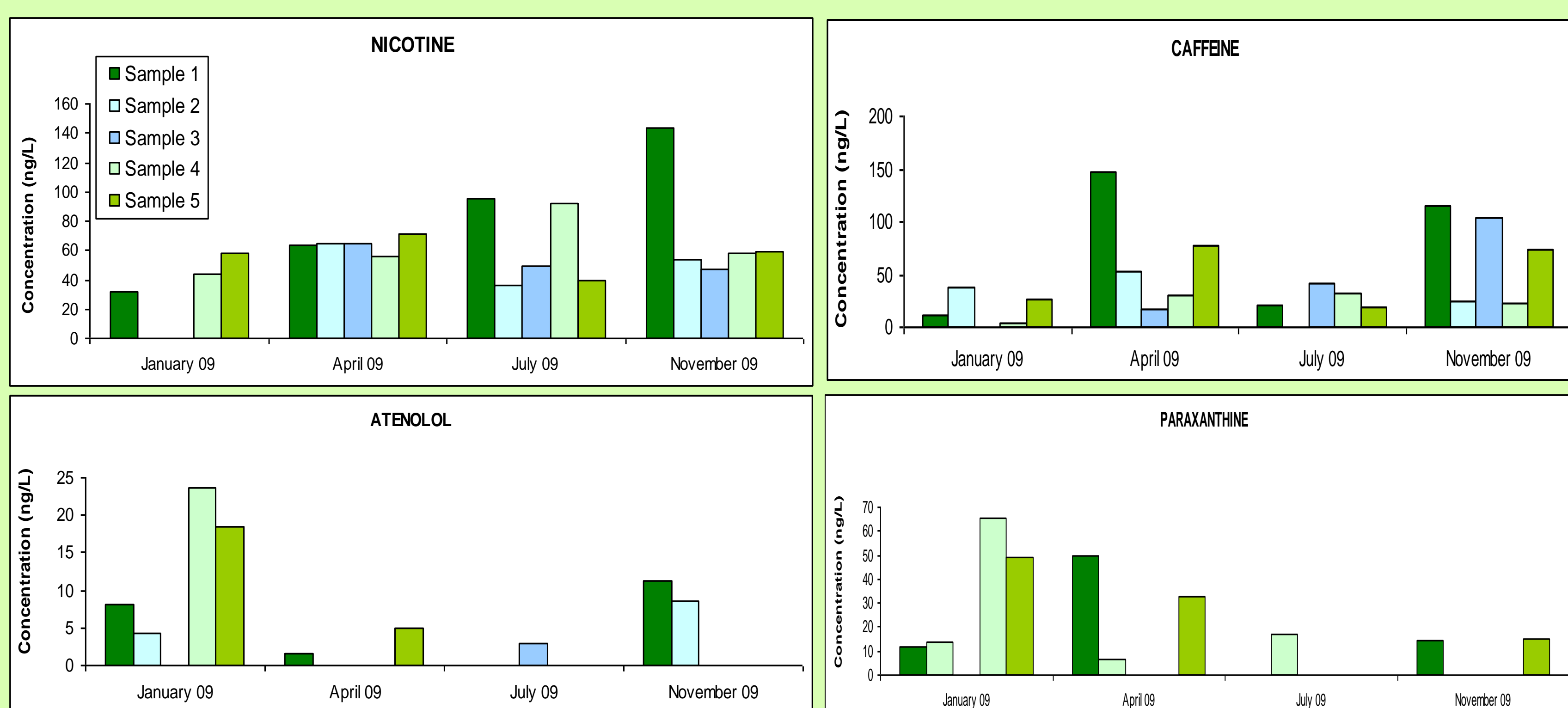
### Sampling



Location of analysed samples in a hydrogeological basin of the NE of Gran Canaria island (sixteen samples including waters from wells, galleries and probes Fig 1).

Five points were chosen for carry out a monitored every three months during one year.

### Temporal evolution



✓ All analytes were detected in some sample, except permethrin, which was not found in any location.

✓ Metamizol and fluoxetine were measured only in a timely manner.

✓ Nicotine and caffeine were detected in almost all samples and throughout the year.

## CONCLUSIONS

The optimized solid-phase extraction-liquid chromatography-electrospray ionization tandem mass spectrometry (SPE-HPLC-ESI-MS/MS) procedure allows to determine the concentrations of different emerging contaminants in environmental samples in a simple, reliable and rapid manner. Almost all the studied analytes were detected in different samples, and its concentration can be measured during all the year in some points, which demonstrates the need of improve the treatments of sewage waters when these are reused.

## REFERENCES

Martínez, M.J., Agüera, A., Gómez, M.J., Hernando, M.D, García-Reyes, J.F., Fernández Alba, A.R. (2007). Application of liquid chromatography/quadrupole-linear ion trap mass spectrometry and time-of-flight mass spectrometry to the determination of pharmaceuticals and related contaminants in wastewater. *Anal Chem.*, 79 (24), 9372-9384; Moldovan, Z. (2006). Occurrences of pharmaceutical and personal care products as micropollutants in rivers from Romania. *Chemosphere*, 64 (11), 1808-1817

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