Pharmaceuticals are found in most worlds' wastewater. The wastewater treatment plants (WWTP) are not designed to remove most of the micropollutants from the wastewaters.
Several decades of research have shown that this continuous leakage of micropollutants from our treatment plants can cause stress on sensitive aquatic ecosystems.
Even if the environmental risks are not fully established, there is a need to protect the environment from emissions of micropollutants. Monitoring of micropollutants such as pharmaceuticals, which are not easy to analyse, plays an important role.

Why are chemical analyses important?

- Effect of micropollutants on environment and human health is still unknown
- Increased monitoring of pharmaceuticals is urgently needed
- Pharmaceuticals in aquatic environment occur at low to very low concentrations
- Special and careful analytical procedures and advanced chemical knowledge are required

This leaflet presents certain sampling procedure and important aspects when performing chemical analyses of pharmaceuticals.

Contact

Training Material

Gdansk Water Foundation

Beata Szatkowska
beata.s@gfw.pl
www.gfw.pl

Gdańska Fundacja Wody

Lead Partner

Kristianstad University

Erland Björklund
erland.bjorklund@hkr.se
www.hkr.se

Kristianstad University Sweden

This leaflet was developed in the MORPHEUS project. The aim of MORPHEUS is to support actions in reducing the constant release of pharmaceutical substances via WWTPs to the South Baltic Sea.

Leaflet content: GWF; Layout: EUCC-D

@morpheus_eu

www.morpheus-project.eu

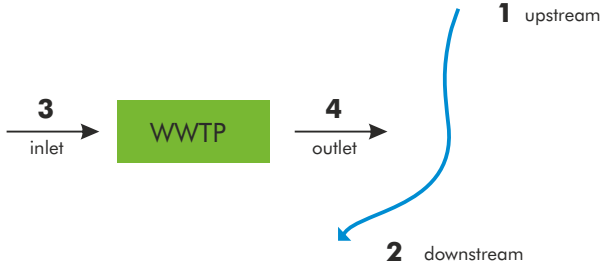
Best practices in chemical analysis of pharmaceuticals in the environment

www.morpheus-project.eu

The procedure related to the analysis of pharmaceuticals includes 3 important steps:

Step 1 - Sampling

Where: Take samples in the river: upstream (1), downstream (2),
in the WWTP: inlet (3), outlet (4)
When: Winter/summer (seasonal differences)
What: Select the substances that shall be monitored according to the EU watchlist



Important:

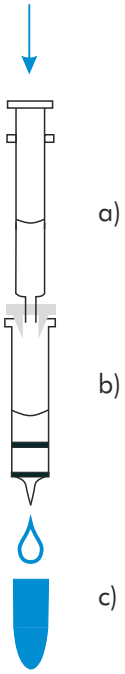
1. Use the national/regional sample material as recommended by authorities,
2. Wear protective gloves,
3. Rinse the container 3 times with the sample (water stream),
4. Hold the jar near the bottom and plunge it below the water surface,
5. Turn the submerged container into the current / water stream,
6. Allow the water to flow into the container,
7. Cover the full container while it is submerged. Remove it from the water.

Step 2 - Sample Preparation

Store the samples at 4 °C for a maximum of 48 h before analysis. If immediate analysis is not possible, samples should be stored at -20 °C.
Remember that the choice of storage might dictate the sample volume and sample bottle type (amber glass or HDPE).
The generals steps in a Solid Phase Extraction (SPE) procedure prior to final analysis is shown in the figure below. The SPE concentrates and cleans the samples.

General protocol for an SPE

1. Transfer sample to the sample holder (a)
2. Add internal standards mixture (a)
3. Load the sample to the SPE cartridge (b)
4. Dry the solid phase (b)
5. Elute analytes (pharmaceuticals) (c)
6. Reduce the volume of eluate
7. Perform analysis (LC MS/MS)



Step 3 - Final analysis

Samples must be sent urgently to the trace organic laboratory for further analysis, most commonly using the technique of SPE-LC-MS/MS or SPE-GC-MS/MS. Different laboratories use somewhat different methods, however the results should of course be the same.
The LC and GC separates the analytes before the final analysis and detection in the mass spectrometer (MS/MS).

Samples ready to be analysed in LC-MS/MS

Robust and sensitive analysis allows to:

- Monitor micropollutants in various environmental recipients
- Evaluate micropollutants effluent loads from WWTPs and their micropollutants removal efficiency
- Prioritize which WWTPs require the upgrade to advanced treatment technologies
- Remember:
  "You can only find what you are searching for"!