Study of Antibiotics and Pesticides in Water Using Automated, Online SPE-LC-MS/MS
Eawag, Dübendorf, Switzerland

Background
Eawag: Swiss Federal Institute of Aquatic Science and Technology is an aquatic research institute committed to the ecological, economical and socially-responsible management of water. The work of Eawag, located in Dübendorf, Switzerland, is based on the philosophy that clean waters are not self-evident. The lab’s goal is to help ensure the quality of the water sources in Switzerland.

One of Eawag’s studies concentrates on antibiotics and pesticides. Antibiotics enter the environment through manure used for agricultural purposes or input from wastewater treatment plants. Pesticides are intentionally introduced into the environment through crop protection in agricultural areas or household use in urban areas. If these compounds reach the aquatic environment, they can potentially cause unwanted side effects on humans and aquatic organisms.

Challenge
In order to determine the occurrence and fate of antibiotics and pesticides in the aquatic environment, Eawag conducts mass flow studies at selected field study sites. For accurate mass balances in rivers and streams, analytical methods must exhibit a high sample throughput and a dynamic measuring range over several orders of magnitude. In addition, high selectivity is required to avoid interference by matrix constituents.

To gain sensitivity in the low ng/L range, water samples require pre-concentration procedures before analysis, which is traditionally performed using offline solid-phase extraction (SPE). Offline SPE is time-consuming and costly. Because technicians need to prepare each sample manually, usually only a limited number of samples can be analyzed at one time. This is inefficient—especially with the large number of samples typically analyzed in Eawag’s studies.

The lab’s goal was to develop a method including online SPE coupled with liquid chromatography-mass spectrometry (LC-MS/MS) to overcome the disadvantages of the offline SPE procedure. Their hope was that this new method would allow for unattended 24/7 operation and the storage of the extraction method within the raw data files. If accomplished, this would reduce the laboratory’s administrative overhead.

Eawag chose the Thermo Scientific EQuan environmental quantitation system because it not only provides the sensitivity levels required for this type of analysis, but it also allows for online sample preparation. The system is the only solution available that utilizes Highly Selective Reaction Monitoring (H-SRM) performance, which improves signal-to-noise in complex matrices.

The Solution
Eawag successfully implemented a fully-automated, online pre-concentration LC-MS/MS EQuan™ method to analyze polar contaminants in natural waters. The sensitivity and robustness of the system proved to be essential factors in developing the online SPE method. The EQuan system exceeded the sensitivity levels required for this type of analysis, thereby supporting the technology’s applicability to environmental studies of this nature.

“The fully-automated, online pre-concentration LC-MS/MS approach using the EQuan system made it possible to achieve high sensitivity and high sample throughput in the low ng/L range with low financial investment,” said Heinz P. Singer, Sr. Scientist, Eawag Department of Environmental Chemistry.
**Business Benefit**

The direct coupling of SPE to LC eliminated several working steps, such as evaporation and reconstitution. This resulted in a 48% time savings and a more precise procedure since the total enriched amount of substance is eluted directly to the liquid chromatograph. In addition, automated, online SPE decreased the potential for human error by eliminating steps in the process. The method successfully reduced manual sample preparation to sample filtration and spiking of the internal standard solution, thereby decreasing the laboratory time by more than a factor of five.

The required high sensitivity and high sample throughput in the low ng/L (ppt) range was successfully achieved using the automated, online pre-concentration LC-MS/MS method with the EQuan system. As a result, Eawag was able to develop an efficient, online pre-concentration LC-MS/MS method that furthers the organization’s mission for water pollution control.

Online SPE-LC-MS/MS chromatograms of a 1 ng/L standard for selected pesticides