Because we have been able to inject and enrich samples followed straightaway by LC-MS/MS analysis [on the Thermo Scientific TSQ Quantum] we were able to significantly reduce analysis times, as well as improve LODs.”

Dr. Sébastien Sauvé
Environmental Analytical Chemistry Laboratory at the University of Montreal

Introduction
The Environmental Analytical Chemistry Laboratory at the University of Montreal comprises approximately 20 researchers focused on finding the best analytical methods for environmental studies. The laboratory is currently collaborating with the City of Montreal, the Drinking Water Research Chair at the École Polytechnique de Montréal, and Environment Canada in order to identify and quantify pharmaceutical residues in the city’s waste and drinking water. To conduct this research, the University has developed a new online sample preparation method using a customized Thermo Scientific EQuan system for solid phase extraction coupled to liquid chromatography and tandem mass spectrometry (SPE-LC-MS/MS) analysis.

The EQuan™ system significantly reduced sample analysis time and considerably improved low limits of detection (LODs) of adverse pharmaceutical residues in drinking and wastewater.

Background
When a person or animal takes medication, the pharmaceutical compounds in that drug last beyond ingestion. Recent concern has arisen about the effects of pharmaceuticals in the environment after excretion by humans or animals. Municipal plants are not engineered to identify or remove residual pharmaceutical products in wastewater. Pharmaceutical compounds can therefore pass untreated through sewage systems into the environment and be ingested in minute quantities by people drinking water.

The University of Montreal is focusing on the identification and quantification of the pharmaceutical products of environmental concern that are present in the St. Lawrence River, which is used as a source of drinking water. Dr. Sébastien Sauvé, Associate Professor with the University of Montreal, Michèle Prévost, co-holder of the Drinking Water Research Chair at the École Polytechnique de Montréal, and Christian Gagnon at Environment Canada are carrying out a study with the City of Montreal. The goal of the study is to identify which compounds the city should remove and which treatment option can accomplish the removal.

Achieving LODs of pesticides, antibiotics and veterinary residues in drinking water is of paramount importance in order to monitor the regulatory levels as stated by U.S., Canadian, Japanese and European environmental and water directives. Since many of these substances could pose a significant health threat, they need to be accurately detected. Traditionally, LC-MS/MS has been used for the identification and quantification of these residues. However, this methodology requires extensive offline sample preparation, which can be particularly time-consuming and expensive.

Implementation
In 2005, Dr. Sauvé purchased the customized EQuan system, which consists of a Thermo Scientific TSQ Quantum series mass spectrometer, two Thermo Scientific Surveyor HPLC pumps with a preconcentration column, an analytical column, a CTC autosampler and an EQuan kit consisting of columns and HPLC accessories. EQuan can use any of the TSQ Quantum series instruments.

The mass spectrometer was equipped with the AM (Accurate Mass) option, which Dr. Sauvé and his laboratory staff members use to accurately measure pharmaceuticals potentially present in the environment. For “known” drugs and their metabolites, accurate mass measurements enable confirmation of the elemental composition using both mass spectrometry (MS) and MS/MS spectra. With the aid of additional chemical information (such as the molecular formula of the parent drug and an understanding of the possible metabolic pathways), the plausible structure of an unknown molecule can be positively identified or the possibilities narrowed to a few close fits.
Benefits

The EQuan environmental water monitoring system is designed for reliable and accurate analysis of environmental and drinking water samples and is specifically developed to monitor trace levels of pesticides, hormones, antibiotics, pharmaceuticals and veterinary products in drinking water. The University has benefited from the EQuan system because of its ability to significantly reduce sample analysis time. Additionally, LODs are considerably improved (up to 100 times) compared to conventional injection techniques. Detection limits of 10ppt (parts per trillion) are easily achieved by simply injecting filtered and acidified water samples – without further sample preparation.

The compounds of interest for the research project are present at trace levels. The sample preparation method requires preconcentration, which typically involves a time-consuming, complicated manual method that is prone to errors.

The researchers at the University of Montreal needed to develop an online preconcentration method for sample preparation of water samples both prior to and after treatment by the city’s wastewater treatment facility.

Dr. Sauvé worked with Thermo Fisher Scientific to develop methods that use the EQuan system to optimize the preconcentration of samples online instead of offline. All the preconcentration is done through column switching by the system before it feeds directly into the mass spectrometer.

“We have to do the sample preconcentration because, in spite of the excellent sensitivity of the Thermo Scientific instrument, the quantities of the pharmaceuticals of interest are very low,” Dr. Sauvé explained. “The biggest difficulty with the preconcentration step is the labor – it takes a lot of time to do it manually. Additionally, all of the sample manipulations that occur during the manual preconcentration step increase the potential for errors and reduce the reliability of the results.”

Besides the increased reliability and improved LODs of the EQuan solution, one of the biggest benefits is the personnel time savings. “As we evaluate the human resources required to do the offline analysis relative to what we can save when we have an online preconcentration method, the personnel time savings are very significant,” Dr. Sauvé commented.

Dr. Sauvé has used the EQuan system for continued research about the pharmaceutical products of most environmental concern that are being released into various water resources around Montreal. One such study involved the extraction, detection and quantification of six of the most common anti-infectives in untreated and treated sewage.

“We have been extremely pleased with the Thermo Scientific EQuan system,” commented Dr. Sauvé. “Because we have been able to inject and enrich samples followed straightaway by LC-MS/MS analysis we were able to significantly reduce analysis times, as well as improve LODs. The added benefit of reduced personnel costs means the investment was well worthwhile.”

Conclusion

Dr. Sauvé has used the EQuan system for continued research about the pharmaceutical products of most environmental concern that are being released into various water resources around Montreal. One such study involved the extraction, detection and quantification of six of the most common anti-infectives in untreated and treated sewage.

“We have been extremely pleased with the Thermo Scientific EQuan system,” commented Dr. Sauvé. “Because we have been able to inject and enrich samples followed straightaway by LC-MS/MS analysis we were able to significantly reduce analysis times, as well as improve LODs. The added benefit of reduced personnel costs means the investment was well worthwhile.”