

ANALYZE PHARMACEUTICAL COMPOUNDS IN SURFACE WATER BY DIRECT INJECTION

The Agilent 6550 iFunnel Q-TOF LC/MS System delivers the highest levels of sensitivity for targeted and untargeted compounds

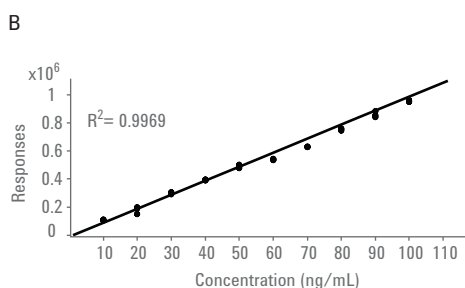
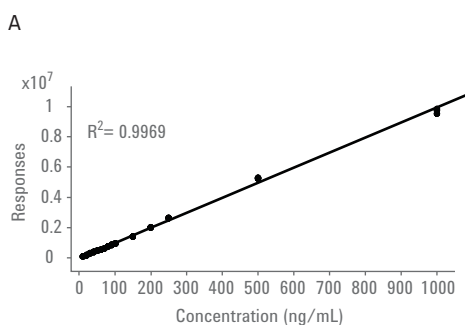


Figure 1. (A) Calibration curve of phenazone from 10 ng/L to 1000 ng/L with excellent linearity. (B) Zoomed view of lower concentration end of curve.

Background

The screening and analysis of surface water requires a sensitive and rapid method to detect emerging organic contaminants before drinking water production. LC/MS analysis has proven to be an ideal solution for routine water analysis, however present screening methods typically require a preliminary sample concentration step to achieve sufficient sensitivity which can result in the loss of highly polar compounds during sample preparation.

The Approach

The new Agilent 6550 iFunnel Q-TOF (6550 Q-TOF) mass spectrometer, when used in combination with an Agilent 1200 Infinity Series LC System equipped with a large volume injection kit, offers superior capabilities for the direct analysis of water samples.

The data presented here demonstrate that the combination of large volume, direct aqueous injection and high performance accurate mass spectrometry eliminates the need for sample preparation processes such as liquid/liquid or solid phase extraction. As a result, the discrimination of impurities is improved and limitations on the ability to identify unknowns during screening are removed. A method was developed to quantify (Figure 1) and unambiguously identify targeted organic contaminants in river water (Figure 2). A collection of known compounds detected by this method, and their LOD and LOQ, is shown in Table 1.



Identification of Unknown Organic Pollutants

A key strength of the method described here is its ability to simultaneously identify unexpected organic pollutants. As demonstrated in Figure 3, an unexpected contaminant, metoprolol, was identified by accurate mass measurement and isotope pattern matching. The identity of metoprolol is further confirmed by matching its MS/MS spectrum to the Broecker, Herre & Pragst Forensics/Toxicology Personal Compound Database and Library (Figure 4).

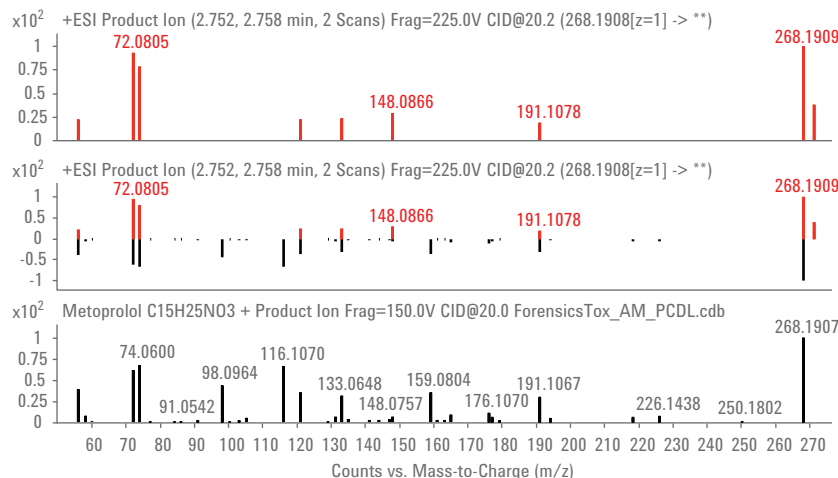


Figure 4. Identification of metoprolol can be further confirmed by MS/MS and retention time matching to a reference compound (top: sample spectrum, middle: mirror spectrum, bottom: library spectrum).

Broad Applicability

The 6550 Q-TOF with Jet Stream and iFunnel technology delivers the utmost sensitivity for a broad range of target compounds (Table 1), enabling ultra-trace compound identification and quantitation.

Table 1. LOD and LOQ values (ng/L) for a broad range of target compounds.

Compound Name	LOD	LOQ	Compound Name	LOD	LOQ
Atenolol	0.84	3.59	Sulfamethoxazole	2.97	10.84
Sotalol	0.44	2.01	Carbamazepine 10,11 epoxide	0.82	3.52
Metronidazole	0.73	3.19	Phenacetin	0.81	3.50
Ronidazole	2.65	9.73	Bisoprolol	1.38	5.52
Sulfadiazine	2.69	9.87	Propranolol	1.13	4.67
Trimethoprim	3.02	11.02	Carbamazepine	0.53	2.39
Sulfamerazine	2.55	9.39	Betaxolol	1.06	4.40
Sulfadimidine	2.59	9.53	n,n Diethyl-3-methylbenzamide (DEET)	0.95	4.02
Phenazone	0.47	2.14	Bezafibrate	1.12	4.62
Pentoxifylline	1.07	4.47	Crotamiton	0.49	2.22
Metoprolol	0.56	2.51	Diazepam	0.43	1.97
Furazolidone	2.27	8.47	Fenofibrate	3.35	12.31
Dapson	3.85	14.70			

*Calculated based on the DIN 32645 using a program courtesy of Dr. Joachim Kleiner. (www.kleiner-j.de)

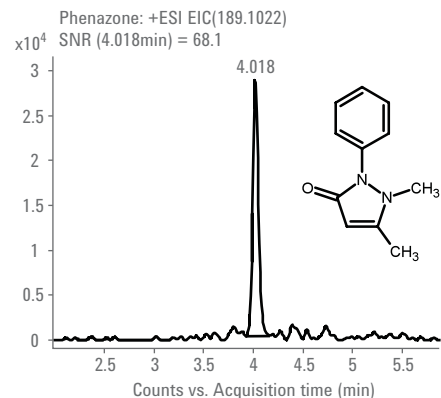


Figure 2. Extracted ion chromatogram of phenazone at the 10 ng/L level, with the structure of the compound (inset).

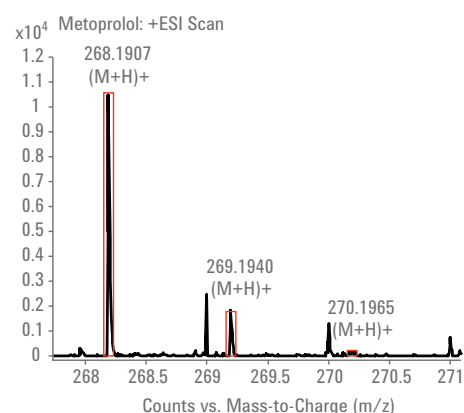


Figure 3. EIC of an unexpected contaminant, metoprolol, with a signal-to-noise ratio of 46:1. Red squares represent the theoretical isotope intensity and position.

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