METHOD STATEMENT

Determinand:
Benzotriazole and Tolytriazole.

Matrix:
Process Water (Deionised Water), Drinking Water, Surface Water, Treated Sewage and Untreated sewage effluent.

Principle of Method:
The compounds of interest are extracted from an aqueous matrix via Agilent's online SPE system, equipped with a Hypersil Gold aQ pre-concentration column. The compounds are then flushed from the pre-concentration column via a gradient run and are separated on an Agilent Eclipse Plus C18 RRHD 3.0x50mm, 1.8um analytical column, before being ionised by Agilent's Jetstream Electrospray technology, and analysed using MRM transitions on an Agilent 6470 LCMSMS.

Sampling and Sample Preparation:
Samples should be taken in a 250ml amber glass bottle containing 1% sodium thiosulphate.

Samples are stored at 3 ± 2°C prior to analysis.

Samples are stable in the following matrices for the times stated, (In-House Data) from sampling:

Compound	Matrix	Stability (days)
Benzotriazole (BZT)	Process Water (Deionised Water), Drinking Water, Surface Water	20
Tolytriazole (TZT)	Process Water (Deionised Water), Drinking Water, Surface Water	20

Samples are stable in the following matrices for the times stated, (Appendix 9a Supplement - Results of CIP2 Stability Test Summary Table 2) from sampling:

Compound	Matrix	Stability (days)
Benzotriazole (BZT)	Treated Sewage Effluent, Untreated Sewage Effluent	15
Tolytriazole (TZT)	Treated Sewage Effluent, Untreated Sewage Effluent	15

Interferences
Interfering compounds would have to display the identical MRM transition at the same retention time. However, any compound, which passes through the extraction procedure, and has a similar liquid chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference.

Performance of Method:

LOD, Precision and Bias

Determinand	LOD µg/l	LOQ µg/l	MRL µg/l	Low Std		High Std	
				%RSD	%Bias	%RSD	%Bias
Benzotriazole	0.0072	0.0141	0.05	3.43%	-0.27%	2.41%	-0.38%
Tolytriazole	0.0105	0.0206	0.05	3.41%	-0.08%	2.70%	-0.52%

Matrix Spike Recoveries

Determinand	Clean Process Water		Drinking Water		Surface Water		Treated Sewage		Untreated sewage	
	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec
Benzotriazole	2.43	98.66	2.12	99.32	3.09	100.65	2.48	100.37	3.28	100.80
Tolytriazole	2.87	98.51	2.43	99.25	3.11	100.11	3.08	98.84	3.84	99.12

Uncertainty of Measurement:

Determinand	Uncertainty of Measurement %
Benzotriazole	6.702
Tolytriazole	7.031

References:

In-house developed method.

DRAFT CIP2 Technical Specification and Guidance Version 30 07/05/2015 Appendix F- Appendix 9a Supplement - Results of CIP2 Stability Test Summary (February 2015).