METHOD STATEMENT

Determinand:
HBCDD

Matrix:
Surface, Ground and Saline waters

Principle of Method:
Approximately 400mls of sample is treated with sodium tetraethylborate (STEB) to ethylate TBT cation and is then extracted with 20mls of iso-hexane. The extract is analysed with a portion of the hexane extract exchanged into 70% methanol by LC-MSMS.

Sampling and Sample Preparation:
Samples should be taken in a 500ml amber or green glass bottle with 25mls of 6.25% 2-Chloroacetamide in methanol as a preservative.

Samples are stored between 5 ± 3°C and analysed with 7 days of sampling. (In-house stability data)

Interferences:
MS/MS is an extremely selective technique and interferences should only be encountered very rarely, however in theory, any compound which is extracted by the procedure, which has a chromatographic retention time similar to the compound of interest and which produces both precursor and product ions similar to that of the compounds in question, may interfere

Performance of Method:

Precision, Bias and Limit of Detection

		Low Standard		High Standard	
Compound	**LOD (ng/L)**	**Bias**	**RSD**	**Bias**	**RSD**
alpha-HBCDD	0.0416	-4.30%	11.43%	4.70%	8.09%
beta-HBCDD	0.0465	-4.47%	6.25%	2.69%	3.08%
gamma-HBCDD	0.0279	-4.73%	4.44%	3.20%	1.94%
HBCDD - Total	0.1378	-3.86%	6.30%	4.14%	3.22%

Matrix Spike Recoveries

	Surface Water		Ground Water		Saline Water	
Compound	**Recovery**	**RSD**	**Recovery**	**RSD**	**Recovery**	**RSD**
alpha-HBCDD	109.85%	9.15%	109.30%	10.01%	112.48%	13.58%
beta-HBCDD	103.27%	6.08%	101.40%	4.61%	102.01%	7.18%
gamma-HBCDD	103.33%	2.31%	102.50%	3.25%	102.95%	2.54%
HBCDD - Total	105.67%	3.69%	104.59%	4.11%	106.13%	3.09%

METHOD STATEMENT

Uncertainty of Measurement

The Uncertainty of Measurement has been calculated following the procedure given in GOP7.6D.

Compound	Relative UoM (%)	Minimum UoM (ug/L)
alpha-HBCDD	23.9	0.000027
beta-HBCDD	27.6	0.000027
gamma-HBCDD	14.4	0.000020
HBCDD - Total	13.4	0.00008

References:
EU Priority Substances Directive 2013
Directive 2013/39/EC