METHOD STATEMENT

Determinand:
Determination of Haloacetic Acids and Dalapon

Matrix:
Sample Type: Potable and Raw Water.

Principle of Method:
A 30-mL volume of aqueous sample is adjusted to a pH of 0.5 or less and extracted with 3 mL of methyl tert-butyl ether (MTBE). The acids that have been partitioned into the organic phase are then converted to their methyl esters by the addition of acidic methanol followed by heating for 2 hours. The solvent phase containing the methylated acids is separated from the acidic methanol by adding 5 mL of a concentrated aqueous solution of sodium sulfate. The aqueous phase is discarded. The extract is then neutralized with further concentrated aqueous solution of sodium sulfate and the solvent layer is removed for analysis. The target analytes are identified and quantified by capillary column gas chromatography using a mass spectrometer detector (GC/MS). Analytes are quantified using procedural standard calibration.

Interferences:
GC-MS is a selective technique and interferences should only be encountered very rarely. However, any compound, which passes through the extraction procedure, and has a similar gas chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference. The methyl ester of 2,3-dibromopropanoic acid, (surrogate used in US EPA method 552.2), and the methyl ester of dichlorobromoacetic acid are not completely resolved under the chromatographic conditions.

Performance of Method:
Range of Application:

Determinands	Operational Calibration Range
MonoChloroAcetic Acid (MCAA)	LOD - 125 µg/l
MonoBromoAcetic Acid (MBAA)	LOD - 125 µg/l
DiChloroAcetic Acid (DCAA)	LOD - 125 µg/l
2,2-DiChloroPropanoic Acid (Dalapon)	LOD - 125 µg/l
TriChloroAcetic Acid (TCAA)	LOD - 125 µg/l
BromoChloroAcetic Acid (BCAA)	LOD - 125 µg/l
DiBromoAcetic Acid (DBAA)	LOD - 125 µg/l
BromoDiChloroAcetic Acid (BDCAA)	LOD - 125 µg/l
DiBromoChloroAcetic Acid (DBCAA)	LOD - 125 µg/l
TriBromoAcetic Acid (TBAA)	LOD - 125 µg/l

Limit of Detection, Bias, Recoveries of Compounds and Uncertainty of measurement:

Determinand	LOD µg/litre	Direct Standards				Banwell Treated Water (Medium Hardness)		
		Low Standard		High Standard		80% Spike		
		Bias	RSD	Bias	RSD	Recovery	RSD	Uncert
MCAA	0.2	-2.4%	1.7%	0.4%	1.9%	100.6%	2.0%	± 4.53%
MBAA	0.5	-2.8%	2.2%	0.5%	1.9%	100.9%	1.5%	± 3.96%
DCAA	0.2	-2.8%	2.4%	0.6%	1.7%	100.6%	3.2%	± 6.95%
Dalapon	0.2	-2.6%	3.3%	0.9%	2.0%	100.2%	2.1%	± 4.54%
TCAA	0.2	-2.3%	2.9%	1.1%	1.6%	99.8%	1.9%	± 3.99%
BCAA	0.1	-2.4%	1.4%	1.2%	0.9%	100.3%	1.9%	± 4.13%
DBAA	0.1	-5.2%	4.0%	1.4%	1.1%	102.3%	2.5%	± 7.22%

Determinand	LOD µg/litre	Direct Standards				Banwell Treated Water (Medium Hardness)		
		Low Standard		High Standard		80% Spike		
		Bias	RSD	Bias	RSD	Recovery	RSD	Uncert
BDCAA	0.7	-5.6%	6.8%	4.4%	4.4%	105.1%	4.7%	± 14.55%
DBCAA	1.2	-2.5%	9.1%	3.4%	5.7%	103.5%	5.5%	± 14.44%
TBAA	1.1	0.3%	10.6%	3.3%	7.0%	105.1%	6.1%	± 17.36%

References:

US EPA Method 552.3, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Microextraction, Derivatization, and Gas Chromatography with Electron Capture Detection, EPA 815-B-03-002, revision 1, July 2003.