

METHOD STATEMENT

Determinand:

Unknown organic compounds extractable in Dichloromethane, which are suitable for analysis by GCMS. Compounds that cannot be analysed by GCMS and extracted with Dichloromethane have the following properties:

1) *Volatility*

- i) Compounds that are too volatile and vaporise in the concentration stage.
- ii) Compounds that are too volatile to be separated from the extracting solvent Dichloromethane in the gas chromatography stage.
- iii) In-volatile compounds unable to pass through a GC capillary column within a reasonable time scale.

2) *Thermal stability*

- i) Thermally unstable compounds that breakdown under high temperature in the injector.
- ii) Compounds that polymerise under high temperature in the injector.
- iii) Compounds of high molecular weight that are too involatile or thermally unstable.

Matrix:

Sample Type: Raw and Potable waters

Principle of Method:

Approximately 1l of sample is extracted with 100±10ml of Dichloromethane. The amount of sample required for extraction is dependent on the physical properties of the sample.

The solvent layer is extracted and collected into a turbovap tube.

The tube is then placed in the turbovap evaporator and concentrated to 1.0ml.

The sample extract is transferred to a 2.0ml autosampler vial and 25ul of Fullscan internal standard is added. The vial is capped ready for analysis.

Interferences:

The instrument is operated in Fullscan mode, and detects and identifies compounds by retention time and presence of ions resulting in a mass spectra characteristic to one compound.

The peaks in the final chromatogram are identified by the library searching of mass spectra obtained against a standard NIST library containing 98,000 spectra and quantified against a standard of target compounds at a known concentration.

Therefore, each peak obtained can be identified providing the mass spectrum of that peak is in the NIST library or a target compound in the standard.

It must be noted that when two or more compounds co-elute, their mass spectra will be distorted by one another. In this instance identification of each compound is difficult (when using the Chemstation software alone to identify the compounds present).

With the introduction of Deconvolution software – the software is now better able to resolve co-eluting peaks by using slight differences in the retention times of the ions being contributed by each compound to split the compounds and therefore identify them.

Performance of Method:

Range of Application:

For the targeted compounds the detection limits of each group of compounds are as follows :-

Chlorinated organic and disinfectant by-products	LT0.1ug/l
PAH compounds	LT0.3ug/l
Phthalate and Adipate Esters	LT0.1ug/l
Purgeable Aromatic compounds	LT0.1ug/l
Phenolic compounds	LT0.24ug/l



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Limit of Detection

0.5µg/l when 1l of sample is extracted for tentatively identified compounds.

References:

In-house method

Hewlett and Packard MSD Hardware manual. Hewlett and Packard MS chemstation user's guide.

