



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 Types: Raw and Potable waters.

Principle of Method:

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

The solvent layer is extracted and water is removed with muffled anhydrous sodium sulphate and then transferred 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 25 μ l of Fullscan internal standard is added. The vial is capped ready for analysis.

Sampling and Sample Preparation:

Samples are brought in 1 litre Green glass bottles, designated as "GCMS", and are stored at $3 \pm 2^\circ\text{C}$ on receipt at the laboratory. The bottles should only be filled to the shoulder - never to the top during the sampling stage as this could compromise any oil layer present at the point of sampling (failure to do this could result in a non-representative sample being taken).

Interferences

The instrument is operated in Sim and Scan 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.

Masshunter Deconvolution is 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.

METHOD STATEMENT



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	LT0.1µg/l
PAH compounds	LT0.3µg/l
Phthalate and Adipate Esters	LT0.1µg/l
Purgeable Aromatic compounds	LT0.1µg/l
Phenolic compounds	LT0.24µg/l

Limit of Quantification:

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

Recoveries of Compounds, Bias and Uncertainty of measurement:

N/A

References:

In-house method