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: Waste waters

Principle of Method:

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

The solvent layer is removed from the bottle (and then centrifuged if necessary due to the formation of a heavy emulsion). This aliquot of Dichloromethane is dried with muffled anhydrous sodium sulphate. At this point a series of serial dilutions of the extract (each a factor of ten more dilute than the last) is carried out (with more of the spiked Dichloromethane) so that the diluted extracts can be analysed first in order to protect the instrument from potential high levels of contamination.

The final extracts are transferred to a 2.0ml autosampler vial and spiked with the current WPC35 internal standard solution, the vials are then capped ready for analysis.

Sampling and Sample Preparation:

All samples are stored in the cold room between 3 ± 2°C on receipt at the laboratory.

The samples are extracted within two weeks of sampling and the extracts analysed no later than two weeks following extraction.

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

The library searching of mass spectra obtained against a standard NIST library containing 98,000 spectra identifies the peaks in the final chromatogram

Therefore, each peak obtained can be identified providing the mass spectrum of that peak is in the NIST library.

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.

Performance of Method:

Range of Application:

Concentrations quoted are derived mathematically by a comparison of responses between the organic compounds detected in the sample and the concentration of the internal standard concentration and must be considered as tentative only.

Limit of Detection:

The limit of detection is dependant on the sample volume extracted for all tentatively identified compounds.

Recoveries of Compounds, Bias and Uncertainty of measurement:

Performance characteristics are not appropriate for this qualitative method.

References:

In-house method