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METHOD STATEMENT SVOC TARGETS

INTRODUCTION

The performance of this method is validated in accordance with internationally recognised procedures.

This method is also suitable for the determination of bromoxynil octanoate, Nonylphenol, Chloronitrotoluenes and poly-chlorinated terphenyls (semi-quantitatively).

Note: The determination of bromoxynil octanoate, nonylphenol, chloronitrotoluenes & poly-chlorinated terphenyls is not UKAS accredited.

This procedure describes the determination of semi-volatile organic compounds in soils and waters by solvent extraction / concentration followed by analysis of extracts by gas chromatography mass spectrometry.

PRINCIPLE

An aliquot of the sample is spiked with isotopically labelled internal standards and then the semi-volatile organic compounds are extracted into a solvent. These extracts are then concentrated by evaporation and analysed by scanning Gas Chromatography Mass Spectrometry (GC / MS). Selected Ion Monitoring methods may also be used where lower LODs are required, usually by measurement of the three most abundant ions for a target analyte.

Deuterated internal standards are added to all test samples, spikes and blanks before analysis. The quantitative results are then corrected for the recovery of these standards.

In instances where a compound is not present in the calibration standards, a tentative identification and result are produced based upon a comparison of the mass spectra obtained with those found in the NIST mass spectral databases. Such tentative results are subject to confirmation by the analyst.

SOIL SAMPLES

0.1 to 10 g aliquots of as received samples are taken, spiked with labelled internal standards, then microwave extracted with an aliquot of dichloromethane. The extracts reduced in volume to circa 1 ml for GC/MS analysis.

WATER SAMPLES

10 ml to 1 L aliquots of the shaken sample are taken, spiked with isotopically labelled internal standards, then extracted by liquid / liquid techniques with dichloromethane. Usually, the sample is extracted with at least two aliquots of solvent as follows. The sample is made alkaline to a pH greater than 11 and a further extract is taken. The sample is then acidified to a pH of less than 2 and a further extract taken. The extracts are combined, then reduced in volume by evaporation to 200 ul – 1 ml for GC / MS analysis.



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Where specific compounds are to be targeted, a combination of neutral / base / acid extractions may be used to ensure acceptable recovery of the analytes.

PERFORMANCE CHARACTERISTICS

SUBSTANCES DETERMINED

A range of semi-volatile organic compounds, ranging in boiling points from circa 180 °C to 600 °C. Standard target suite includes polyaromatic hydrocarbons (PAHs), OCPs, OPPs, phthalates, phenols, triazines and other priority pollutants.

RANGE OF APPLICATION

- Soils 0.01 to 100 mg / kg (compound dependent)
- Waters 0.01 ug / ml to 10 mg / L (compound dependent)
- Nonylphenol 1 ppm to 100 ppm

LIMIT OF DETECTION

- Soils 0.01 mg / kg
- Waters 0.01 ug / L
- Bromoxynil Octanoate 0.2 ug / L
- Nonylphenol 0.5 ug / ml
- Poly-chlorinated Terphenyls 0.1 mg / kg
- Chloronitrotoluenes 0.1 ug / L

ANALYTICAL QUALITY CONTROL

Analytical quality control is maintained by a number of measures:

- Multi-point calibration with authentic standards (with defined minimum performance characteristics).
- Analysis of control samples within each analytical batch, such as independent standards, matrix spikes or reference materials.
- Analysis of reagent / method blanks within each analytical batch.
- Ongoing quality assured by the use of control charts in conjunction with warning and action limits for the QC sample data.
- Participation in external proficiency testing and inter-laboratory schemes such as LGC Standards CONTEST and AQUACHECK where available.

REFERENCES

- US EPA Method 8270, Revision C, Semi volatile Organic Compounds by Gas Chromatography-Mass Spectrometry (GC/MS).
- US EPA Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.