



CONCEPT LIFE SCIENCES

METHOD STATEMENT VOC TARGETS BY HEADSPACE GC / MS

INTRODUCTION

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

This procedure describes the determination of volatile organic compounds (VOC) in soils and waters by headspace gas chromatography mass spectrometry (GC / MS).

This method is also suitable for the determination of mercaptans, heptane and methyl acetate.

Note: The determination of mercaptans, heptane and methyl acetate are not UKAS accredited

PRINCIPLE

The sample is heated and shaken vigorously. This carries the volatile components into the headspace of the extraction vessels. A volume of the headspace is sampled and passed onto the head of gas chromatography (GC) column which separates the different components. The eluent from the GC column passes into the ion source of a mass spectrometer which, records mass spectra continually at a regular interval and with unit mass resolution.

A series of aqueous standards are analysed by this method and the data are used for reference and calibration. Deuterated internal standards are added to all samples, spikes and blanks prior to analysis.

In instances where a compound is not present in the calibration material, a tentative identification and result is 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.

Samples are stored in a refrigerator (5 +/- 4 °C) prior to and following analysis.

SOIL SAMPLES

0.1 to 10 g aliquots are spiked with the internal standards, immersed in 10 ml of deionised water and then analysed.

WATER SAMPLES

A 1 to 10 ml aliquot is removed directly from the sample vessel, spiked with the internal standards, then analysed.

PERFORMANCE CHARACTERISTICS

SUBSTANCES DETERMINED

A range of volatile hydrophobic organic compounds, ranging in boiling points from circa -10 °C to 200 °C. Standard target suites include chlorinated solvents, 'BTEX' and other priority pollutants.



CONCEPT LIFE SCIENCES

RANGE OF APPLICATION

- 1 to 2000 ug / L, ug / kg (compound dependent)
- Mercaptans 25 to 300 mg / L

LIMIT OF DETECTION

SOILS

- Typically 5 ug / kg for VOC 624 suite
- Typically 1 ug / kg for BTEX
- Methyl Acetate 100 ug / kg
- Heptane 20 ug / kg

WATERS

- Typically 1 ug / L
- Mercaptans 0.1 – 5 mg / L (compound dependent)
- Methyl Acetate 100 ug / L
- Heptane 20 ug / L

VALIDATION

Initial method validation was conducted via the analysis of 6 x 2 replicate spike samples prepared and analysed independently, along with a three point calibration, method blanks and independent QC samples. The precision, bias and uncertainty for each analyte was determined from the data acquired and found to be as follows for all analytes:

- Precision: < 5 %
- Bias: < 8 %
- Uncertainty: < +/- 5 %

Ongoing method performance is monitored via analytical quality control charts.

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.



CONCEPT LIFE SCIENCES

REFERENCES

- US EPA Method 8260, Revision B, Volatile Organic Compounds by Gas Chromatography-Mass Spectrometry (GC/MS).