Application Note No. 017

Automating the Analysis of Selected Phenols Using The Focus Sample Processing Robot

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Introduction

Phenols are currently prepared in using a traditional solvent extraction technique. The method involves the simultaneous extraction and derivatisation of selected phenols using pentafluorobenzoyl chloride as the derivatising agent from a buffered aqueous sample.

The requirements of the method are that pentachlorophenol should have a detection limit of 100 ng/l or less and a RSD value of 15% at 1000 ng/l.

The current method typically uses 200 ml of sample and an overall concentration factor of 50 with a small OPTIC 2-200 injection (30µL) which easily reaches the 100ng/L detection requirement using a HP5970 MSD.

This method has been adapted to extract and derivatise a 15 ml sample followed by a large volume injection of the organic extract via an OPTIC 2-200 onto a HP5972 MSD using the FOCUS XYZ Sample Processing Robot.

Good quality results can be obtained using this approach which scales down the use of harmful materials and saves substantial labour time.

Instrumentation

- ATAS FOCUS XYZ Sample Processing Robot
- ATAS OPTIC 2-200 Programmable Injector
- HP 5972 GC-MSD System

Principle

1. Prepare sample vial with 15 ml of sample + 1ml of saturated borax + 25µl 13C pentachlorophenol internal standard.
2. Allow the FOCUS XYZ Sample Processing Robot to add 2ml of 0.1% pentafluorobenzoyl chloride in 2,2,4-trimethyl-pentane to the sample vial from the solvent reservoir.
3. Vortex shakes in dual directions for 15 min.
4. Allow the extract to settle for 5 min.
5. Crack any emulsion formed.
6. Allow to settle for 5 min.
7. Remove 100 µl from the sample vial and inject onto the HP5972 MSD system via an OPTIC 2 Injector.

Compound List

Phenol	Cresols (3)
Xylenols (6)	Chlorophenols (3)
Dichlorophenols (2)	2,4,6-Trichlorophenol
2,3,5,6-Tetrachlorophenols	Pentachlorophenol
Chloromethylphenols (2)	Chloroxylenol (1)

Performance of the automated method

Linear range using the GC-MS:
     0-200 µg/l for all selected phenols.

The spiked river water samples run in these tests cover the range of 0.1 to 180 µg/l.

Reproducibility of the extraction and injection combined:
     Data supplied for pentachlorophenol (the smallest peak in the chromatogram).

7.8% RSD for 11 River Tyne samples spiked at 100 ng/l. (The required detection limit).

Estimated detection limit using HP5972 MSD system in EI for pentachlorophenol following automated extraction 25 ng/l.

Relative recovery as estimated against a Milli-Q extracted standard at 200 ng/l:
     104% for 200 ng/l in borehole water,
     103% for 200 ng/l in river Tyne water,
     101% for 200 ng/l in seawater,
     97.1% for 200 ng/l in final effluent.

Total cycle time for first analysis 55 minutes per sample.

Conclusion

The FOCUS XYZ Sample Processing Robot delivers comparable results to the existing manual method. Both precision and recovery performance are marginally better with the automated system although the working range is reduced.
All statistical figures for the automated method are for pentachlorophenol. The figures for the remaining phenols (see compound list) are comparable or better.