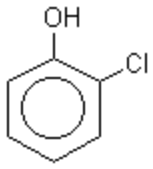
SPE Application Note for
Phenols from Water (Optimized for GC analysis)

This method was developed for the extraction of phenols from water samples using a non-polar retention mechanism. This method was developed for use with subsequent analysis by GC. If the analytical method of choice is reversed phase HPLC, please refer to application note IST1036A.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: ENV+ 200 mg/6 mL (Part # 915-0020-C)

Pre-treatment:	Adjust the sample (1L) to around pH 2.0 using 6.0 M hydrochloric acid. Add internal standard solution at this stage.
Solvation:	Solvate the column with methanol (3 mL).
Equilibration:	Rinse the column with deionized water adjusted to pH 2.0 with 6.0 M HCl (3 mL).
Sample application:	Apply the sample at a flow rate of up to 60 mL/min.
Interference elution:	Elute inteferences with deionized water (10 mL). Dry the column thoroughly by vacuum aspiration for at least 10 mins.
Analyte elution:	Elute analytes with 5:95 (v/v) acetic acid / ethyl acetate (2 x 1.5 mL). Apply the first aliquot to the column, and soak for 1 minute. Apply the second aliquot and combine the fractions. Evaporate the eluent under nitrogen at low temperature if further concentration is required.
Structure	Various. 2-chlorophenol is shown. 
Structural considerations	The analytes are generally small, relatively polar compounds. The -OH group is ionizable, and the sample is pH adjusted to ensure protonation of this group, and therefore retention on the sorbent.
Matrix considerations	The analytes are extracted from a polar aqueous matrix.
Analytical method	GC

Column: DB-624, 30m x 0.32mm i.d., 1.8um film
Carrier gas: helium, 2 mL/min
Injection: splitless at 250 C
Temperature: 60 C for 1 minute, 60-250 C, 10 C/min, 250 C for 10 mins
Detector: FID, 280 C

Reagents

General comments

1. The internal standard should be added to the water matrix in a water miscible carrier solvent such as methanol.

2. It may be helpful to pre-rinse all glassware used in the method with acidified water (pH 2) to prevent loss of analyte. The sample bottle should be rinsed with acidified water after the sample has been poured out, and the rinsings added to the column.

3. It is very important to dry the column thoroughly by vacuum aspiration prior to elution with a non water miscible solvent.

4. The soak step used during elution allows the analytes to be eluted in the minimum volume.

5. The following recoveries were achieved using this method:-

	% recovery
2-chlorophenol	101
phenol	103
2-methylphenol	106
2-nitrophenol	98
2,4-dichlorophenol	100
3-chlorophenol	108
4-chloro-2-methylphenol	106
2,4,5-trichlorophenol	90
3-nitrophenol	106

6. Reference: Liesel von Metz, personal communication, 1995.

7. Previous # IST3011.

ISOLUTE column part numbers represent the product configuration of choice for use with a vacuum sample processing station. For 96-well and alternative column configurations compatible with any SPE automation system, please contact Biotage.

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