Introduction

The mixed-mode GC and GC/MS determination of pollutants in the environment remains a challenge for the analytical laboratory. Acidic compounds, such as phenols, are typically determined using GC or GC/MS, but these techniques cannot be used for the determination of basic compounds. Basic drugs such as ibuprofen and basic drugs such as phenylpropanolamine were effectively extracted and analyzed at sub-µg levels. However, these SPE methods were designed to be used with liquid chromatography and atmospheric pressure ionization mass-spectrometry (LC/MS). The mixed-mode sorbents utilized in these methods, such as methanol with aqueous ammonia, are not generally suitable for use with gas chromatography. In this study, we have applied the mixed-mode sorbent technology for the analysis of acidic, basic and neutral compounds. The mixed-mode sorbent technology has been applied to the analysis of a test mixture containing 33 compounds (acids, bases and neutrals) listed in EPA method 8270C. Structures for a representative acid, base and neutral compound from this list are shown below.

Mixed-Mode Cation Exchange Sorbents

Mixed-mode cation-exchange sorbents, such as Oasis MCB, have been utilized for GC applications because aqueous ammonia or organic amines in methanol/water have been used for elution of the cationic species. In this study, we utilized an SPE eluent prepared from anhydrous ammonia (7 M) in methanol (vol/vol 49:1:46). The eluent used in this study was 10% of the methanolic ammonia in methylene chloride. This eluent was effective for simultaneous elution of acids and bases from the Oasis MCB sorbent.

Results

SPE Protocol for This Study

Figure 1 shows the general SPE protocol for this study. Prior to analysis, the sample (250 ml) was acidified with HCl and neutralized with sodium thiosulfate. Oasis MCX cartridges (6 cc, 150 mg) were used in this study.

GC/MS and GC/NPD

Figure 4 shows GC/MS chromatograms obtained from a 20 µg/L chlorinated tap water sample. The area highlighted in blue represents the pH range with high retention of both acids and bases.

Discussion

For good recovery of compounds 1-9, it is essential to avoid evaporative losses; do not evaporate to dryness. Recovery of chlorinated phenols was reduced because of interactions with the sodium sulfate in the presence of ammonia. To improve recovery of chlorinated phenols use a two step elution of the cartridge, first elute with 3 ml of 95:5 DCM/methanol, then with 3 ml of 90:10 DCM/methanolic ammonia.

Recovery of some compounds is lower from the high humic content river water (10 mg/L TOC). Since much of the humic material is retained on the sorbent, a 500 mg cartridge is recommended for high humic content samples.

Preliminary experiments indicate that for LOQ below 5 µg/l, a 0.5 l sample should be processed using a 500 mg cartridge, with a final volume of 0.5 ml.

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