Solid Phase Extraction (SPE) of Illicit Drugs - Can a Single Sorbent Cater to Acidic, Neutral and Basic Drugs in Biological Matrices?

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AIMS: Illicit drugs encountered in forensic and toxicological laboratories consist of acidic, neutral and basic compounds of widely different polarities. To isolate these drugs from biological matrices such as urine, plasma and whole blood, solid phase extraction (SPE) is commonly used. Documented literature indicates the use of a variety of silica and polymer based sorbents with hydrophobic and cation exchange interaction characteristics for eliminating proteinaceous and other organic/inorganic contaminants for SPE-based clean up of biological samples prior to GC/MS or LC/MS analysis. However, sorbent selection still remains a dilemma. In this presentation, we explore three silica-based and two polymeric sorbents for the purification of (1) THC and its metabolite THC-carboxylic acid, (2) Cocaine and its metabolites benzoylecgonine (BE) and cocaethylene (CE) and (3) Diazepam and related benzodiazepine drugs. Both GC/MS and LC/MS were used for analysis.

METHODS: Urine samples containing THC, THC-COOH, cocaine, BE, CE, diazepam, lorazepam and temazepam (15-100 ng/mL each), were extracted through Strata-X-C, -Screen-C, -X, C-18E and C-8 cartridges (30-100 mg/mL) using a variety of washes and elutions.

RESULTS: Strata-X-C is a styrene-divinylbenzene polymer with sulfonic acid functionalities and is a strong cation exchange sorbent. It retains THC and THC-COOH, as well as some of the benzodiazepines, even with a 30% acetonitrile or 50% methanol wash and elution with methanol furnishes very clean extracts. Cocaine and its metabolites and some benzodiazepines are eluted from this sorbent with 5% ammonium hydroxide in methanol. For all drugs, recoveries are greater than 90%. Similar results are obtained with the mixed mode silica-based strata-Screen C (hydrophobic and strong cation exchanger), but for hydrophobic molecules like THC and THC-COOH, a less stronger organic wash (20% methanol) had to be used for maximizing recoveries. For neutral sorbents such as the silica based C18 or C8, only a less stringent organic wash can be performed, which resulted in incomplete elimination of contaminants for all the drugs tested. The polymeric polar neutral sorbent strata-X could sustain a stronger organic wash for THC and THC-COOH, but for benzodiazepines, only a milder wash could be used. From this comparative evaluation of silica and polymeric SPE sorbents, it emerges that the polymeric strong cation exchanger strata-X-C is the most versatile for all kinds of drugs in furnishing cleaner extracts along with excellent recovery yields. However, caution should be exercised in the case of nitrogen containing compounds, since factors like pKa (acidity) and structural features play a significant role in determining whether such drugs would elute in the methanol fraction or under basic elution conditions. For example, Diazepam elutes from strata-X-C only with methanol containing 5% ammonium hydroxide, while Lorazepam and Temazepam come down in methanol. On the other hand, the strongly basic Cocaine and metabolites need basified methanol elution.

CONCLUSIONS: Overall, strata-X-C appears to be closest to a universal solution for the SPE-based sample purification of drugs in biological matrices.

Keywords: Solid phase extraction, Sorbents, Biological matrices