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SPE Application Note for Barbiturates from Urine using C8 on RapidTrace

The following method was developed for the extraction of barbiturate drugs from urine using a non-polar retention mechanism.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: C8 100 mg / 1mL, Part number 290-0010-A or C8 100 mg / 3 mL, Part number 290-0010-B

Pre-treatment: Add 1 mL phosphate buffer pH-6 to 2 mL of urine.

Solvation: Condition column with 2 mL of methanol at 10 mL per minute.

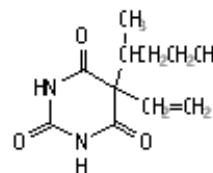
Equilibration: Rinse column with 2 mL of deionized water at 10 mL per min.
Rinse column with 1 mL of phosphate buffer, pH-6 at 10 mL per min.

Sample application: Load sample at 2 mL per min.

Interference elution: Rinse column with 3 mL of deionized water at 5 mL per minute.
Purge cannula with 4 mL of deionized water at 30 mL per min.
Dry under -20 psig vacuum or 20 psig nitrogen for 1/2 minute.
Rinse column with 2 mL of deionized water at 4 mL per minute.
Dry under -20 psig vacuum or 20 psig nitrogen for 1/2 minute.

Analyte elution: Elute analyte with 2 mL of hexane/ethyl acetate (1:1, v/v) at 2 mL per minute.
Purge cannula with 5 mL of methanol at 30 mL per min.
Purge cannula with 5 mL of deionized water at 30 mL per min.
Reconstitute in 50uL BSTFA. Heat for 15 min at 60 C.

Structure These compounds are barbituric acids. The structure of secobarbital is shown here.



Structural considerations Advantage is taken of the hydrophobic character of this group of analytes for the extraction.

Matrix considerations The analyte is being extracted from an aqueous matrix of high ionic strength.

Analytical method GC-MS

Reagents 1. 0.05 M sodium or potassium phosphate buffer
2. Methanol

IST 1063 A

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3. Hexane/ethyl acetate 1:1

General comments Derivatization is recommended but not required prior to GC analysis.

ISOLUTE column part numbers represent the product configuration of choice for use with a vacuum sample processing station. For alternative configurations compatible with any SPE automation system, please consult your IST Distributor.

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