SPE Application Note for Phencyclidine from Urine

This application was developed for the extraction of phencyclidine and other basic drugs from urine for drugs of abuse applications. Typical recovery for PCP is > 85%.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: Confirm HCX, 130 mg 1 or 3 mL (Part # 902-0013-A or 902-0013-B)

Pre-treatment: To 2 mL urine add 100 uL of internal standard (phencyclidine -D5 for GC-MS) and 1 mL of 0.05 M phosphate buffer, pH 6.0. The sample pH should be in the range of 4-6. Adjust if necessary.

Solvation: Condition column with 1 mL of methanol at 2-4 mL/min

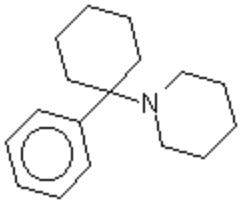
Equilibration: Equilibrate column with 1 mL of deionized water at 2-4 mL/min, followed by 1 mL of 0.05 M phosphate buffer, pH 6 at 2-4 mL/min

Sample application: Apply the sample at a flow rate of 1-2 mL/min

Interference elution: Rinse column with 2 mL of deionized water at 2-4 mL/min, followed by 1 mL of 0.01 M HCl at 2-4 mL/min, and then 2 mL of methanol at 2-4 mL/min Dry column for 0.5 min at -20 psig.

Analyte elution: Elute sample with 1.2 mL of methanol/ammonia, 98:2 at 1.5 mL/min Evaporate to dryness with nitrogen at room temperature. Reconstitute in 50 uL of BSTFA and heat at 70 C for 15 min. Inject 2 uL into GC.

Structure This is a 3 ring compound with a single tertiary amine group.



Structural considerations The analyte has an ionizable amine group, which is exploited through cation exchange interactions. The pKa is approximately 8, so the compound is fully ionized at pH values of less than 6.

The analyte also has significant non-polar characteristics, and both hydrophobic and ion exchange retention mechanisms are used to give a clean extract.

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

Analytical method GC-MS

Reagents

a) 0.05 M phosphate buffer, pH 6.0. Weigh 6.8 g of potassium dihydrogen orthophosphate into a 1 L volumetric flask. Dissolve in 900 mL of deionized water. Adjust pH to 5.9 - 6.1 using 1.0 M potassium hydroxide. Dilute to 1 L with deionized water.
b) 0.01 M HCl To a 250 mL volumetric flask containg 200 mL of deionized water, add 2 mL of concentrated HCl.
c) Methanol/ammonium hydroxide 98:2 Add 2 mL of concentrated ammonium hydroxide to 98 mL methanol. Mix thoroughly. Prepare fresh daily.

General comments

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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