



SPE Application Note for LSD from Whole Blood (HPLC)

This method was developed for the extraction of lysergic acid diethylamide (LSD) from whole blood using a mixed non-polar and cation exchange retention mechanism.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: Confirm HCX 130 mg/10 mL Part # 902-0013-H

Pre-treatment: To the blood sample (1 mL) add 50 ul of internal standard solution (50 ug/mL lysergic acid methylpropylamide in methanol), 5 mL of phosphate buffer (0.1 M, pH 6.0) and 0.5 mL of methanol. Vortex for 1 min, and centrifuge at 3500 g for 20 mins.

Solvation: Solvate the column with methanol (3 mL) at a flow rate of 1-2 mL/min.

Equilibration: Rinse the column with distilled deionized water (2 mL) followed by phosphate buffer (0.1 M, pH 6.0, 2 mL) at a flow rate of 1-2 mL/min.

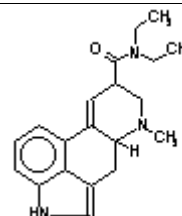
Sample application: Apply the supernatant at a flow rate of 1-2 mL/min.

Interference elution: Elute interferences with distilled deionized water (2 mL), 1.0 M acetic acid (1 mL) and methanol (3 mL) respectively, at a flow rate of 1-2 mL/min. Dry the column under vacuum (-10 to -15 "Hg) for 5 mins.

Analyte elution: Elute the analyte with 3 mL of 4:1 (v/v) dichloromethane/isopropanol containing 2% ammonia (v/v). Consider applying the elution solvent in two aliquots of 1.5 mL, with a soak step in between.

Evaporate the eluate to dryness under a gentle flow of nitrogen on a heating mantle set to 40 C. Redissolve the sample in 50 ul of HPLC mobile phase, and use 20 ul for the HPLC analysis.

Structure As shown.



Structural considerations The analyte has both non-polar and basic characteristics, and these can both be utilized to produce a very clean extract.



Matrix considerations

Whole blood is viscous, and needs to be diluted to ensure a smooth flow through the SPE column.

The addition of methanol in the sample pre-treatment stage precipitates blood proteins, and leads to increased recoveries of analyte.

The mixed mode extraction procedure allows a rigorous interference rinse stage, leading to very clean extracts.

Analytical method HPLC with fluorescence detection.

Column: 25 cm x 4.6 mm Hypersil ODS
Mobile phase: 0.12 M ammonium acetate buffer, pH 8.0/acetonitrile (68:32, v/v) containing 0.3% triethylamine (v/v), isocratic
Flow rate: 1.2 mL/min
Detection: excitation 303 nm, emission 413 nm

N.B. Mobile phase (and elution solvent) should be made up fresh daily to ensure full recoveries and improved peak shapes.

Reagents

General comments 1. Reference: Battah AH, Oliver JS and Anderson RA, Journal of Substance Misuse (1996) 1, 155-159.

2. Previous # IST2015PM

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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