

**LYSERGIC ACID DIETHYLAMINE AND PHENCYCLIDINE**

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**DETERMINATIVE METHOD****A. INTRODUCTION****Theory**

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The drugs of interest are extracted from an alkaline tissue sample with ethyl acetate. The organic phase is then shaken with dilute hydrochloric acid. The pH of the acid is adjusted with base and extracted with chloroform. The chloroform layer containing the drugs is then removed and spotted on two separate TLC plates, developed, and visualized with the appropriate spray reagents.

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## **DETERMINATIVE METHOD**

### **B. EQUIPMENT**

#### **Apparatus**

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An equivalent can be substituted for any apparatus listed below if necessary.

- a. 50 mL polypropylene screw-top disposable centrifuge tubes.
  - b. Mechanical horizontal shaker (240 cycles/min).
  - c. Vortex test tube mixer.
  - d. Centrifuge capable of 3000 rpm.
  - e. Lab timer.
  - f. 15 mL glass centrifuge tube.
  - g. Water bath, 15-20° C.
  - h. Repipet dispensers, 5 mL and 30 mL.
  - i. 10 mL class A volumetric flasks.
  - j. 100 mL volumetric flasks.
  - k. 100  $\mu$ L syringe (glass).
  - l. E. Merck 60F-254 silica gel plates.
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## DETERMINATIVE METHOD

## C. REAGENTS AND SOLUTIONS

Reagent and  
Solution List

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- a. Ethyl acetate—distilled in glass.
  - b. 50% NaOH—reagent grade.
  - c. Granular sodium sulfate.
  - d. 0.5N HCl—reagent grade.
  - e. Chloroform—distilled in glass.
  - f. LSD (lysergic acid diethylamide)—0.02 mg/mL in ethyl acetate.
  - g. PCP (phencyclidine HCl)—0.117 mg/mL in ethyl acetate.
  - h. Acetone—distilled in glass.
  - i. Methanol—distilled in glass.
  - j. Iodoplatinate (potassium)—for alkaloids and other organic nitrogen compounds. Mix 3 mL 10% hexachloroplatinic (IV) acid solution with 97 mL water and add 100 mL 6% potassium iodide solution in water. Freshly prepare the reagent before use.
  - k. Dimethylaminobenzaldehyde-hydrochloric acid—for indole derivatives. Dissolve 1 g 4-dimethylaminobenzaldehyde in 50 mL 36% hydrochloric acid and add 50 mL ethanol.
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**DETERMINATIVE METHOD**

**E. EXTRACTION PROCEDURE**

**Sample Extraction**

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- a. Add 1 mL of 50% sodium hydroxide to 10 g of homogenized meat in a 50 mL polypropylene centrifuge tube.
  - b. Add 5 g sodium sulfate and 30 mL ethyl acetate to the mixture.
  - c. Shake the solution on a horizontal shaker for 10 min.
  - d. Centrifuge the tube at 3000 rpm for 5 min.
  - e. Decant the ethyl acetate upper layer into another 50 mL polypropylene centrifuge tube.
  - f. Remove and discard any oily residue that transfers over with a Pasteur pipet.
  - g. Add 5 mL of 0.5N hydrochloric acid, shake for 3 min using a horizontal shaker, and centrifuge at 2000 rpm for 3 minutes.
  - h. Aspirate and discard the ethyl acetate (upper layer).
  - i. Transfer the 5 mL of 0.5N hydrochloric acid solution to a 15 mL graduated glass centrifuge tube.
  - j. Cool the solution in a water bath for 5 min and add 1 mL of a 50% NaOH solution; cool again for 3 min.
  - k. Agitate by vortexing for 30 sec.
  - l. Cool the solution again in a water bath for 2 min.
  - m. Add 100 mL of chloroform to the solution (alkaline); shake manually for 3 min.
  - n. Centrifuge for 5 min at 200 rpm.
  - o. Aspirate and discard half of the aqueous alkaline layer:
    - i. With a 100  $\mu$ L syringe, remove two 40  $\mu$ L aliquot portions of the chloroform layer and spot separately on different silica gel plates. Spot a detectable amount of LSD and PCP separately on each plate.
    - ii. Develop the plates in the following solvent system—acetone:methanol (80:20). Saturate the tank prior to use.
    - iii. After development, air dry the plates and spray for LSD using P-dimethylaminobenzaldehyde. Spots will be violet on a yellow plate. Spray for PCP using iodoplatinate. Spots will be brown on a yellow plate.

NOTE: Spray lightly—do not overspray.

Rf LSD—0.33; Rf PCP—0.25.

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**DETERMINATIVE METHOD**

**H. HAZARD ANALYSIS**

<b>1. Method Title</b>	Analysis of Lysergic Acid Diethylamide and Phencyclidine in Tissue.		
<b>2. Required Protective Equipment</b>	Safety glasses, plastic gloves, lab coat.		
<b>3. Procedure Steps</b>	<u>Hazards</u>	<u>Recommended Safe Procedures</u>	
C. Reagents			
a. Ethyl acetate e. Chloroform h. Acetone i. Methanol	These organic reagents are very flammable and corrosive and the vapors are extremely irritating to the skin, eyes, and respiratory system.	These reagents should only be used in an efficient fume hood away from any heat-generating devices.	
b. 50% NaOH	Caustic, may cause irreversible skin damage.	Flush immediately with water if any spattering occurs.	
E. Extraction			
o. (iii) Spray for LSD, PCP	Acidic vapors	Spray in fume hood and use some form of a cardboard partition to trap the deflected spray.	
<b>4. Disposal Procedures</b>			
Organic solvent mixtures	See above	Segregate chlorinated from nonchlorinated and hold in designated storage cans until disposed of by the contractor or in-house specialist.	
Extracted tissue waste	Harmful vapors	Allow organic vapors to dissipate in the fume hood and then store waste in a plastic bag awaiting disposal.	