

|                                                   |                                                                    |             |
|---------------------------------------------------|--------------------------------------------------------------------|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL |                                                                    |             |
| Procedure D-03                                    | Extraction and Separations<br>Extraction of Volatile Organic Bases |             |
| Effective Date:                                   | November 20, 2006                                                  | Page 1 of 3 |

**Name of Procedure:**

Extractions and Separations  
Extraction of Volatile Organic Bases

**Suggested Uses:**

This is a general procedure used to isolate and purify volatile basic drugs for further analysis.

**Apparatus Needed to Perform Procedure Including Preparation of the Reagent:**

Fume hood  
Gloves  
Eye protection  
Laboratory coat  
Sodium Hydroxide  
Sulfuric Acid  
Ethyl Ether  
Sodium Sulfate, anhydrous  
Hydrochloric Acid, concentrated  
Conway diffusion dish  
Crystallizing dish  
Watch glass (or glass plate)  
pH Test paper  
Reagent bottles  
Spatula, small  
Glass stirring rod  
Beakers  
Pipets, glass, disposable  
Rubber bulbs  
Filter paper  
Graduated cylinder

|                                                   |                                                                    |             |
|---------------------------------------------------|--------------------------------------------------------------------|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL |                                                                    |             |
| Procedure D-03                                    | Extraction and Separations<br>Extraction of Volatile Organic Bases |             |
| Effective Date:                                   | November 20, 2006                                                  | Page 2 of 3 |

### **Formula for Preparing Reagent:**

#### **20% Sodium Hydroxide Reagent**

1. Weigh out 20 grams of sodium hydroxide.
2. Dissolve in 100 milliliters of water.
3. Pour into a reagent bottle.
4. Properly label reagent bottle.

#### **5% Sulfuric Acid Reagent**

1. Measure 95 milliliters of water into a 100 milliliter graduated cylinder.
2. Add sulfuric acid to a total volume of 100 milliliters.
3. Pour into a reagent bottle.
4. Properly label reagent bottle.

#### **Ethyl Ether Saturated with Hydrochloric Acid Reagent**

1. Place approximately 10 milliliters of concentrated hydrochloric acid into a separatory funnel.
2. Add approximately 50 milliliters of ethyl ether and shake the separatory funnel.
3. Allow the layers to separate.
4. Separate the ethyl ether layer and store in a reagent bottle.
5. Properly label reagent bottle.

### **Expiration Date of Chemical:**

The 20% sodium hydroxide reagent and the 5% sulfuric acid reagent can be used until depleted provided they are stored in an airtight reagent bottle.

The Ethyl ether saturated with hydrochloric acid reagent is prepared as needed and discarded after use.

### **Application of Procedure on Evidence:**

1. Add an amount of sample, equivalent to 5-100 milligrams of the basic drug to 10-20 milliliters of sodium hydroxide reagent in the outer well of a Conway diffusion dish. Crush and grind any tablet or hard material before adding to the well. Place 2-3

|                                                   |                                                                    |             |
|---------------------------------------------------|--------------------------------------------------------------------|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL |                                                                    |             |
| Procedure D-03                                    | Extraction and Separations<br>Extraction of Volatile Organic Bases |             |
| Effective Date:                                   | November 20, 2006                                                  | Page 3 of 3 |

milliliters of the sulfuric acid reagent (5%) in the inner well, cover, and place on a warm surface such as a steambath or hot plate.

**NOTE:** A crystallizing dish fitted with a glass cover and a small beaker can be substituted for the Conway diffusion dish.

2. After 20-30 minutes, remove the dish from the heat source and allow to cool.
3. Transfer the contents of the inner well to a small beaker or test tube and make basic by dropwise addition of sodium hydroxide solution.
4. Extract the basic solution with three 5 milliliter portions of ethyl ether by using a disposable pipet and rubber bulb to mix and separate the organic and liquid phases.
5. The solvent extracts may be dried using magnesium sulfate or sodium sulfate.
6. Add dropwise ether/hydrochloric acid reagent to form the hydrochloride salt, being careful to avoid an excess of acid.
7. If the hydrochloride salt precipitates to produce crystals, collect the crystals on a filter paper, wash with 10-15 milliliters of ether, and allow the crystals to dry on a warm surface. For situations where crystals do not form, evaporate the ether under a stream of nitrogen while applying a moderate amount of heat from a heating device.

### **Safety Concerns:**

Ethyl ether is extremely flammable. Sodium hydroxide and hydrochloric acid are caustic and can cause chemical burns.

### **Literature References:**

Clark, C. C., A Simple Identification Procedure For Some Volatile Amines , **MICROGRAM**, Vol. VI, NO. 5, May 1973, p. 78.

**Forensic And Analytical Chemistry of Clandestine Phenethylamines**, CND Analytical, Auburn, 1994, p. 8.