# Western Regional Laboratory Drug Chemistry Procedure Manual Effective Date: June 19,1997

# Name of Procedure:

Extractions and Separations
Extraction of Diethylpropion from a Time-release Preparation

## Suggested Uses:

This procedure is used to isolate diethylpropion from a time-release tablet.

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

Fume hood

Gloves

Eye protection

Laboratory coat

Hydrochloric Acid

Ethyl Ether

Sodium Sulfate, anhydrous

pH Test paper

Volumetric flask, 10-25ml

Reagent bottles

Spatulas, small

Scalpel and blade

Glass stirring rods

**Beakers** 

Pipets, glass, disposable

Rubber bulbs

Filter paper

Centrifuge, Hamilton Bell, Vanguard V6000

Centrifuge tubes, 15ml polypropylene

Sodium Hydroxide Solution, concentrated

## Formula for Preparing Reagent:

# 5% Hydrochloric Acid Reagent

- 1. Measure 95 milliliters of water in a 100 milliliter graduated cylinder.
- 2. Bring to total volume (100ml) with concentrated hydrochloric acid.
- 3. Pour into a reagent bottle.
- 4. Properly label reagent bottle.

# Formula for Preparing Reagent (continued):

# 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:**

5% Hydrochloric acid reagent may be used until depletion if stored in an airtight reagent bottle.

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

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

- 1. Crush and grind tablet (or portion), after first removing film coating with a scalpel if necessary, and then transfer 200-300 milligrams of the powder to a centrifuge tube.
- 2. Mix the powder well with 10 milliliter of hydrochloric acid solution, centrifuge for 3-5 minutes, and
- 3. Extract with ethyl ether and discard washings.
- 4. Make the solution basic by adding sodium hydroxide solution, checking pH with test paper.
- 5. Extract the basic solution with ethyl ether.
- 6. Ethyl ether extracts may be dried using magnesium sulfate or sodium sulfate.
- 7. Add ether/hydrochloric acid solution to precipitate diethylpropion hydrochloride,

being careful

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# <u>Application of Procedure on Evidence</u> (continued):

8. The diethylpropion hydrochloride can be recovered for further analysis by evaporating the ether using heat and nitrogen, or by collecting the crystals on filter paper and washing with ether.

## **Safety Concerns:**

Ethyl ether is extremely flammable. Sodium hydroxide and hydrochloric acid solution are caustic and corrosive.

## **Literature References:**

Adapted by Chemist J. R. Daniel, N. C. State Bureau of Investigation, Raleigh, 1992.

Shriner, R. L., Fuson, R. C., Curtin, D. Y., <u>The Systematic Identification of Organic Compounds</u>, 5th Ed., Wiley and Sons, New York, 1964, pp.88-106.

Moffat, A. C., Ed., <u>Clarke's Isolation and Identification of Drugs</u>, 2nd Ed., The Pharmaceutical Press, London, 1986, p. 52.