

Drug Chemistry Section
Drug Chemistry Procedure Manual
Effective Date: September 1, 1996

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.

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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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Application of Procedure on Evidence (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., **The Systematic Identification of Organic Compounds**, 5th Ed., Wiley and Sons, New York, 1964, pp.88-106.

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