

Name of Procedure:

Extractions and Separations
Extraction of Organic Bases

Suggested Uses:

This is a general procedure used to isolate and purify basic compounds (alkaloids) for further analysis.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume Hood
Gloves
Eye protection
Laboratory coat
Pipet with bulb
Graduated cylinder
Glass stirring rod
pH Test paper
Separatory funnel
Funnel
Hydrochloric Acid, concentrated
Ethyl Ether
Sodium Sulfate, anhydrous
Sodium Hydroxide Solution, concentrated
Methylene Chloride
Hexane
Chloroform
Isopropanol
acetonitrile
Sodium Bicarbonate
Magnesium Sulfate, anhydrous
Beakers
Filter Paper

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.

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 Reagents:

5% Hydrochloric acid reagent can be used until depleted provided it is stored in an airtight reagent bottle.

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

Application of Procedure on Evidence:

1. Dissolve the sample in the 5% hydrochloric acid reagent. Check the pH with test paper to assure solution is acidic.
2. Extract the acid solution with ethyl ether and discard the ether washings.
3. Make the acidic solution basic by adding concentrated sodium hydroxide solution or sodium bicarbonate. Check the pH of the solution with test paper to assure solution is basic.

Application of Procedure on Evidence (continued):

4. Extract the basic solution with a suitable organic solvent [such as, ethyl ether, chloroform, hexane, methylene chloride, 3:1 chloroform/isopropanol]. The chemical properties of the organic base being extracted and the chemical properties of the other substances mixed with the organic base being extracted will determine which organic solvent is used.
5. Organic solvent extracts may be dried using magnesium sulfate or sodium sulfate.
6. If the organic base being extracted is not volatile, evaporate the solvent in the hood, leaving the extracted organic base.
7. If the organic base is volatile or if the HCl salt form of the organic base is desired, add dropwise, the ethyl ether saturated with hydrochloric acid reagent. Check the pH with test paper to avoid excess hydrochloric acid being added.
8. If the HCl salt precipitates to produce crystals, collect and wash crystals with ethyl ether and allow crystals to dry.
9. If crystals do not form, evaporate the organic solvent using dry nitrogen and low heat, leaving the HCl salt of the organic base.

NOTE: The extractions may be carried out using separatory funnels, test tubes, glass sample vials or beakers.

Safety Concerns:

Ethyl ether and other organic solvents are extremely flammable. Sodium Hydroxide and Hydrochloric Acid are caustic and corrosive.

Literature References:

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