

Name of Procedure:

Extractions and Separations
Extraction of Organic Acids

Suggested Uses:

This is a general procedure used to isolate and purify acidic drugs 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
Sodium Hydroxide
Sodium Sulfate
Magnesium sulfate
Ethyl Ether
Beakers
Filter paper
Chloroform

Formula for Preparing Reagent:

5% Sodium Hydroxide Reagent

1. Weigh out 5 grams of sodium hydroxide.
2. Dissolve in 100 milliliters of water.
3. Pour solution into a reagent bottle.
4. Properly label reagent bottle.

Expiration Date of Reagent:

The reagent can be used until depleted provided it is stored in an airtight reagent bottle.

Application of Procedure on Evidence:

1. Add an amount of sample, equivalent to 5-200 milligrams of the acidic drug to be extracted, to 5-10 milliliters of sodium hydroxide solution in a separatory funnel. The size of the separatory funnel used will depend upon the amount of sample used. Crush and grind any tablet or hard material and remove the powder from capsules before adding to the separatory funnel.
2. Extract the basic solution with two 10-20 milliliter portions of ethyl ether and discard the ether washings.
3. Make the solution in the separatory funnel acidic by adding concentrated hydrochloric acid. Check the pH of the solution with test paper.
4. Extract the acid solution with two 10 milliliter portions of ethyl ether or chloroform. The solvent extracts may be dried using magnesium sulfate or sodium sulfate.
5. Evaporate the solvent to give the free acid compound.

Note: The extractions can be carried out using beakers or test tubes instead of a separatory funnel. For this procedure use glass pipets equipped with rubber bulbs to mix the organic solvents with the aqueous solutions, and to separate the layers. The rest of the procedure is the same in respect to solvent drying, and evaporation.

For more concentrated samples, or for smaller amounts of drugs where only mass spectra data is to be obtained, the separations and sample recovery can be carried out in 3.7 milliliter glass vials.

Safety Concerns:

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

Literature References:

Shriner, R. L., Fuson, R. C., and Curtin, D. Y., **The Systemic Identification of Organic Compounds**, 5th Ed., Wiley and Sons, New York, 1964, pp 67-106.

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

Berg, E. W., **Physical and Chemical Methods of Separation**, McGraw-Hill, New York, 1963, pp. 6-7.

Adams, R., and Johnson, J. R., **Laboratory Experiments in Organic Chemistry**, 4th Ed., MacMillan, New York, 1949, pp. 103-115.

Canaff, R. F., **A Basic Training Course for Forensic Drug Chemists**, BNDD publication, U. S. Government Printing Office, 1972.

Leffler, J. E., **A Short Course in Modern Organic Chemistry**, MacMillan, New York, 1973, pp. 17-27.