
Appendix L:

Extraction Procedures

Extraction Solubility Guidelines

- Basic drugs are soluble in acidic ($\text{pH} < 7$) water solutions, but acidic drugs are not.
- Acidic drugs are soluble in basic ($\text{pH} > 7$) water solutions, but basic drugs are not.
- Neutral drugs can be soluble in acidic and basic water solutions.
- Freebase, free acid, and neutral drugs are soluble in organic solvents.
- Freebase and free acid drugs are insoluble in water.
- To determine if a drug is acidic or basic, look at the salt form name:
 - Basic drugs have an acid as part of their salt form [e.g., hydrochloride (HCl), sulfate (H_2SO_4), acetate (CH_3COOH), etc.].
 - Acidic drugs have an alkali metal as part of their salt form name [e.g., sodium (Na) or potassium (K)].
 - Neutral drugs do not have an associated salt form.

Particle Picking

- Remove particles that appear to be the substance in question with tweezers.
- Analyze by IR spectroscopy.

Dry Extraction

- Place powder sample in an organic solvent in which the component of interest is soluble and the diluents and adulterants are not.
- Isolate and evaporate the solvent.
- Analyze by IR spectroscopy.

Dry Wash

- Place powder sample in an organic solvent in which the component of interest is NOT soluble and the diluents and adulterants ARE.
- Remove the solvent from the dry solid.
- Analyze by IR spectroscopy.
- Multiple solvents may be required to remove all diluents and adulterants.

General Liquid/Liquid Extraction Procedure

- Dissolve the sample in an acidic aqueous solution. (The basic drugs will dissolve into the acidic aqueous solution.)
- Add an organic solvent, and agitate. (The acidic and neutral drugs will dissolve into the organic solvent.)
- Allow the liquids to separate.
- Remove and discard the organic solvent.
- Make the aqueous liquid basic. (The basic drugs will come out of solution.)
- Add an organic solvent, and agitate. (The basic drugs will dissolve into the organic solvent.)
- Allow the liquids to separate.
- Remove and retain the organic solvent.
- Analyze the solvent.
 - The organic solvent can be analyzed by GCMS.
 - Or, the solvent can be evaporated, and the residue can be analyzed via IR spectroscopy.

General Ion-Pairing Extraction Technique

- Dissolve the sample in an acidic aqueous solution with a high concentration of halide ions. (The use of HCl or the addition of a chloride salt will provide the needed environment. The basic drugs will dissolve into the acidic solution.)
- Add a chlorinated solvent (chloroform) and agitate. (The ion-pairing drug will extract into the organic solvent along with the acidic and neutral drugs.)
- Allow the liquids to separate.
- Remove and save the organic solvent.
- Add an acidic aqueous solvent, void of chloride ions, to the organic solution, and agitate. (The ion-pairing drugs will dissolve into the acidic solution.)
- Allow the liquids to separate.
- Remove and discard the organic solvent.
- Make the aqueous liquid basic. (The ion-pairing drugs will not be soluble in the basic aqueous solution.)
- Add an organic solvent, and agitate. (The ion-pairing drugs will dissolve into the organic solvent.)
- Allow the liquids to separate.
- Remove and retain the organic solvent. (The organic solvent can be analyzed by GCMS. The solvent can be evaporated, and IR spectroscopy can be used to analyze the residual freebase drug.)

