

**CHEM 8M, Experiment 2** – Two-Base Extraction of Excedrin Components

(1) Extraction / separation

(2) Analysis: TLC &amp; IR

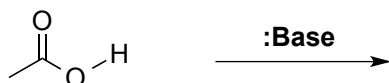
**ACIDIC & BASIC FUNCTIONAL GROUPS (FGs)**(accepts or donates protons,  $H^+$ )

- Carboxylic acids, phenols, amines

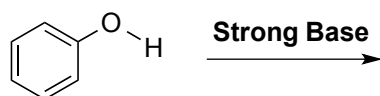
**vs. NEUTRAL FGs**

(not acidic or basic)

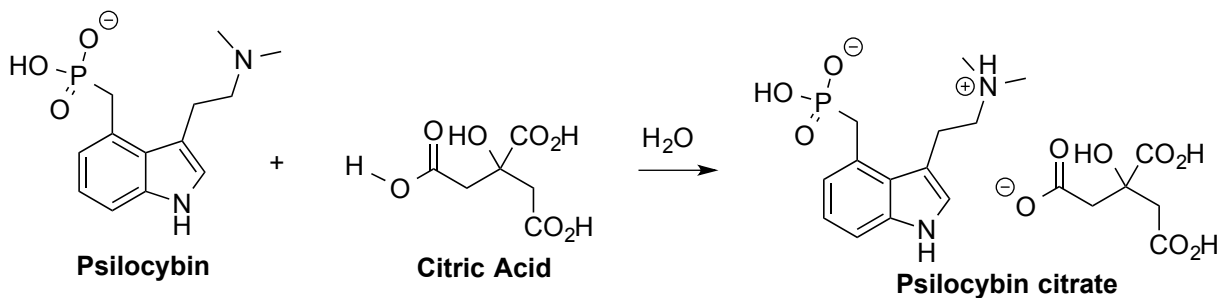
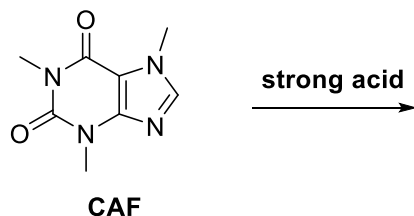
- Esters, alcohols, hydrocarbons



- Phenols



- Amines





**LIQUID-LIQUID EXTRACTION**

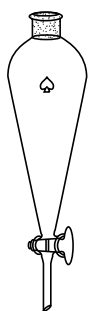
- 2 immiscible liquids have distinct densities and polarities
- Component(s) have a solubility preference for one layer: aqueous (AQ) & organic (ORG)
- PARTITION COEFFICIENT = ratio of solubilities in 2 solvents

*Procedural Lingo...***Basic Extraction**

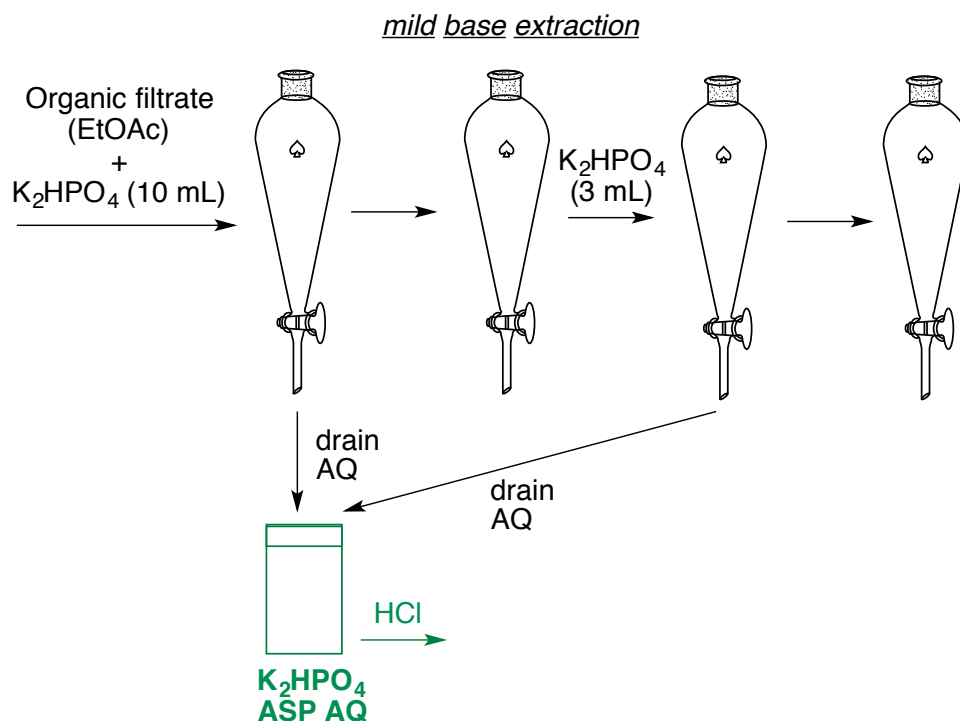
"Extract the ORG layer  
w/ AQ base"

**Acidic Extraction**

"Extract the AQ layer  
w/ ORG solvent"

**Washes**

"Wash the ORG layer  
with sat. NaCl (brine)"

**1. Isolation of Aspirin**

The flowchart illustrates the extraction process for ORG CAF. It begins with a series of five separatory funnels. The first funnel receives 10 mL of KOH. The second funnel receives 3 mL of KOH. The third funnel receives 10 mL of brine. Arrows indicate the flow of the mixture between the funnels. From the first funnel, an arrow labeled 'drain AQ' points to a red rectangular container labeled 'KOH ACE AQ'. From the second funnel, an arrow labeled 'drain AQ' points to the same container. From the third funnel, an arrow labeled 'drain AQ' points to a small cone-shaped flask labeled 'Waste'. From the fourth funnel, an arrow points to a blue cone-shaped flask labeled 'ORG CAF'.

The flowchart illustrates the extraction and purification process for AQ. It begins with a rectangular container on the left. An arrow points from this container to a round-bottom flask labeled "acidic ACE AQ sol'n + EtOAc (15 mL)". From this flask, two arrows branch out to two smaller round-bottom flasks labeled "ORG" and "AQ". An arrow labeled "+ EtOAc (15 mL)" points from the "AQ" flask to a second round-bottom flask. This second flask is labeled "extract AQ" above it. An arrow points from this flask to a third round-bottom flask, which is labeled "combine & wash ORG layers" above it. An arrow labeled "brine (10 mL)" points from this flask to a fourth round-bottom flask. Finally, an arrow labeled "drain AQ" points from the fourth flask to a fifth round-bottom flask labeled "ORG".

**Analysis of Excedrin Components via TLC and IR Spectroscopy**

1. **TLC** – same as Exp 1! TLC mobile phase = 1:2 Hex / EtOAc w/ 1% HAc

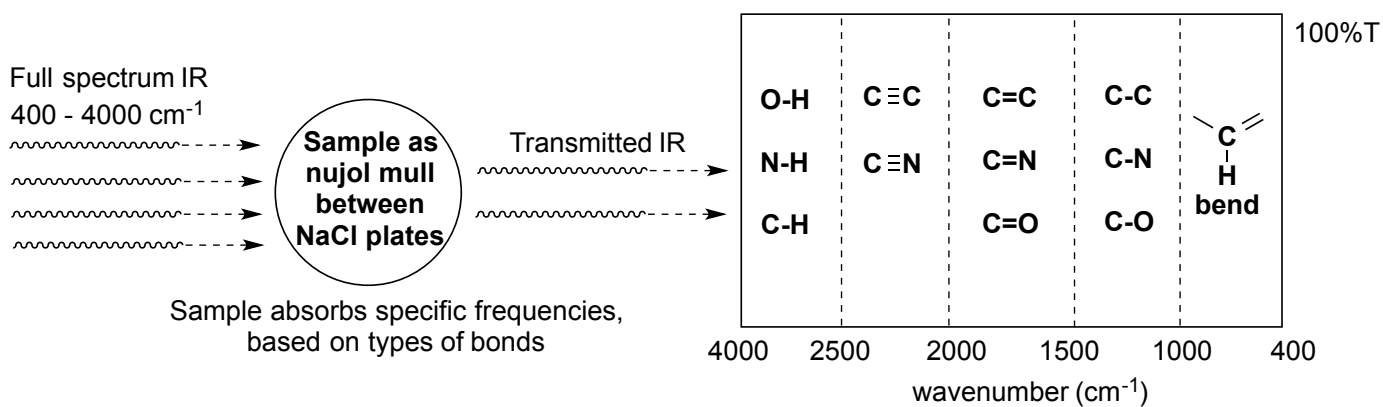
- dilute solids & check for purity before IR analysis

**ASP**

**ACE**

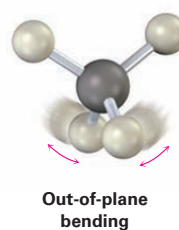
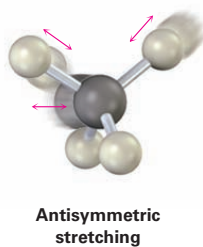
**CAF**

2. **IR analysis** = detection of bonds within specific functional groups

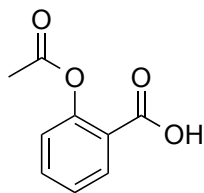
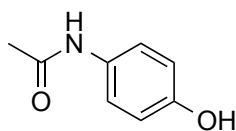
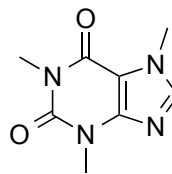


- Frequency of vibration depends on **bond length**

- **Longer bonds** have greater distance to expand/contract = **slower frequency**,  $\downarrow \nu$



Identify the **functional groups** and **expected IR ranges** from IR table on Canvas.

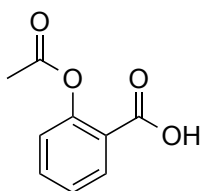
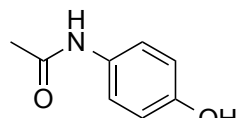
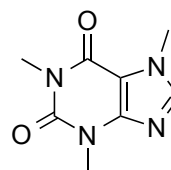
**ASP****ACE****CAF**

### Conjugated vs. saturated double bonds

### Exp 2 Overview



1. EtOAc  
 $\text{K}_2\text{HPO}_4$  (aq)
2. KOH (aq)
3. HCl (conc.)

**ASP****ACE****CAF**