

Name \_\_\_\_\_

Lab Partner \_\_\_\_\_

TA Name \_\_\_\_\_

Section Day \_\_\_\_\_ Time \_\_\_\_\_

**Experiment 2 Worksheet – Column Chromatography**

Each student submits this individually on Canvas after lab

**Pre-Lab Requirements**

1. **Dress for lab** – see safety rules – please arrive a few minutes early
2. **Lab Notebook:** copy templates below into designated notebook
  - **Purpose, scheme, and reagent table**
  - **Procedure Diagrams** – copy templates provided, follow instructions to complete diagrams

**A. Experimental Purpose and Structures of Excedrin Components****B. Reagent Table**

Name	Volume	Density	Mass	milli moles	Molecular Mass	Boiling or melting point	Hazards
Excedrin	-						
Ethyl acetate							
K <sub>2</sub> HPO <sub>4</sub>			-				
KOH			-				
HCl			-				
Aspirin	-						
Acetaminophen	-						
Caffeine	-						

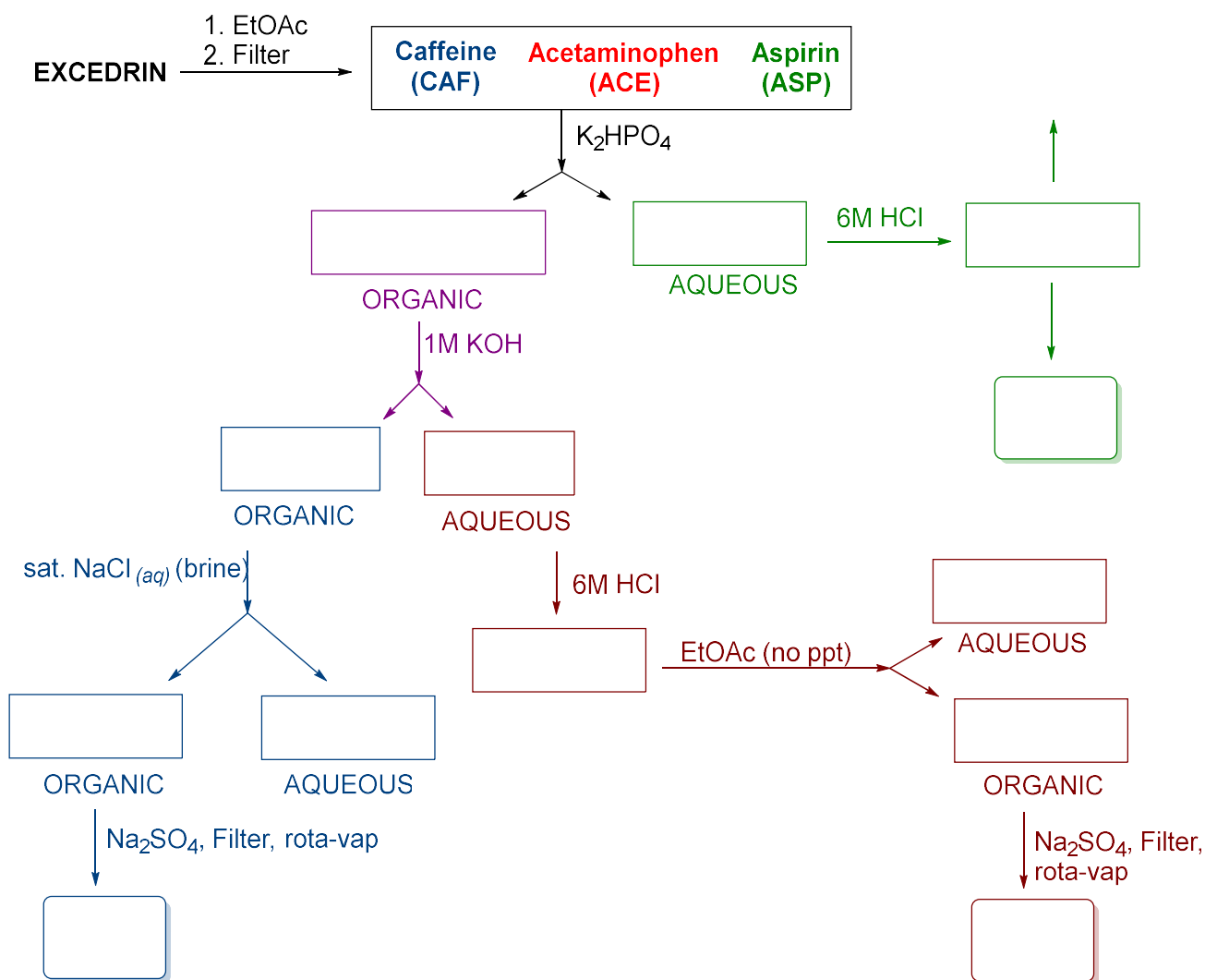
**C. Procedure Diagrams** of key procedural segments on provided templates below

- All labeled equipment, chemical names with amounts, and pertinent safety notes in every step.
- Slugs@home Exp 2 website - Equipment & Safety pages; pictures & videos of each part of the lab.
- The class notes include useful diagrams as well!

**1. Active Ingredients in Excedrin**

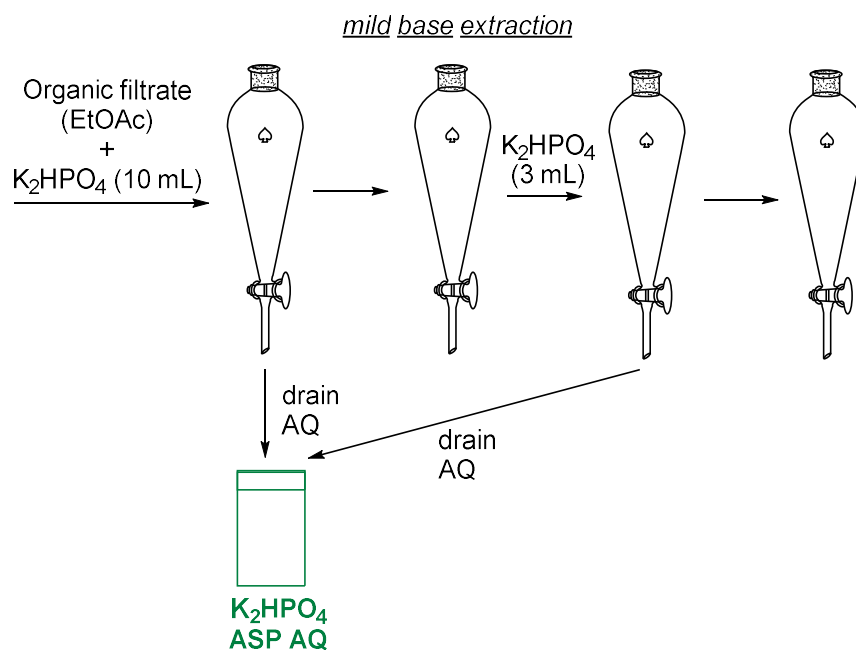
– crush, dissolve, & filter into separatory funnel

Fill in the boxes in this separation overview:



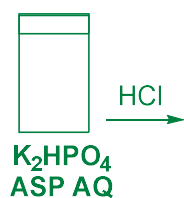
Indicate the layer(s) present in each separatory funnel diagram & label what components are in each layer

## 2a. Extraction with weak base

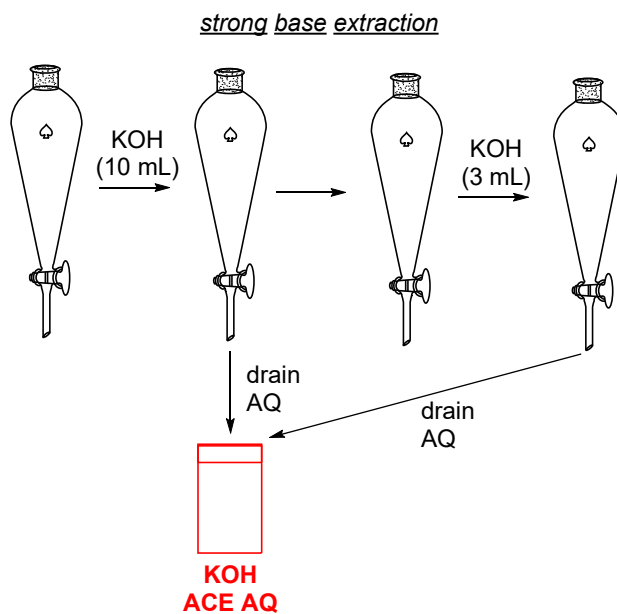


## 2d. Acidification & Isolation of Aspirin

\* include pH testing and filtration

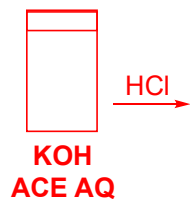


## 2b. Extraction with strong base



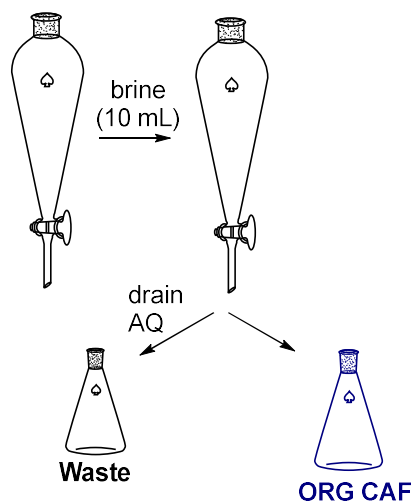
## 2e. Acidification of Acetaminophen

\* include pH testing and filtration

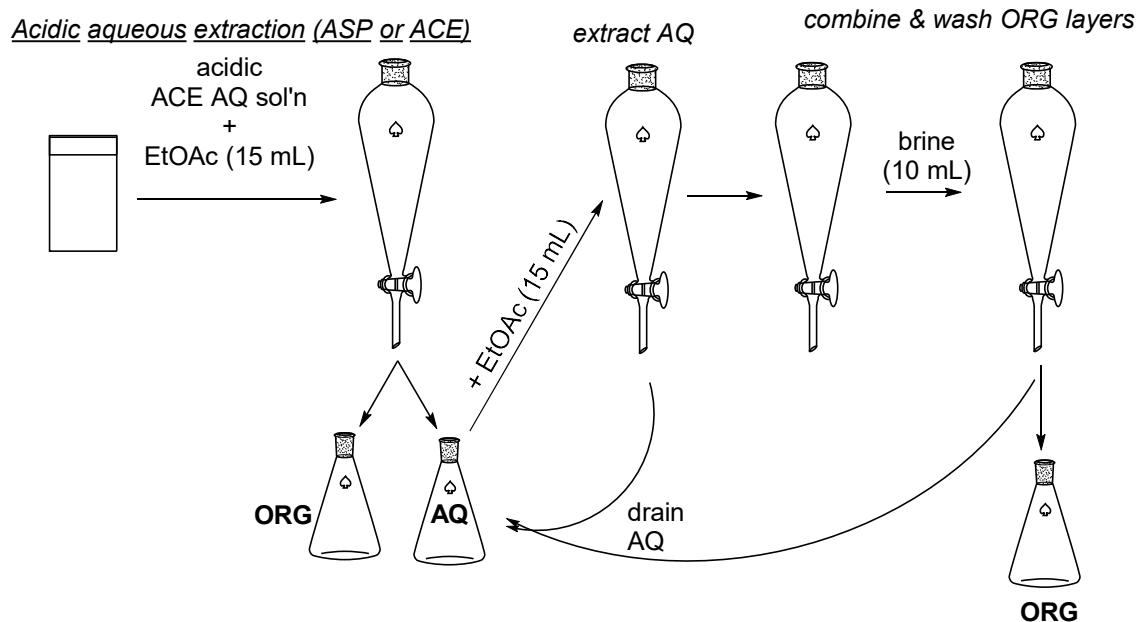


## 2c. Isolation of caffeine

\* include steps to dry, filter, and concentrate CAF



## 2e. Acidic extraction and isolation of acetaminophen



\* include steps to dry, filter, and concentrate ACE

**3. Analysis Procedure**

(a) **TLC** – spot & run one plate; rough sketch of UV lamp and developed plates with labels

(b) **IR** – prepare and obtain the spectrum of one sample; rough sketch of IR spectrum

**E. Data**

Mass of Excedrin tablet \_\_\_\_\_ g

Sketches of TLC plates and calculated  $R_f$  values for each spot:

Standards

(pure ACE, ASP, & CAF)

Extracts

TLC results for Excedrin column separation (Retention Factor,  $R_f$  values)

	<b>ASP standard</b>	<b>ASP extract</b>	<b>ACE standard</b>	<b>ACE extract</b>	<b>CAF standard</b>	<b>CAF extract</b>
<b>ASP</b>						
<b>ACE</b>						
<b>CAF</b>						

Mass recoveries after concentration:

ASP \_\_\_\_\_ g      ACE \_\_\_\_\_ g      CAF \_\_\_\_\_ g

Percent recoveries = (mass recovery) / (mass of tablet) x 100%

ASP \_\_\_\_\_ %      ACE \_\_\_\_\_ %      CAF \_\_\_\_\_ %

**IR Analysis** – see pre-lab questions for how to spectra from structure. Observe your IR spectrum and identify any signals within the expected range from the IR Tables. It is acceptable for a signal to be “not observed.”

**ASP**

Functional Group	Bond	Expected Wavenumber Range ( $\text{cm}^{-1}$ )	Observed Wavenumber ( $\text{cm}^{-1}$ )

**ACE**

Functional Group	Bond	Expected Wavenumber Range ( $\text{cm}^{-1}$ )	Observed Wavenumber ( $\text{cm}^{-1}$ )

**CAF**

Functional Group	Bond	Expected Wavenumber Range ( $\text{cm}^{-1}$ )	Observed Wavenumber ( $\text{cm}^{-1}$ )