

## Equipment & Chemical Preparation

### Synthesis of Sulfanilamide

In this two-week experiment, students will work in pairs to prepare sulfanilamide from acetanilide. Students will prepare acetaminobenzenesulfonamide the first week (2 steps in the reaction sequence) and prepare and analyze sulfanilamide the second week.

### Week 1

In the first step of the reaction, students will treat acetanilide with chlorosulfonic acid. The procedure requires that the acetanilide is placed in a clamped 125 ml Erlenmeyer flask. The chlorosulfonic acid is added dropwise (1-2ml at a time) using a burette that we will set up for the students in the hood. The flask is then fitted with a stopper that has a glass tube inserted into it and attached via tubing to a gas trap. (See Figure 4.4 in the Experiment Description for illustration). We will construct the traps for the students. The reaction mixture is heated in a water bath.

| Equipment  |
|--|
| 12 Hot Plate/Stirrers (one in each hood)**   |
| 24 Ring Stands with a ring and clamp**(2 in each hood)                                     |
| 12 Magnetic Stir Bars  |
| 12 125 ml Erlenmeyer Flasks**  |
| 12 burettes  |
| 12 water baths (must be glass)<br>(to fit around 125 ml Erlenmeyer flasks)                 |
| 12 gas traps fitted with stoppers and hoses  |
| 12 ice baths (plastic okay)<br>(to fit around 125 ml Erlenmeyer flasks with ice and water) |
| 3 Boxes of disposable pipets with bulbs  |
| 6 boxes of filter paper (5.5cm)  |
| 12 Buchner funnels (5.5cm)**   |
| 12 125 ml vacuum flasks**  |

| Chemicals                                   |
|---|
| Acetanilide (~1kg)                          |
| Chlorosulfonic acid (~2 liters)             |
| Concentrated Ammonium hydroxide (~2 liters) |

\*\*125 ml Erlenmeyer flasks should be in individual student drawers, but we should have 12 or so additional flasks on reserve for use. Buchner funnels, vacuum flasks, filter paper should be in student/common drawers. Hot plate/stirrers and ring stands should be in the hoods, but rings are in the storage drawers and should be set out in the hood. Double check to make sure these items are available.

### Instructions

- Set up twelve bins, one for each hood, containing each of the following items. Bins should be kept in the hood or a vented cabinet at all times.

| Equipment                       | Chemicals                                 |
|---------------------------------|---|
| 1 plastic ice bath              | 50g acetanilide                           |
| 1 glass water bath              | ~100ml conc ammonium hydroxide in bottles |
| 1 magnetic stir bar             | ~100 ml chlorosulfonic acid in bottles*** |
| 1 burette clamped to ring stand |   |
| 1 gas trap                      |   |

\*\*\* Chlorosulfonic acid will be dispensed each day into the burettes by the lab instructor. Fill each burette with 12.5 ml of chlorosulfonic acid. Burettes should be checked at the end of each lab period to be sure that no chlorosulfonic acid remains. Drain any excess chlorosulfonic acid back into the storage bottles at the end of each lab period.

- Check each hood for the following items:

|                     |
|---------------------|
| 1 Hot Plate/Stirrer |
| 2 Ring Stands       |
| 2 Clamps            |

- Set out three boxes of disposable pipets with bulbs on benches A, C and E.
- Check chemical bins throughout the week and refill as needed.

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### Week 2

| Equipment  |   |
|--|---|
| 12 magnetic stir bars  | Filter paper (150-200mm size, for gravity filtration) |
| 12 water baths (must be glass)<br>(to fit around 125 ml Erlenmeyer flasks)                 | 24 vials  |
| 12 ice baths (plastic okay)<br>(to fit around 125 ml Erlenmeyer flasks with ice and water) | 12 125 ml Erlenmeyer flasks**                         |
| 1 glass rods   | 6 sets of mini-presses for IR spectroscopic analysis  |
| 12 125 ml separatory funnels**   | 6 mortar and pestles                                  |
| 12 packs of pH paper   | 12 25ml volumetric flasks                             |
| 24 ring stands**   | 6 boxes melting point cover slips                     |
| 12 rings**   | 2-3 boxes weigh paper                                 |
| 6 jars TLC plates  | 6 UV lamps  |
| 6 iodine chambers  | 12 pencils  |
| 6 packs of microcapillary tubes  | 12 rulers   |

\*\*125 ml Erlenmeyer flasks and separatory funnels should be in individual student drawers, but we should have 12 or so additional flasks/funnels on reserve for use Hot plate/stirrers and ring stands should be in the hoods, but rings are in the storage drawers and should be set out in the hood. Double check to make sure these items are available.

| Chemicals                                   |                                      |
|---|--------------------------------------|
| 4M HCl (~2 liters)                          | ethanol (~2 liters)                  |
| 4M NaOH (~2 liters)                         | Hexane (~3 liters)                   |
| CH <sub>2</sub> Cl <sub>2</sub> (~5 liters) | Potassium bromide (IR grade)         |
| Magnesium sulfate (~10g)                    | Phosphate Buffer (pH ~7.4; 2 liters) |

### Instructions

- Set up twelve bins, one for each hood, containing each of the following items.

| Equipment  | Chemicals   |
|--|---|
| 1 plastic ice bath   | ~100ml of 4M HCl  |
| 1 glass water bath   | ~100ml of 4M NaOH (keep outside of bin, but in hood, separate from HCl) |
| 1 magnetic stir bar  | 1 vial MgSO <sub>4</sub> (~1g)  |
| 1 125ml separatory funnel                                    | ~200ml CH <sub>2</sub> Cl <sub>2</sub>                                  |
| 2 ring stands  | 1 vial benzoic acid (~500mg)  |
| 1 ring   |   |
| 1 glass funnel   |   |
| 1 pack pH paper  |   |
| 1 glass rod  |   |
| 5-6 sheets filter paper (150-200mm size, gravity filtration) |   |

- Set up six TLC bins, one for each bench, containing each of the following items.

| TLC Bins                     |                |
|------------------------------|----------------|
| 1 jar TLC plates (~25plates) | 1 UV lamp      |
| 1 iodine chamber             | 2 pencils      |
| 1 pack microcapillary pipets | 2 rulers       |
| ~100 ml ethanol              | ~200 ml hexane |

- Set out a pack of weigh paper next to each balance and a box of cover slips next to each melting point apparatus. Set KBr in oven for use in IR spectroscopic analysis
- Check bins throughout the week and refill as needed.