B.S. Sophomore Year – Spring Semester Lab

CEM 355 Organic Laboratory I

Description: Organic laboratory techniques. Distillation. Spectroscopy. Melting points. Recrystallization. Chromatography. Measuring physical properties.

Credit: 2 Credits (6 hours laboratory per week)

Prerequisite: Credit or concurrent enrollment in CEM 355

Experiments:

Students learn both normal scale and microscale techniques used in organic chemistry and perform a few synthetic experiments including a multi-step synthesis. They perform hands–on–experiments with ^{1}H NMR (300MHz) and FTIR spectroscopy. Informal lab reports are submitted for each.

1. **Distillation**

   (simple, fractional, and fractional with packed column distillation; microscale experiment using a Hickman Hinkle apparatus; compare distillation set-up efficiencies using theoretical plate calculations; cyclohexene synthesis from cyclohexanol; fractional distillation purification of cyclohexane).

2. **Thin Layer Chromatography (TLC)**

   (identification of a three-component mixture using standards)

3. **Column Chromatography**

   (alumina column separation of fluorene from fluorenone; fluorene purification using microscale sublimation)

4. **Extraction**

   (separation of a carboxylic acid from an ester by performing an acid–base extraction)

5. **Recrystallization**

   (purification of mixture (compound, 10% impurity (a different reagent), and traces of Sudan red))

6. **Diels Alder Reaction**

   (preparation of both of the starting materials for the Diels Alder reaction (benzoquinone and cyclopentadiene) followed by their immediate reaction; product is then exposed to UV light in a photoreactor to induce a 2+2 cycloaddition)

7. **Microscale Grignard Reaction**

   (microscale reaction of phenyl magnesium bromide (Grignard reagent) with methyl benzoate)

8. **Multistep Synthesis**

   (conversion of 2-chlorobenzoic acid into 2–chloro–5–nitrobenzamide in four steps)

9. **Isolation of Caffeine from One Tea Bag**

   (create their own experimental procedure to isolate caffeine from a tea bag)

10. **Azo Dye synthesis**

    (preparation of an X–(substituted phenylazo)–phenol by scale down of lab manual)

11. **Sandmeyer Reaction**

    (synthesis of 4-chlorotouene from 4–toluidine)

12. **Spectroscopy**

    (students are trained/checked out on the FTIR and NMR (300MHz) instruments; collect IR spectra for aspirin, recrystalization product, and their extracted caffeine; obtain ^{1}H NMR for the following experiments: Diels Alder, 2–chloro–5–nitrobenzamide, methyl 2–chloro–5–nitrobenzoate, 4–chlorotoluene, and caffeine extraction)