

Equipment and Chemical Preparation

Diels- Alder Reaction

In this two-week experiment, students will work in pairs to the Diels-Alder adduct of α -terpinene and N-phenyl maleimide. The procedure involves a relatively simple reaction set-up involving refluxing the starting materials in ethyl acetate and filtering the product that precipitates from the reaction mixture. The product will be analyzed by TLC, melting point and IR spectroscopy during the second week. NMR data of the targeted product will be provided for analysis. The procedure is taken from McDaniel, K.F.; Weekly, R.M. "The Diels-Alder Reaction of 2,4-hexadien-1-ol with Maleic Anhydride", *J. Chem. Edu.* 74 (12) 1465-1467, 1997.

Week 1

Equipment
24 16mm test tubes
24 Hot Plate/Stirrers (one in each hood)**
24 Clamps
24 Wooden boiling sticks
24 250ml beakers
6 boxes of filter paper (5.5cm)
12 Buchner funnels (5.5cm)**
12 125 ml vacuum flasks**

Chemicals
2,4-hexadien-1-ol
Maleic anhydride
Ethyl acetate
Toluene

**125 ml Erlenmeyer flasks should be in individual student drawers. 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.

Instructions

1. Set up six equipment and six chemical bins, one for each bench containing each of the following items. Use the exact sizes listed

Equipment Bins	Chemical Bins
4 16 mm test tubes	1 10g bottle of 2,4 hexadien-1-ol
4 250ml beakers	200 ml toluene
4 boiling sticks	1 10g bottle of maleic anhydride
	Solution of 2,4 hexadien-1-ol for TLC
	Solution of maleic anhydride for TLC

2. Check each hood for the following items:

2 Hot Plate/Stirrers
2 Ring Stands
2 Clamps

3. Check chemical bins throughout the week and refill as needed.

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Week 2

Equipment
6 boxes melting point cover slips or tubes
2-3 boxes weigh paper
6 jars TLC plates
6 iodine chambers
6 packs each of microcapillary tubes (6 open for TLC; 6 closed-end for melting point)
6 UV lamps
12 pencils
12 rulers

Chemicals
2-3 liters Ethyl acetate

Instructions

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

TLC Bins
1 jar TLC plates (~25plates)
1 iodine chamber
1 pack microcapillary pipets
1 UV lamp
2 pencils
2 rulers
~100 ml ethyl acetate

3. Set out a pack of weigh paper next to each balance and a box of cover slips and/or closed end capillaries next to each melting point apparatus.
4. Check bins throughout the week and refill as needed.