Experiment 15
The Wittig Reaction

Flow Chart

Please ignore step one under "crude stilbenes"

50 mL round bottom equipped with condenser and magnetic stirring

1. Add 5 mmole benzaldehyde
2. Add 5 mmole benzyltriphenyl phosphonium chloride
3. Add 5 mL CH₂Cl₂
4. Vigorously stir
5. Add 3.3 mL of 10 M NaOH through neck of flask
6. Establish gentle reflux for 30 minutes with vigorous stirring
7. Cool in ice bath
8. Add to 125 mL separatory funnel
9. Add ca. 3 mL H₂O

aqueous

organic

discard down hood drain

receiver

residue

CH₂Cl₂

crude stilbenes

1. Dry over Na₂SO₄
2. Filter into 50 mL round bottomed flask
3. gently rotavap (no heat)

filtrate

residue

1. separate out ~0.3 grams for chromatographic analysis & purification
2. Dissolve the remaining material in ca. 3.5-4 mL of warm absolute ethanol
3. Cool
4. vacuum filter

discard in non halogenated waste

filtrate

residue

1. recrystallize from ca. 3 mL of ethanol /water mixture
2. vacuum filter

discard in nonhalogenated waste

weigh, m.p. analysis use as TLC std.