

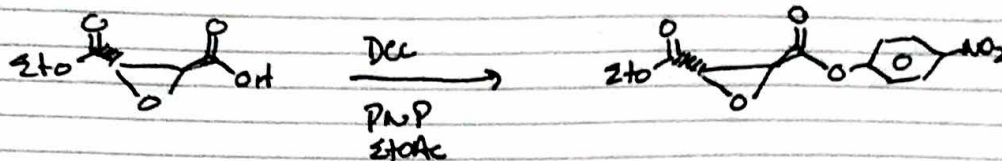
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7.24.10



8.99 mmol monosster + 8.99 mmol PNP (1.25g)

→ 250ml RB flask 124.09g Dry wt

Solid + oil only: 3x vacuum → Ar (g) cycles

Flush syringe 5x Ar (g)

8ml EtOAc (Anhy.) Add against Ar (g) back pressure (success)

Fully dissolved + chilling under Ar (g)

1.86g (9.0 mmol) DCC

→ 25ml RB flask

Solid only: 3x vacuum → Ar (g) cycles

Flush syringe 5x Ar (g)

Add 4ml EtOAc An (Dry) (success) against Ar (g) back pressure

DCC dissolved readily, w/out stirring

Flush syringe 5x Ar (g)

Fill w/ DCC/EtOAc solution

Dropwise addition over ~15 minutes against Ar (g) back pressure

→ white ppt formed after ~2ml

Addition finished ~1:15 pm → ice until 4 pm, stir until 4:15 pm

(3hrs on ice (4-6°C)), 1hr @ RT

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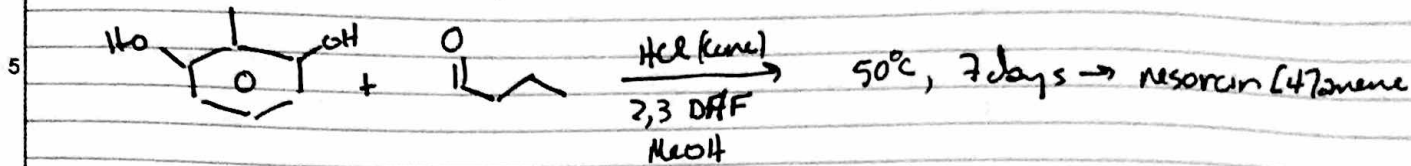
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9.09.10

Phosphonate Cavitation



10

2-Methylresorcinol : 9.52g, 60ml MeOH

→ cooled in ice bath to ~0°C (15 min)

1.45ml (19.12mmol) 2,3 DHP (1ml + 0.45 ml)

5.05ml (57.4mmol) butyraldehyde (4x 1ml, 2x .525 ml)

14.40ml conc. HCl dropwise via dropping flask (~4s between drops)

→ to ice cooled solution

After addition :

oil bath → 50°C set temp

Reflux condensor, slow flow of cool H₂O

150 rpm stir

! slight yellow color, mostly clear

→ bright yellow color by ~15 min of heat, still clear

→ slowly darkening yellow color

! temp settling around 48-55°C, check in 1 hr after equilibration

2pm : settled to 49°-51° temp variation

color : dark orange - rust, still clear sol'n.

3pm : Bright orange ppt forming

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