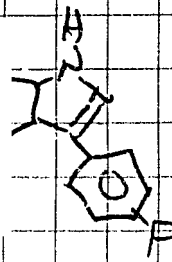


- 1.
- 2.
3.  $\text{NH}_2\text{NH}_2$  HOAc



6/8/00

Name  
mm  
used  
ml  
ml eq.

H<sub>2</sub>O

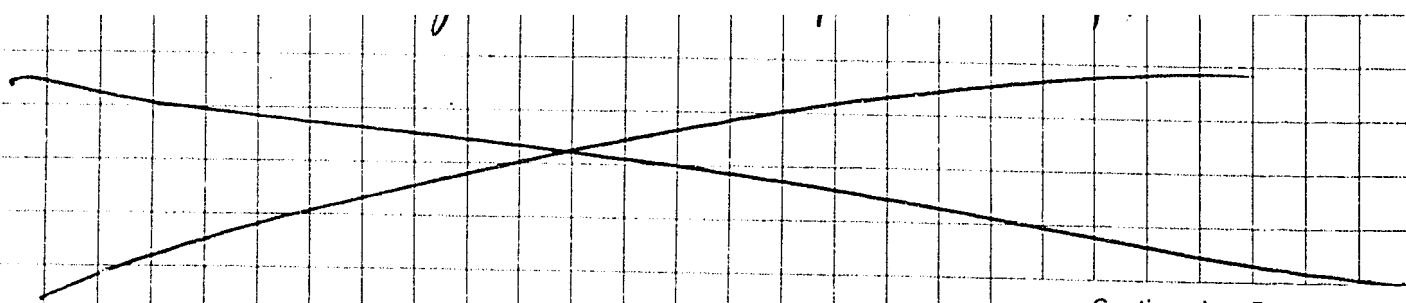
30%  
30 ul

EtOH

40 ul

6/9 Took up in DMF acetal (100 ul) and heated to 90°C for 6 hrs. LC/MS showed no sm. Rotovap'd to an oil then added  $\text{NH}_2\text{NH}_2$  (2 ul) in HOAc (100 ul). let stir overnight at rt

Add H<sub>2</sub>O and filter. Purified on prep HPLC - After (30-90% AcN/H<sub>2</sub>O). After 2 rounds to prep



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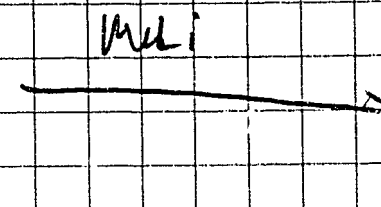
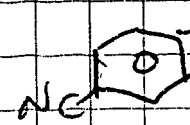
Read and Understood By

Signature

Date

✓ Signed

Date



Name  
mm  
used  
ml eq.

Meli

1.0M

1.0

1.5

THF

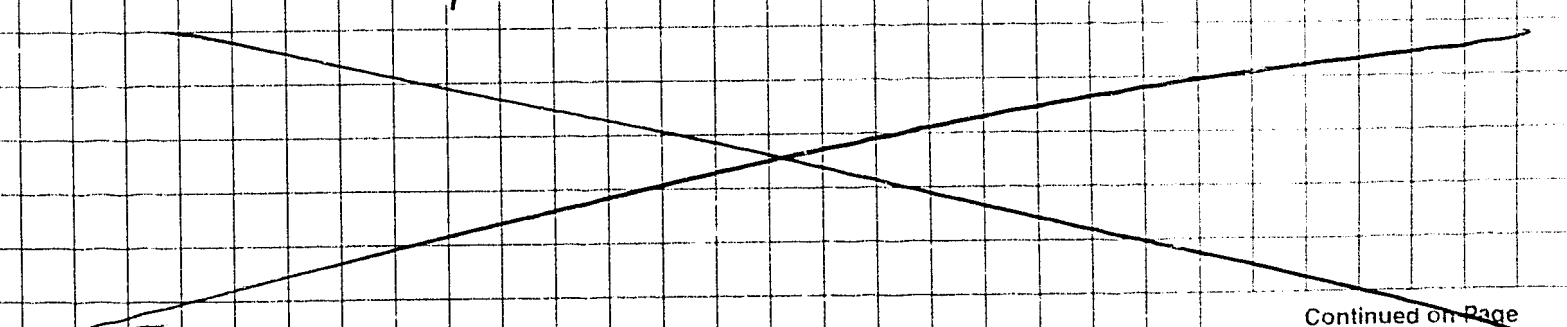
10 ul

6/22 To a soln of the nitrile in THF at -78°C, stir ~ 3 hrs. when it was quenched w/ H<sub>2</sub>O and extracted w/ EtOH. Combined EtOH layers to

6/23 Chromatographed on SiO<sub>2</sub> w/ 20% EtOAc/hex. to

6/26 Took up solid in MeOH (30 ul) and 12N HCl

$\text{Na}_2\text{CO}_3$  filtered w/ dried solid to recover



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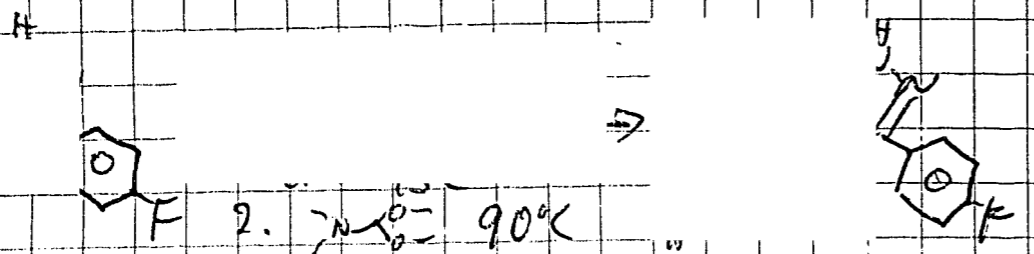
Signature

Signed

Date

✓ Signed

Date



2.  $\text{H}_2\text{O}_2$  90°C
3.  $\text{NH}_4\text{NH}_2$ ,  $\text{H}_2\text{O}_2$  v.t.

name Nihil NaOH

MW 237 6N  
15 ml

ml 42 ml  
ml 1

11/15 To a solution of the nihil in EtOH / NaOH was added the  $\text{H}_2\text{O}_2$ . Stirred at 45°C for ~3 hrs. TLC showed no sm. Added 3 more to pH < 1. Then removed off EtOH.

Submit 1.8 g to SPC

11/17

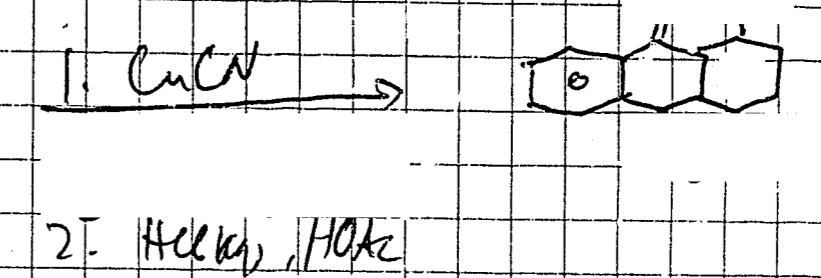
400 ml H<sub>2</sub>O spiked w/ 10 ml  $\text{NH}_4\text{OH}$ . Stirred at rt. Added to H<sub>2</sub>O & filtered. Chromat. w/ 5-15%  $\text{MeOH}$ .

Submit 1.9 g to SPC. lots of >93% pure stuff

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Signed \_\_\_\_\_ Date \_\_\_\_\_ Signed \_\_\_\_\_ Date \_\_\_\_\_



name NMP

MW 89.5 70 mg 2nd  
0.79 1

11/17 A solution of the, chloride, CuCl in NMP was heated

11/20 Added XS CuCl & heated again to 185°C. rxn. never went to complete.

11/21 removed 550 mg. took up in conc HCl (50 ml) & H<sub>2</sub>O (50 ml). heated

11/28 Potaged to a solid.  $\text{H}_2\text{O}$

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Signed \_\_\_\_\_ Date \_\_\_\_\_ Signed \_\_\_\_\_ Date \_\_\_\_\_