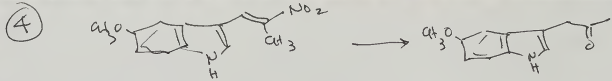
(4)

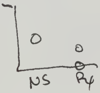


{ 8 ml HOAc
{ 2 ml H_2O } Δ on S.B. add

1.7 g electrolytic iron dust, when hot, add

0.5 g κ_3O-NS a glob at a time, over 2 minutes

TLC
CH_2Cl_2
hexane



10 min all NS gone - new spots at origin & in between

30 min off (had to wait for TLC)

Into 100 ml H_2O and CH_2Cl_2 - 2 phases and secondary inbetween - filter through paper - separate, 2×50 ml CH_2Cl_2 more - wash c̄ sat K_2CO_3 - flash → 0.28 g film.

KR at 0.1 mm. 130°
160
180° (vac to 0.08)
215-230° over → 0.24 g oil - off white

IR c=O at 1710 cm^{-1} mass spec OK.

stand a couple of months → deep brown color.

Repeat:

20 mL HOAc
5 mL H_2O } Δ SB ~ 60°. add

4.55 g Fe electrolytic - up to ~ 70° - bubbling. add

1.24 g nitrostyrene - over 2 minutes - color discharges

5 minutes OFF. - into 250 H_2O, 50 ml CH_2Cl_2 NOW

Filter through paper - wash c̄ CH_2Cl_2. filtrate - separate - xtnt aq. c̄ 2×50 CH_2Cl_2 - combine, flash → 1.17 g amber oil - slight acetic acid smell

Stand 3 days in cold lab → xtals! mp - not yet by 95° - polymer?

8:4

TLC (CH_2Cl_2 2: hexane) OK however.