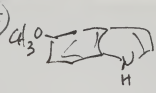
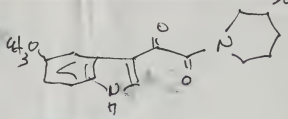


914



COCOCl
 →
 piperidine



A solution of 125 g 5-methoxyindole in 15 ml TBME was stirred, and a solution of 1.1 g oxalyl chloride in 15 ml TBME added over ^{20 min} ~~10 min~~ dropwise - 10 min tomato-red xtabs start to appear, ~~stir~~ stir another 10 min, filter. - wash with a little TBME - loose "hard" tomato xtabs - add a bit at a time to 2 ml piperidine - stirred - exothermic & fume, of course - end up adding ~1 ml more piperidine to get everything wet & all tomato gone. to RT - + 80 ml 1N HCl - quirky - stir for quite a while - dig out gunk - get most of the water off by cleaning dabbing - grind ~~under~~ under MeOH ~ 2 ml? solids - filter →

← aqueous, filter
 → 0.01g pink solids out

0.82g cream xtabs
 mp 153-158 → 0.65g totally dry
 nextst f. 2g MeOH
 in hot -
 out (seed) ▽
 → 0.37 pale
 mp cream
 107-109 fine xtabs

Page 115 continued - filter → wet cream colored solids - dry in SP ^{100°} → 1.13g cream colored powder
 Sample for MP 115B crude. mp 180-200 (195 area)

Recrystallizing from 15 ml boiling MeOH (decant from a wee bit of insoluble) clear hot - ▽ RT → xtabs - filter → white solids, xtabs - amber ML
 → air dry. pale cream .75 wet c MeOH.
 .65 dry

insoluble - wash acetone - air dry - white. mp 211-212 ← IR's identical → mp 210-211

acetone / insoluble