

0.95 g ^{5mm} S-MeOT, free base TCI - beautiful - looking stuff into 10 ml IPA - Δ in SB to dissolve & RT. add

- + 2.8 ml CCN(C)C ($2.61 = 15mm \cdot d. 742 MW 129$)
- + 1.2 ml (used 1.3) EtBr ($1.13 = 15mm \cdot d. 1.46 MW 109$)

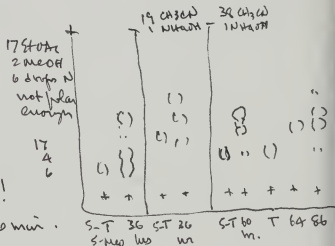
Stir RT 3PM Wed.
36 hrs - all three there still

duplicate + 2.8 ml amine
+ 1.2 ml EtBr

another 34 hrs.
36 hrs.

strip → 94A 3.4g same dab. - 2 phases!

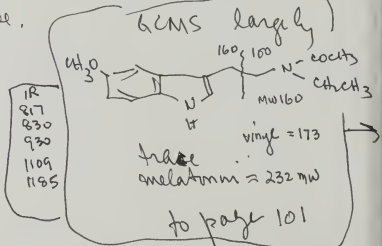
add 1.6g Ac₂O (.5 MeO) onto SB. ~20 min.
into beaker - + 3 ml NMeOH.
+ 100 ml 0.5 N H₂SO₄ (H⁺OK) xrt + 3 x 50 ml CH₂Cl₂



pool CH₂Cl₂ x 100 ml H₂SO₄
3 x 50 ml CH₂Cl₂

pool strip ^{0.44g} **94B** - xrtalizes! (brownish)

0.60 N MeOH (cloudy) flash
xrt 3 x 15 ml CH₂Cl₂ 0.80 g sharp
0.5mm 80° no
110° no
160° no
190-200° yes. \uparrow 0.45g white oil smell.



all into 2.5 ml IPA RT
+ cover vial (6 with enough
dropwise & too much)
sprout starts start. slowly
to final vol 20 ml & another

→ 0.52 beautiful white xtals. call 0.50g 34%