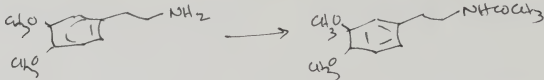


(168)



11 p 138

9.0 g fresh - clear amine from TCI add
 15 ml pyridine add
 6 ml Ac₂O - quite hot - let cool to RT over 1 hr
 + 150 ml H₂O - some HCl → red (a few mL)
 xft = 3 x 75 ml CCl₄

prod - wash 1 time = 50 ml 5% NaOH. flash.

11.45g ambient
 to IR pot = CCl₄ 10.58 g solids. ← I don't trust the balance

0.15mm RT IR.

200 μ. 100° melting.

150 μ. 140° nothing.

150° up volts.

150 μ 165° over - white beautiful.

175° 1/2 over - cool = CCl₄

180° 10% left - 7 = CCl₄

185° done - receiver xbars

100 μ.

QuickKals finds

mp (p. 141) 97-98°C ex EtOAc

MS (p. 139) 164 (100%, styrene) 150 (49%)

149 (23%) 223 7%
 parent

9.73 in receiver - don't trust balance

7.63 physically removed.

pale yellow solids -

9.50 closer, almost white.
 to right weight.

0.50 g sawe
 9.00 g to p. 109