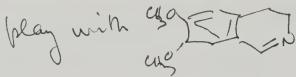
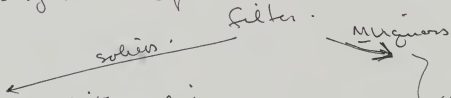


(172)



just under 7g of distilled product - colorless when distilled - 2 days later - noticeably descolored. Into 20 ml IPA. - add conc. HCl dropwise - checking acidity in pH paper. - never really red - ~40 drops added - xbars start (yellow). stir - add - 10 ml ether - heavy mass of xbars - (yellowish) -



good quantity of solids. warm in 15 ml 2/1 IPA/ether. IT IS DISSOLVING!!!

flask. → several ml heavy oil.

Suck dry (hygroscopic? no). onto petry dish ~ > 3g (172A) for GCMS 2.22g

oil into ~100 ml CH2Cl2 soln!

xbar 4 x 75 ml diel HCl

→ CH2Cl2 flask → 50mg of sample (172B)

make basic in 25% N2O4 xbar 3 x 50 CH2Cl2 - flush

for GCMS - best out. No eventually with see page

oil fraction 4.47g (172C)

(174. 175)

MS 191 (100%) 176 (32%) 190 (32%) 133 (25%)
parent p-1

clean up - add neat 1 ml Ac2O (hot, becomes thick) + 20 ml CH2Cl2 - dissolve total ~150 ml CH2Cl2 xbar 3 x 50 ml in HCl - wash 1 x CH2Cl2 - 84.2%

SM20 xbar 3 x 50 CH2Cl2 flush → 2.57 amber oil (172D)