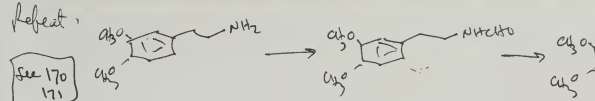
Repeat.



See 170
171

10 g DMPEA, saturated ĉ carbonate - the ancient bottle of Aldrich chem. stuff - all that would drain - carbonate now solid in bottle.

25 g isobutyl formate → cloudy - Δ SB 1 hr ⇒ brown soln. - add to 200 ml H₂O, @ → pH ≤ 1. extract 3 × 75 ml CH_2Cl_2 - pool - wash ĉ 5% NaOH - flask → 10.85 g crude oil - distil

KR 150-160°/120 μ, → 7.52 g viscous oil pale amber. into 45 mL $POCl_3$ - spill - nasty - onto SB 1 hr (1.5 hrs actually.) - off - cool ON. Into 400 ml cold water - exotherm of course - ∇ ĉ H₂O - xtxt ĉ 2 × 50 ml CH_2Cl_2 OH^- ĉ 25% NaOH to pH ~13 - ∇ → solids, H₂O to 1300 ml to dissolve - xtxt in ½ batches ĉ 3 × 50 ml CH_2Cl_2 each - pool. wash ĉ H₂O - flask → 4.24 g viscous brown oil. KR.

neutral

200 μ 130-140° 2.73 g. save ~0.2 g to see how the oil discolors colors.
2.50 g to page 200

neutral ↔ → this all from memory - lost the paper ĉ the notes and weights. flash of CH_2Cl_2 → a lot of material - 2-3 g that tends to go solid. TLC a bit checking against reference sample of formamide. solvent 80/20 hexane/EtOAc. Cinnic ~0.2 Rf. this stuff orig m. Could the salt of DMHH-2 be extractable into CH_2Cl_2!? Partition in OH^- H₂O & CH_2Cl_2 - CH_2Cl_2 to dryness ~1.5 g? to HR. So, so vacuum (~200 μ?) and push to ~200°. Some 60 mg came over and the pot was left with a dark residue ≅ too much — with some fluidity. OUT.

2.16