



1.5 g DMHH-2 · HCl (from p 172A) - into
50 ml H₂O

0.5 g NaBH₄ - + 1N HCl

With good stirring add hydride a bit at a time, keeping the Rx mix acidic = squirts of 1N HCl. Wash \bar{c} 1x30 ml CCl₄, extract \bar{c} 2x40 ml CCl₄. Make basic \bar{c} 5% NaOH, extract \bar{c} 2x40 ml CCl₄ - flash on R. evap \rightarrow residue that crystallizes - Distill at KR 110-130° 100 μ - all sublimes over as white solids. Wt (in flask) 1.12 g

Scrape out. 1.00 scrapped out - not sol. IPA, Sol. H₂O - it must be the carbonate - into ~ 100 ml H₂O some soluble - add 5% base \rightarrow pH blue - xht 3x40 ml CCl₄ - flash \rightarrow

~~Scrape out~~
 .86g oil that sets to white xtal - dissolve in 2 ml hot IPA - into beaker - volume \bar{c} 2 ml IPA - clear soln - add 10 too little 13 too much HCl (solids at 3!) + a bit of ether - stand - filter - wash \bar{c} ether air dry \rightarrow beautiful white crystals of DMHH-4 · HCl 0.92 (207A)