

206



1205

2.6g DMMH-2 (page 169C) MW ²⁰⁵/₂₀₇ ~ 12 mM into
 100 ml H₂O (IT IS WATER SOLUBLE!!!), acidify with
 1 N HCl (~ 12 ml → red pH paper)
 1g NaBH₄. add a bit at a time (good stirring) + N HCl
 as needed to keep the solution acidic. No exotherm.
 wash \bar{c} 50 ml CH₂Cl₂, basic \bar{c} 5% NaOH - exhaust 2x
 50 ml CH₂Cl₂ - strip on R.E. → "1.17g" crude - obviously
 I misread the balance, see below - KR 110-120°/40 μg →
 2.13g free base - white oil - ON start of xbalize

picking up CO₂? GC/MS conc A31A (SM21)
 save a bit of free dilute A33A
base solids - contains CO₂ (206A)

Partition between CH₂Cl₂
 and dil NaOH - 3 x 40 -

Wash → 2.24 white oil - dissolve in 8 ml IPA - 30-32 drops H₂O →
 pH red. no xtals - add ether to turbid - no xtals - forget it out
 externally - seed - sorta takes - I feel I am fighting two xtals?
 (hydrate) - more ether → mush of solids - filter - wash \bar{c} ether -
 air dry → 2.70 g of spectacular white solids - ether slow to evap. 10 H₂O?
~~2.06B~~ (206B) acetone soluble!! but the carbonate is not!

