



Base 212

0.5g harveline $\cdot \text{Hce}$ in
 8 ml abs EtOH
 8 ml conc. HCl

to this, add

0.25 ml of 1.42 conc. HNO_3 in
 7 ml abs EtOH

Δ S.B. - almost hot - bubble bubble bubble! -
 30 seconds - off - ∇ \bar{c} cold water \rightarrow xtals stand
 filter - wash a bit of EtOH (acetone would have been better) 2 hrs

air-dry on \rightarrow 0.31 g ~~of~~ pale yellow xtals. (67% Y)

IR \equiv absolutely \bar{c} aldrich reference spectrum.

save a small amount

133A

mp 267°

rest - to free base for GC cleanlines
 verify IR,

dissolve in 3.4 ml RT H₂O. 3.1 would have been OK. add ~ 6-8 drops conc. NH_4OH \rightarrow
 globs of almost white solids
 grind, stir, breakup, filter

\rightarrow let air dry, no 100° dry

small sample - without purification
 for IR
 for GCMS

133B

0.22g
 off-white
 solids

mp 253° dec?