

4.0g gold-orange xtals f. p200, into 100 ml H₂O
 squirt of N HCl - not quite clear, but very yellow, solution
 1g NaBH₄ added a bit at a time, with occasional
 squirts of N HCl. Color dissipated with the first
 bit of Bor₃ - add 5%, then 25% NaOH to pH > 10 - heavy
 gel of maybe sodium borate? Dilute to ~ 600 ml
 H₂O which pretty much clears it
 xht is 3 x 50 ml concs - absolute colorless! pool, flask
 → 2.46g crude white oil, xtals in flask - KR →
 !! ↓ 276g white oil that distills at 120-130°C at 300 μ
 reweigh when dry out of the receiver. Beautiful GCMS.
 SM-20-A98A

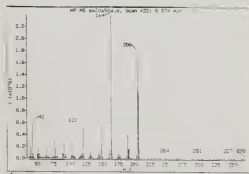
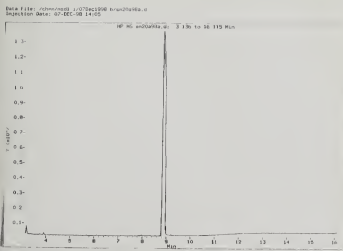
Picked out the rest

0.1g white xtal -
 I am pretty sure I
 started pick it out
 when the big Sunday
 occurred
 to GCMS as (probably)

← 0.10g 201 certainly
 GCMS - if same as
 this, combine &
 hold as reference

1.94g → 1.84g to
 dissolve in
 9 ml IPA.
 Δ needed. ▽.
 + conc HCl (this ~ 15
 drops - used ~ 25, small
 drops?) start of xtals
 + ~ 5 ml ether →

heavy white mass - filter
 wash 2x ether + IPA, then other
 white air-stable solids!
 xhtal ethyl ac - fair →
 xhtal 9 to Ac - bad
 xhtal MeOH, too sup.
 xhtal IPA. fine →
 crude wup →



Data File: chem0001_1070121000_0000000_0
 Injection Date: 07-EE-08 14:05

MS MS Scan 206. Scan 4321 0 274 200