



101

0.44 g awfully good crude N-St. melatonin
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grind under a few ml MeOH → almost white solids - Refill
 a few milligrams from MeOH → white xtabs.

some for mp
 rest for IR
 trace for TLC ←

0.30 g amide - 6 ml 5% NaOH - into SB - 2 hrs - still
 white solids - H₂O evaporating - + 3 ml 25% NaOH -
 A another hr. Still solids - no change - add ~ 2 ml
 IPA, lower - 2 hrs - going into sol. a couple of globs
 remain, keep at 100°.

overnight - into 100 ml H₂O (is basic
 of course) xht 3x40 ml CH₂Cl₂ - pool -
 xht CH₂Cl₂ = 3x50 ml 1N H₂SO₄

0.2g
 CH₂Cl₂

0.1g

work in CH₂Cl₂
 OH⁻ = 25% NaOH
 xht = 3x50 ml CH₂Cl₂
 flash
 → 0.05 g

IR. carbonyl:
 1608 cm⁻¹
 Fingerprint
 790, 829, 929, 1031, 1068
 1108, 1182, 1199.

flash
 → 0.24 g residue
 film goes to solids
 amide?
 when dry 0.19 g
 almost white solids