

Repeat 115 - Surfer → dark product + coc in Et_2O - H₂O ↑

96 ← 2.13 deak
dry. wet

119

Repeat 115 - we 1.25 mols
1.05 cocococ (inadequate) (fresh) 15 MTBE
2.0 ml pyrrolidine 15 MTBE

differences, fresh cocococ, from ACROS.

addn - 20 min → light tomato - stir 15 min -
filter - wash (by suspension) in TBME (RT).
Air dry several min. to rather loose xtds.

Add (scatter, not bunch-wise) to 2.0 ml
pyrrolidine. The few dark spots rubbed in
w/ spatula → dark cream viscous oil. Add
some 1N H₂O → off-white solids - grind up
chunks, filter. wash w/ H₂O → 3.42 g wet solids

grind under 3 ml acetone to almost white paste - it seems
to firm as I work with it - Filter - wash in very small
amt. acetone. → 1.42 wet. ON air dry 0.97 g

0.94 g
xtd
Removals

0.94 g into 12 ml boiling Et₂O - clear, ⚡ to RT
let stand ~ 1 hr. filter, wash sparingly w/ Et₂O → 0.72 g wet
0.69 g dry

mp 212-213°.

0.58d wet

p120

3.42
wet

3 ml acetone

1.42
(wet)

0.97 air dry.