

Dry some CHCl_2 by boiling 200 ml \rightarrow 100. ml on SB. cover immediately.

In 2 250 RB flasks - w/ stirrer - add

10 ml dry CHCl_2 - add

1 g indole - stir \rightarrow solution (under Ar) - cool to 0° \bar{c} external ice bath, add

12.5 ml 1 M SnCl_2 (in CHCl_2) \bar{c} over \approx 2 minutes, dropwise - turns a slightly cloudy grey color. Then add:

1.0 g neat Cl_2C(=O)OCH_3 slowly (stir, still at 0°) turns a tomato color.

stir 15 min 0° - then

1 hr RT. Pour into ice water, make basic \bar{c} ag. NaOH \rightarrow orange CHCl_3 phase & colorless ag.

Separate - extract ag. \bar{c} 25 ml CHCl_3 - combine wash \bar{c} \approx 1% NaOH. Separate & flash.

pale yellow phase \rightarrow 1.78 g crude viscous oil.

Into \approx 10 ml MeOH - let stand on \rightarrow

stabs down

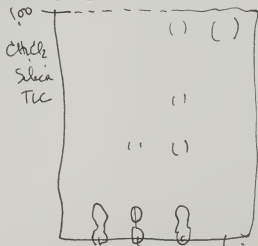
ML up

flash - also film on top out

grind up \rightarrow 0.46 brownish-brown solids

into big fatri dish cover on \rightarrow w/ oil & stabs w/ \bar{c} 2 x 5 ml hexanes \rightarrow indole smells

brown solids ~~0.23g~~ sl. gummy solids



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