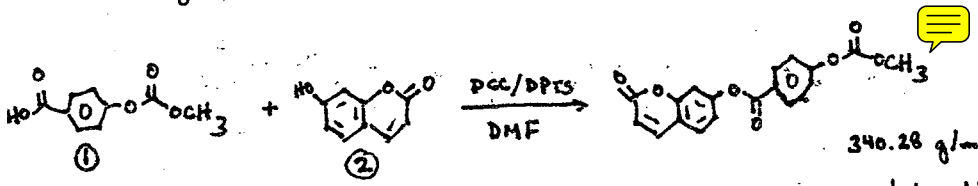
Work continued from Page



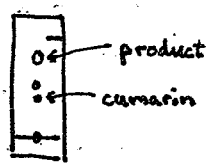

340.28 g/mol

expected yield ⇒ 1.46 g

SSB-10318

| | ① | ② | DCC | DPTS | DMF |
|---|---|---|---|---|---|
| mw | 196.16 | 162.145 | 206.33 | 312.5 | — |
| mass | 0.84 g | 0.69 | 1.32 | 2.00 | — |
| mmol | 4.28 | 4.28 | 6.42 | 6.42 | — |
| vol | — | — | — | — | ~30 mL |
| eq. | 1 | 1 | 1.5 | 1.5 | — |

3/24 oven-dried 2-neck 100-mL RB and stirbar charged w/ ①, ②, DCC, and DPTS
DMF added to dissolve
mixture stirred under Ar overnight @ RT




3/25 next morning TLC in 15% EtOAc/DCM shows nearly no starting material
rxn quenched w/ 200 mL DI water
off-white ppt. forms
ppt. filtered and left to dry on bench

3/31 off-white solid dissolved in DCM and preadsorbed onto silica
loaded onto column (15" x 12") and eluted w/ 10% EtOAc/DCM
white solid collected and placed under high vac to remove remaining solvent
NMR in DMSO shows product w/ trace impurities, impurities may be magnified due to slight insolubility of compound in DMSO file ⇒ DLII-202 (name will need changing later)
0.90 g collected ⇒ 61.6% yield

4/1 material preadsorbed and run through a silica plug eluted w/ 5% EtOAc/DCM to 10% EtOAc/DCM
white powder collected
NMR in DMSO ⇒ DLII-02 (040208) ⇒ pure



Work continued to Page

SIGNATURE

DATE 3/24/08

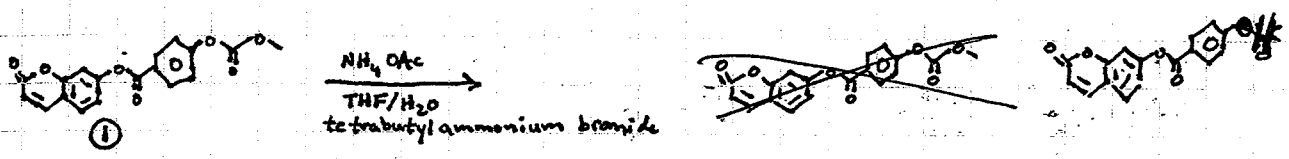
DISCLOSED TO AND UNDERSTOOD BY

DATE 7/22/09

WITNESS

DATE

Work continued from Page




| | ① | $NH_4OAc$ | tetrabutylammonium bromide | $THF/H_2O$ ⇒ 3:1 ratio |
|---|---|---|---|---|
| mw | 340.28 | 77.08 | 322.38 | — |
| mass | ~~.80~~ 2.35 g | 1.45 | ~~—~~ .30 | — |
| mmol | ~~.00~~ | 18.8 | ~~0.076~~ .94 | — |
| vol | — | — | — | 80 ml ⇒ 60 ml THF, 20 ml $H_2O$ |
| eq. | 1 | 8 | ~~.10~~ .40 | — |

General Procedure from SJB-1061:

stir ① in $THF/H_2O$ mixture w/ $NH_4OAc$

add TBABr to distribute layers

Extraction performed w/ EtOAc

4/2 500-mL 1-neck RB charged w/ ① and dissolved w/ $THF/H_2O$ soln.

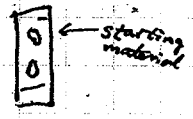
mixture stirred as $NH_4OAc$ and TBABr added

Rxn stirred @ RT overnight

mixture settles into two layers when stirring stopped

soln is milky white in color

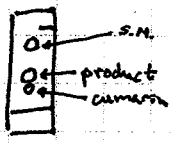
TLC after 3 hrs shows possible product formation (eluted in 5% EtOAc/DCM)




4/3 TLC the next morning shows two spots w/ equal intensity

previous procedures in ~~SBS~~ SJB perform a work up at this time, however rxn will be allowed to procede to see if the reaction progresses any further

TLC shows cumarin starting to form, can be seen under both long and short wave




rxn mixture extracted w/ 4x50 ml EtOAc

some compound still ppt. in aqueous layer, extracted 3x50 mL DCM

organic layers washed 3x50 ml DI $H_2O$ and ~~dried over~~ combined.

organic layers dried over $Na_2SO_4$ and condensed

4/4 material preadsorbed onto silica and eluted on silica column w/ 6% EtOAc/DCM to 8% EtOAc/DCM

starting material, pure product, and product w/ cumarin fractions collected



Work continued to Page

SIGNATURE

DATE 4/2/08

DISCLOSED TO AND UNDERSTOOD BY

DATE 7/24/09

WITNESS

DATE