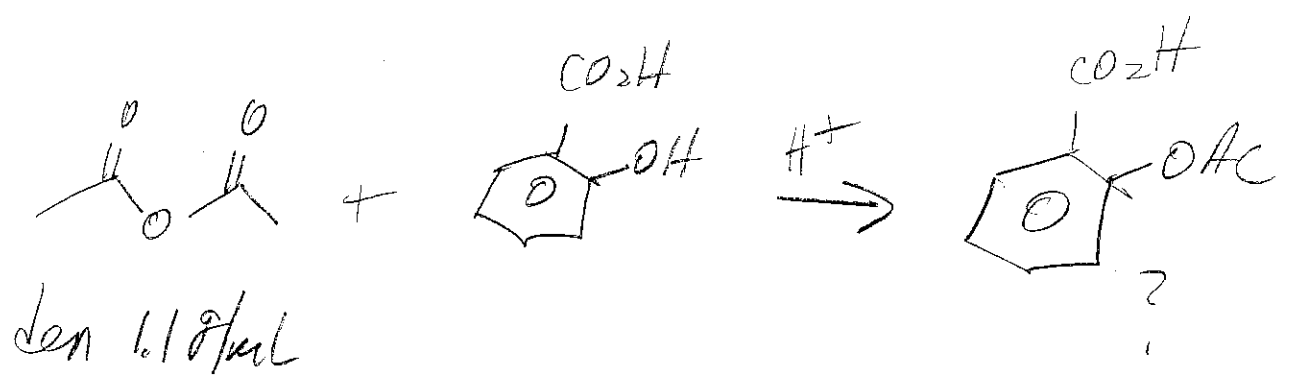


1/11/06

Reaction of Acetic Anhydride + Salicylic Acid



2.2g (21 mmols) (8.0 mmols)
 2 mL Ac_2O added slowly to 1.1 g salicylic acid in 50 mL Erlenmeyer. Solid dissolved after some swirling. 1 dr 85% H_3PO_4 the added. Flask... was heated for 15 mins in 80-90°C water bath. Flask was removed from bath + after cooling slightly, water (~20 dr) slowly added (to destroy Ac_2O).

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Ac_2O + Salicylic acid (cont'd)

48

10 mL H_2O then added. After cooling in ice bath large mass of needle-like white xtls formed.

Xtls isolated by suction filtration. Xtls then allowed to dry in desiccator.

1/13/06. Mass of Dried xtl from above 0.92 g.

Theor. yield 8.0 mmoles aspirin = 1.44 g

Crude yield $\frac{0.92}{1.44} (100) = 64\%$

mp. of crude xtl 127-135°C (lit. 134-136°C)

Crude xtl recrystall'd from hot MeOH/ H_2O .

Ca 2 mL hot MeOH req'd to just dissolve ~~xtls~~ xtl. Hot H_2O added

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(49)

dr-wise until faint cloudiness - ca 6 dr req'd.
Soln cooled in ice bath and mass
of white needles formed. After
suction filtration, x'tals dried in
heated vacuum desiccator (30 mmHg,
50°C) for 1 hr. Mass of
dried x'tals 433 mg.

$$\left(\frac{433}{1440} \times 100 = 31\% \text{ yield} \right)$$

mp. 133-134°C.

* IR ~~th~~ nujol mull on NaCl plates

3300 - 2900
~~3400 - 3250~~ cm⁻¹ broad
1725 cm⁻¹ sharp, strong
1690 cm⁻¹ sharp strong

* ¹H-NMR 60 MHz CDCl₃ ~5% soln

δ 11.9 1H (disappears w/ D₂O)

δ 7-8 4H multiplet

δ 2.4 1H singlet

* spectrum IR-623
NMR-83
in the book in Appendix