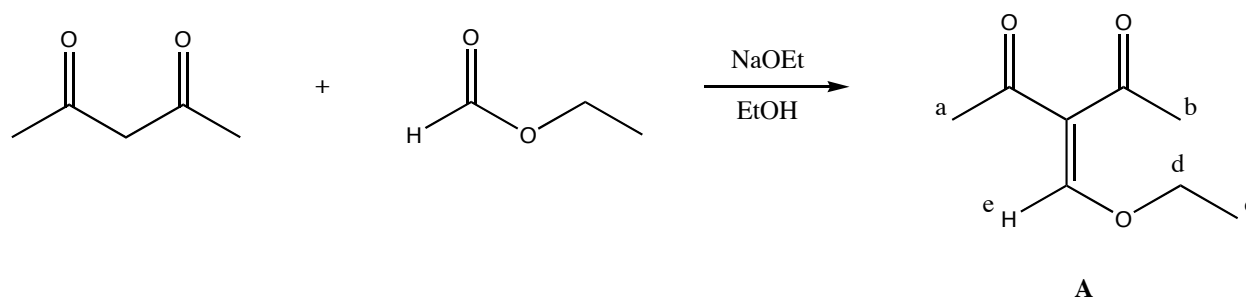


## Model Lab Report Discussion

I isolated product A from the reaction of 2,4-pentanedione and ethyl formate.<sup>1</sup>



NMR and IR spectroscopy confirm the product's identity and also confirm that little or no starting materials or other products were present. I predicted the following NMR spectrum for the labeled product:

Hydrogen type	Integration	Chemical Shift (ppm)	Splitting
a	3	2.5-3	singlet
b	3	2.5-3	singlet
c	3	1.5-2	triplet
d	2	3.5-4	quartet
e	1	5-6	singlet

The two ketone CH<sub>3</sub> groups should be different because the molecule is not symmetrical (there is restricted rotation around the C=C).<sup>2</sup> The five predicted signals are present in the NMR spectrum of my product.<sup>3</sup> The integrations and splitting are all as predicted, but the chemical shift for e is higher (8 ppm) than I expected. I suspect the chemical shift is higher than a normal alkene proton because of its proximity to the oxygen of the OCH<sub>2</sub>CH<sub>3</sub> group.<sup>4</sup> All of the other chemical shifts are within the expected range. I should also note that the ketone CH<sub>3</sub> groups are two separate singlets as I predicted. There appears to be no 2,4-pentanedione present since the CH<sub>2</sub> group does not appear at 3.3 ppm as seen in the standard spectrum.<sup>5</sup> There is a very small triplet at 1.7 ppm and a quartet at 3.8 ppm. These signals probably indicate an ethyl group (CH<sub>2</sub>CH<sub>3</sub>). I know it is not from the starting ethyl formate because the aldehyde proton is not present at 9-10 ppm. Perhaps it is the ethyl group of some ethanol that was not distilled off during the fractional distillation. The OH proton of the ethanol does not show up in the NMR spectrum because it has exchanged with the deuterium from CDCl<sub>3</sub>.

The IR spectrum of my product also supports its identity. The strong, sharp C=O absorption is present at 1690 cm<sup>-1</sup>.<sup>6</sup> This absorption is a little lower than usual for a ketone C=O which usually occurs at about 1720 cm<sup>-1</sup>. The conjugation of the C=O with the C=C accounts for the difference.<sup>7</sup> The medium, sharp C=C of the product is also present at 1590 cm<sup>-1</sup>, again a little low because of conjugation. The strong, sharp C-O is present at 1210 cm<sup>-1</sup> and the medium, sharp =C-H is just to the left of the sp<sup>3</sup> C-H absorptions at about 3050 cm<sup>-1</sup>.<sup>8</sup> There is a medium, broad absorption at 3400 which is probably due to the O-H of water or ethanol.<sup>9</sup>

The product I isolated was the one that I predicted based on the mechanism of the reaction. The 2,4-pentanedione has two types ( $\text{CH}_3$  and  $\text{CH}_3$ ) of protons that can be abstracted by the base in the first step of the reaction. A proton from the  $\text{CH}_2$  group is abstracted because the resulting negative charge can be stabilized better by resonance.<sup>10</sup>

*(Don't worry about the rest of the mechanism—you'll learn it in class soon!)*

Despite the fact that I isolated the correct product, my percent yield was a little low. I would improve the experiment in the following ways...

#### NOTES

- (1) Rewrite the reaction you are discussing with labels on the product for the NMR
- (2) If there are any odd features in your predicted NMR you should discuss them
- (3) **ALL** of the signals of a molecule must be present for you to conclude that it's there! Sometimes a signal can be obscured and you would have to state that and where it should be.
- (4) If a signal falls outside of the range you predicted, you should speculate on the reasons for that. Similarly, if integration is different than predicted (this should never happen) or splitting is different, then those discrepancies should be explained.
- (5) You can conclude a compound is not present if a key signal is not present. Note the difference to #3!
- (6) When discussing IR absorptions it is necessary to describe them as strong/medium/weak and sharp or broad.
- (7) Once again, any deviations from the expected are explained.
- (8) **ALL** expected absorptions in the product that are **different** than the starting materials should be present.
- (9) Be careful when attributing absorptions to entire compounds. I state that the absorption is due to the OH of ethanol not the absorption is due to ethanol. The same holds true for NMR.
- (10) You must offer mechanistic proof when multiple products are possible. The only way you can do this is by writing out the mechanism and discussing any key parts of it.