Synthesis of Dibenzalacetone

In a 10 mL Erlenmeyer flask, place 2 mL (30 drops)of 3M NaOH, 0.212 g (7 drops) of benzaldehyde and 1.6 mL (20 drops) of 95% ethanol containing 58 mg acetone per 1.6 mL. It is important to add the acetone solution last (why?). Mix the flask vigorously. The initially cloudy solution should become clear pale yellow. After a few minutes, the solution should become suddenly cloudy and a yellow precipitate should begin to form. Continue to mix the flask occasionally for 30 minutes. If no precipitate forms, scratch the inside of the tube with a glass rod and try to beg a seed crystal from someone more fortunate.

Collect the product by vacuum filtration. Wash the crystals with several small portions of cold water and then draw air through them for several minutes to remove most of the water.

Recrystallize the crude material using 70:30 ethanol:water as solvent. Cool very slowly to obtain the most attractive crystals. Once the solution is at room temperature and crystallization is no longer occurring, cool in an ice bath for a few minutes and then vacuum filter. After the crystals have air-dried (next week), calculate the %yield, determine the melting point (literature value for dibenzalacetone is 110-112° C), and obtain an IR spectrum.

Points to ponder:

1) There are actually three stereoisomeric dibenzalacetones. What are they?

2) In the ^{1}H-NMR spectrum of dibenzalacetone, the vinylic protons have chemical shifts of 7.08 ppm and 7.75 ppm. Each signal is a doublet and the coupling constant is 17 Hz. What does this tell you?

3) What side products are probably produced in the reaction?