

Preparing a Laboratory Record

Use the following steps to prepare your laboratory record. The letters correspond to the completed laboratory records that appear at the end of this chapter. Because your laboratory notebook is so important, two examples, written in alternative forms, are presented.

- A. Number each page. Allow space at the front of the notebook for a table of contents. Use a hardbound, lined notebook, and keep all notes in ink.
- B. Date each entry.
- C. Give a short title to the experiment, and enter it in the table of contents.
- D. State the purpose of the experiment.
- E. Write balanced equation(s) for the reaction(s).
- F. Give a reference to the source of the experimental procedure.
- G. Prepare a table of quantities and physical constants. Look up the needed data in the *Handbook of Chemistry and Physics*.
- H. Write equation(s) for the principal side reaction(s).
- I. Write out the procedure with just enough information so that you can follow it easily. Do not merely copy the procedure from the text. Note any hazards and safety precautions. A highly experienced chemist might write a procedure in a formal report as follows: "Dibenzalacetone was prepared by condensing at room temperature 1 mmol acetone with 2 mmol benzaldehyde in 1.6 mL of 95% ethanol to which was added 2 mL of aqueous 3 M sodium hydroxide solution. After 30 min the product was collected and crystallized from 70% ethanol to give 0.17 g (73%) of flat yellow plates of dibenzalacetone, mp 110.5–111.5°C." Note that in this formal report, no jargon (e.g., EtOH for 95% ethanol, FCHO for benzaldehyde) is used and that the names of reagents are written out (sodium hydroxide, not NaOH). In this report the details of measuring, washing, drying, crystallizing, collecting the product, and so on are assumed to be understood by the reader.
- J. Don't forget to note how to dispose of the byproducts from the experiment using the "Cleaning Up" section of the experimental procedure.
- K. Record what you do as you do it. These observations are the most important part of the experiment. Note that conclusions do not appear among these observations.
- L. Calculate the theoretical yield in grams. The experiment calls for exactly 2 mmol fluorobenzaldehyde and 1 mmol acetone, which will produce 1 mmol of product. The equation for the experiment also indicates that the product will be formed from exactly a 2:1 ratio of the reactants. Experiments are often designed to have one reactant in great excess. In this experiment, a very slight excess of fluorobenzaldehyde was used inadvertently, so acetone becomes the limiting reagent.
- M. Once the product is obtained, dried, and weighed, calculate the ratio of product actually isolated to the amount theoretically possible. Express this ratio as the percent yield.
- N. Write out the mechanism of the reaction. If it is not given in the text of the experiment, look it up in your lecture text.

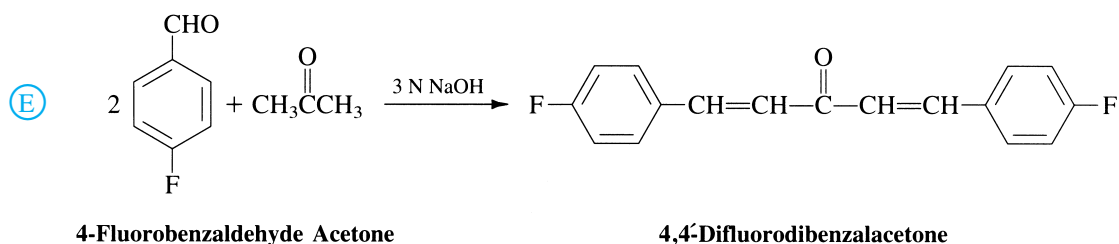
- O. Draw conclusions from the observations. Write this part of the report in narrative form in complete English sentences. This part of the report can, of course, be written after leaving the laboratory.
- P. Analyze TLC, IR, and NMR spectra if they are a part of the experiments. Rationalize observed versus reported melting points.
- Q. Answer assigned questions from the end of the experiment.
- R. This page presents an alternative method for entering the experimental procedure and observations in the notebook. Before coming to the laboratory, enter the procedure in outline form on the left side of the page. Then enter observations in brief form on the right side of the page as the experiment is carried out. Draw a single line through any words incorrectly entered. Do not erase or obliterate entries in the notebook, and never remove pages from the notebook.

Two samples of completed laboratory records follow. An alternative method for recording procedure, cleaning up, and observations is given on p. 24. The other parts of the report are the same.

(A) p. 35
(B) Sept. 23, 2004

(C) DIFLUORODIBENZALACETONE

(D) *Purpose:* To observe and carry out an aldol condensation reaction.



(F) *Reference:* Williamson, *Macroscale and Microscale Organic Experiments*, p. 792.

Table of Quantities and Physical Constants (G)

Substance	Mol wt	G/mol Used or Produced	Mol Needed (from eq.)	Density	mp	bp	Solubility
4-Fluorobenzaldehyde	124.11	0.248/0.002	0.002	1.157		181°C	Slightly soluble in H ₂ O; soluble in ethanol, diethyl ether, acetone
Acetone	58.08	0.058/0.001	0.001	0.790		56°C	Soluble in H ₂ O, ethanol, diethyl ether, acetone
4,4'-Difluoro-dibenzalacetone	270.34	0.273/0.001			167°C		Insoluble in H ₂ O; slightly soluble in ethanol, diethyl ether, acetone
4-Fluorobenzalacetone	164.19					54°C	Insoluble in H ₂ O; slightly soluble in ethanol, diethyl ether, acetone
Sodium hydroxide	40.01						Soluble in H ₂ O; insoluble in ethanol, diethyl ether, acetone

(H) *Side Reactions:* No important side reactions, but if excess acetone is used, product will be contaminated with 4-fluorobenzalacetone.

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- I** *Procedure:* To a reaction tube, add 0.248 g (0.002 mole) of benzaldehyde and then 1.6 mL of an ethanol solution that contains 0.58 mg of acetone. Mix the solution and observe it for 1 min. Remove sample for TLC. Then add 2 mL of 3 M NaOH(aq). Cap the tube with a septum and shake it at intervals over a 30-min period. Collect product on Hirsch funnel, wash crystals thoroughly with water, press as dry as possible, determine crude weight, and save sample for mp. Recrystallize product from 70% ethanol in a 10-mL Erlenmeyer flask. Cool solution slowly, scratch or add seed crystal, cool in ice, and collect product on Hirsch. Wash crystals with a few drops of ice-cold ethanol. Run TLC on silica gel plates using hexane to elute, develop in iodine chamber. Run NMR spectrum in deuteriochloroform and IR spectrum as a mull.
- J** *Cleaning Up:* Neutralize filtrate with HCl and flush solution down the drain. Put recrystallization filtrate in organic solvents container.
- K** *Observations:* Into a reaction tube, 0.250 g of 4-fluorobenzaldehyde was weighed (0.248 called for). Using the 1.00-mL graduated pipette and pipette pump, 1.61 mL of the stock ethanol solution containing 0.58 mg of acetone was added (16 mL called for). The contents of the tube were mixed by flicking the tube, and then a drop of the reaction mixture was removed and diluted with 1 mL of hexane for later TLC. The water-clear solution did not change in appearance during 1 min.

Then about 2 mL of 3 M NaOH(aq) was added using graduations on side of reaction tube. The tube was capped with a septum and shaken. The clear solution changed to a light yellow color immediately and got slightly warm. Then after about 50 sec, the entire solution became opaque. Then yellow oily drops collected on sides and bottom of tube. Shaken every 5 min for ½ hr. The oily drops crystallized, and more crystals formed. At the end of 30 min, the opaque solution became clear, and yellow crystals had formed. Tube filled with crystals.

Product was collected on 12-mm filter paper on a Hirsch funnel; transfer of material to funnel was completed using the filtrate to wash out the tube. Crystals were washed with about 15 mL of water. The filtrate was slightly cloudy and yellow. It was poured into a beaker for later disposal. The crude product was pressed as dry as possible with a cork. Wt (damp) 270 mg. Sample saved for mp.

Recrystallized from about 50 mL of 70% ethanol on sand bath. Almost forgot to add boiling stick! Clear, yellow solution cooled slowly by wrapping tube in cotton. A seed crystal from Julie added to start crystallization. After about 25 min, flask was placed in ice bath for 15 min, then product was collected on Hirsch funnel (filter paper), washed with a few drops of ice-cold solvent, pressed dry and then spread out on paper to dry. Wt 0.19 mg. Very nice flat, yellow crystals, mp 179.5–180.5°C. The crude material before recrystallization had mp 177.5–179°C.

TLC on silica gel using hexane to elute gave only one spot, R_f 0.23, for the starting material and only one spot for the product, R_f 0.66. See attached plate.

The NMR spectrum that was run on the Bruker in deuteriochloroform containing TMS showed a complex group of peaks centered at about 7 ppm with an integrated value of 17.19 and a sharp, clear quartet of peaks centered at 6.5 ppm with an integral of 8.62. The coupling constant of the AB quartet was 7.2 Hz. See attached spectrum.

The IR spectrum, run as a Nujol mull on the Mattson FT IR, showed intense peaks at 1590 and 1655 cm^{-1} , as well as small peaks at 3050 , 3060 , and 3072 cm^{-1} . See attached spectrum.

Cleaning Up: Recrystallization filtrate put in organic solvents container. First filtrate neutralized with HCl and poured down the drain.

L *Theoretical Yield Calculations:*

$$0.250\text{ g fluorobenzaldehyde used } \frac{0.250\text{ g}}{124.11\text{ g/mole}} = 0.00201\text{ mole}$$

$$0.058\text{ g of acetone used } \frac{0.058\text{ g}}{58.08\text{ g/mole}} = 0.001\text{ mole}$$

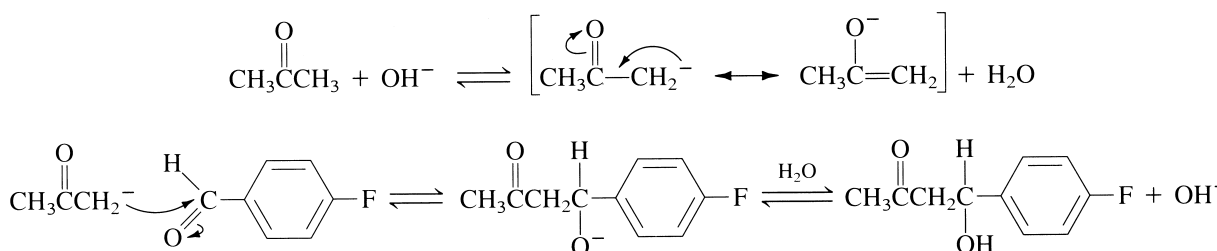
The moles needed (from equation) are 2 mmol of the benzaldehyde and 1 mmol of acetone. Therefore, acetone is the limiting reagent, and 1.00 mmol of product should result. The MW of the product is 270.34 g/mol.

$$0.001\text{ mol} \times 270.34\text{ g/mol} = 0.270\text{ g, theoretical yield of difluorodibenzalacetone}$$

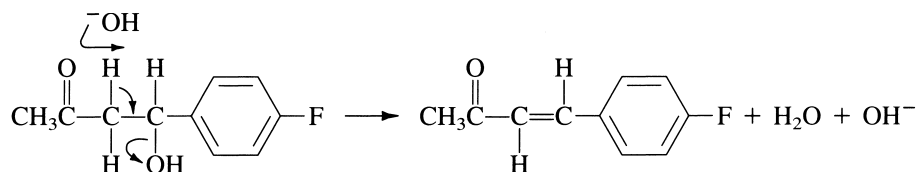
M *Percent Yield of Product:*

$$\frac{0.19\text{ g}}{0.270\text{ g}} \times 100 = 70\%$$

N *The Mechanism of the Reaction:*



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Discussion and Conclusions: Mixing the fluorobenzaldehyde, acetone, and ethanol gave no apparent reaction because the solution did not change in appearance, but less than a minute after adding the NaOH, a yellow oil appeared and the reaction mixture got slightly warm, indicating that a reaction was taking place. The mechanism given in the text (above) indicates that hydroxide ion is a catalyst for the reaction, confirmed by this observation. The reaction takes place spontaneously at room temperature. The reaction was judged to be complete when the liquid surrounding the crystals became clear (it was opaque) and the amount of crystals in the tube did not appear to change. This took 30 min. The crude product was collected and washed with water. Forgot to cool it in ice before filtering it off. The crude product weighed more than the theoretical because it must still have been damp. However, this amount of crude material indicates that the reaction probably went pretty well to completion. The crude product was recrystallized from 5 mL of 70% ethanol. This was probably too much, since it dissolved very rapidly in that amount. This and the fact that the reaction mixture was not cooled in ice probably accounts for the relatively low 70% yield. Probably could have obtained second crop of crystals by concentrating filtrate. It turned very cloudy when water was added to it.

Theoretically, it is possible for this reaction to give three different products (*cis, cis*-; *cis, trans*-; and *trans, trans*-isomers). Because the mp of the crude and recrystallized products were close to each other and rather sharp, and because the TLC of the product gave only one spot, it is presumed that the product is just one of these three possible isomers. The NMR spectrum shows a sharp quartet of peaks from the vinyl protons, which also indicates only one isomer. Since the coupling constant observed is 7.2 Hz, the protons must be *cis* to each other. Therefore, the product is the *cis, cis*-isomer.

The infrared spectrum shows two strong peaks at 1590 and 1655 cm^{-1} , indicative of an α - β -unsaturated ketone. The small peaks at 3050, 3060, and 3072 cm^{-1} are consistent with aromatic and vinyl protons. The yellow color indicates that the molecule must have a long conjugated system.

The fact that the TLC of the starting material showed only one peak is probably due to the evaporation of the acetone from the TLC plate. The spot with R_f 0.23 must be from the 4-fluorobenzaldehyde.

Answers to assigned questions are written at the end of the report.

An alternative method for recording procedures, cleaning up, and observations:

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Ⓡ	Sept. 23, 2004 Procedure	Sept. 24, 2004 Observations
	Weigh 0.248 g of 4-fluorobenzaldehyde into reaction tube.	Actually used 0.250 g.
	Add 1.6 mL of ethanol stock soln that contains 0.58 g of acetone.	Actually used 1.61 mL.
	Mix, observe for 1 min.	Nothing seems to happen. Water clear soln.
	Add 2 mL of 3 M NaOH(aq).	Clear, then faintly yellow but clear, then after about 50 sec, the entire soln very suddenly turned opaque. Got slightly warm. Light yellow color.
	Cap tube with septum; shake at 5-min intervals for 30 min.	Oily drops separate on sides and bottom of tube. These crystallized; more crystals formed. Tube became filled with crystals. Liquid around crystals became clear.
	Filter on Hirsch funnel.	Filtered (filter paper) on Hirsch.
	Wash crystals with much water.	Washed with about 150 mL of H ₂ O. Transfer of crystals done using filtrate.
	Press dry.	Crystals pressed dry with cork.
	Weigh crude.	Crude (damp) wt 0.27 g.
	Save sample for mp.	Crude mp 177.5–179°C.
	Recrystallize from 70% ethanol; wash with a few drops of ice-cold solvent.	Used about 50 mL of 70% EtOH. Too much. Dissolved very rapidly.
	Dry product.	Dried on paper for 30 min.
	Weigh.	Wt 0.19 g.
	Take mp.	Mp 179.5–180.5°C.
	Run IR as mull.	Done.
	Run NMR in CDCl ₃ .	Done.
	Neutralize first filtrate with HCl, pour down drain.	Done.
	Org. filtrate in waste organic bottle.	Done.