

Chem 228 WS/2009  
Synthesis Project-Library Assignment  
(50 pts., due week of 3/18-3/20)

Name \_\_\_\_\_

Section \_\_\_\_\_

StdntNo \_\_\_\_\_

**Attach this Sheet to Your Search Results**

The following searches are to locate needed information about compounds involved in your synthesis project and to familiarize you with some of the standard reference sources for organic compound information.

I would suggest working in groups for efficiency in obtaining information, however you should actually be present to see how SciFinder works, where the reference books are located, how they are indexed, etc. These reference sources are useful for all reagents and intermediates produced in your synthesis. You might want to assign a different intermediate to each member of a team and make copies for the others.

Q #1 & 2 use the web.

Q #3-5 require a trip to the library.

Q #6 requires ChemDraw, found in CLC rm. 120 of chemistry.

1. Using *SciFinder*, select "Locate" and do a "Substance Identifier" search for

**3-buten-2-one, 4,4-diphenyl- and 1,1-diphenyl-1-hydroxy-3-butanone.**

Determine the CAS #. Click on the microscope icon, then click the experimental properties link. Locate the BP at the listed pressures and the IR spectrum. Click the predicted properties link and print out the <sup>1</sup>H NMR.

Record compound name, CAS#, BP data, IR, and <sup>1</sup>H NMR for both compounds and attach.

Click "Get References" at the bottom of the *SciFinder* screen, then "OK" for all references.

Locate the article by D.R. Paulson in J Chem Ed, 1973 titled "The Ethylene Ketal Protecting Group in Organic Synthesis".

Click on the page icon at the right of the citation to connect to the online J Chem Ed. Select "previous issues" on the left of the J Chem Ed page and select the proper issue.

Print out the J Chem Ed article and attach. This is your basic experimental procedure.

**In Lab: We will begin the first step of the synthesis the week after St. Pat's.**

Expect a quiz over step 1.

You should come prepared with a procedure scaled to one half of the amounts given in the J Chem Ed paper and substituting toluene for benzene in the first step.

Calculate the amount of water predicted in step 1 based on ethyl acetoacetate as limiting. You might want to look up acetals and acetals, cyclic in Solomons "Organic Chemistry".

You should also have the usual prelab property table prepared in your lab book and an MSDS form covering chemicals to be used in the first step.

Further modifications will be provided for later steps.

2. Go to "*The Purification of Laboratory Chemicals*", **TP 156.P83 P47** (in hardcopy only, at the MST library in the reference section) and find purification information for ethyl acetoacetate.

Xerox, or scan to pdf and attach.

3. Go to "*The Dictionary of Organic Compounds*", **QD246.D5** (in hardcopy only, at the MST library in the reference section) and find the information for ethyl acetoacetate. Using the CAS# in the CAS# index to locate is probably easiest.

Xerox, or scan to pdf and attach.

4. Go to "*The Handbook of Data on Organic Compounds*", **QD 257.7 H36** (in hardcopy only, at MST library in the reference section) and find the information for ethyl acetoacetate. Use the CAS# index to locate.

Xerox, or scan to pdf and attach.

5. ChemBioDraw: Go to the chemistry CLC in rm 120 of chemistry.

Rm 120 PCs (pgm. not on Macs): Follow this path:

*Start* → *Programs* → *ChemBioOffice 2008* → *ChemBioDraw Ultra 11.0*

In Chem Draw, From the **Structure** menu, select "**Convert Name to Structure**".

Type: **3-buten-2-one, 4,4-diphenyl-** and hit Return.

From the **View** menu, select "**Show Analysis Window**".

Select the Formula and Mol. Wt. boxes and hit Paste to add this to the structure.

From the **Structure** menu, select "**Predict 1H NMR shifts**".

A new window containing the spectrum will appear.

Copy and paste the formula and MWt from the previous page on the NMR.

If you need to resize anything to make it fit, just grab the lower rt. corner of the selection border, hold down the mouse button and drag the corner diagonally toward the upper left to make it smaller.

Print and attach the 1H NMR spectrum for **3-buten-2-one, 4,4-diphenyl-**.

Repeat to generate the 1H NMR for **1,1-diphenyl-1-hydroxy-3-butanone**.

Note that if you compare the spectra generated in Q #1 and Q #5, they may differ slightly. Both are simulated, but by different programs.

The actual experimental spectrum may differ from both of these, since they are not actual spectra but simulated from tables of data for similar structures.