The project this semester is the synthesis of an ester fragrance of your choosing. You need to determine what fragrance you want to make, then find adequate preparative directions to allow you to do this. A total of three reactions are needed to fill up the available time. You may either make the same ester via different routes or possibly make the starting compounds (alcohol & acid, acid chloride or anhydride) to give you a total of three reactions. All products will need to be purified and characterized by IR & NMR and possibly GC or GC/MS. The results of these syntheses will be written up at the end of the semester as a formal paper as if you were publishing it in the Journal of Organic Chemistry. You should compile a list of all chemicals and equipment that you will need prior to beginning and obtain approval by me as to the feasibility of your procedures.

In this paper you should give the exact procedures you will be using, along with a list of needed chemicals and amounts. This should be scaled to about 100 mmol of product. MSDS info should be obtained to identify any hazards. References should be cited for information used in the paper.

<u>Sources for specific experimental details:</u>

Online:

JChemEd (online, see 228 web page link)-numerous experiments and background articles.

SciFinder: Provides access to the primary chemical literature. You may need to narrow down topic searches to limit the number of articles listed. Some journals may be accessed directly from the citation if the library has an electronic subscription. Patents may be a useful source for info on reaction conditions and product isolation by distillation.

Organic Synthesis (online), http://www.orgsyn.org/default.asp-may have a few ester preps.

Books (on 228 class reserve at library, except for **reference** books)

Organic Functional Group Preparations, chapt. 10,Sandler-example preps via various methods
Vogel's Textbook of Practical Organic Chemistry-various prep procedures
Esterification, Otera-general review of lit prep procedures & some specific ester preps
Organic Synthesis, chapt. 7, Migrdichian-overview of prep procedures
The Chemistry of Carboxylic Acids & Esters, chapt. 11, Patai-general info on esters
Comprehensive Chemical Kinetics, v.10, chapt. 2, -kinetics of hydrolysis and formation of esters
Azeotropic Data, QD1.A355 no.6, v. 1 & 2, no. 116, v. III-data on azeotropes
Merck Index (online via MST library, see 288 web page link), also MST **reference** RS51.M4
Purification of Laboratory Chemicals, MST **reference** TP156.P83 P47, also online via library
Handbook of Data on Organic Compounds, MST **reference** QD257.7.H36
Dictionary of Organic Compounds, MST **reference** QD246.D5

<u>Additional Info needed for a complete prep procedure:</u>

Isolation/purification of product
Drying of product (selection of proper drying agent)
Neutralization of residual acid catalyst (if used)
IR, NMR, MS of product (look online in SDBS database for ref spectra)
GC of product

NOTE: Xerox machines on the first floor of the library can scan documents to pdf which can be stored on a thumb drive allowing free copying of reference materials.

Limits for using a literature procedure: The following reagents, conditions or procedures may preclude use of a prep procedure in org lab.

Time

Reflux time >3 hr. Extended times at RT are OK.

Conditions

High temp >200 ^{o}C
Low temp < -80 ^{o}C
High pressure > 1 atm
Low pressure < 15 torr (hi vac distillation)
Microwave heating is possible with our available equipment.

Reagents

Pyrophoric reagents: NaH, $LiAlH_4$, Na, K metal
Expensive catalysts: Pt, Pd, Rh, Ti
Enzymatic catalysts
Gaseous reactants: H_2 gas, other gaseous reactants (CO, BF_3)
Unavailability of reactants, solvents (check with me)

Other Procedural Details

Column chromatography used on colorless compounds for product isolation is very tedious.
Insufficient details on time, concentrations, workup or product isolation provided in procedure.
More than three reaction steps required.
Reaction requires N_2 atmosphere-is doable, just more complex apparatus setup.