

Chem 228 WS/2011
Synthesis Project-Library Assignment
(due week of 3/16/11 - 3/18/11)

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.

1. Procedure-do a search of the Journal of Chemical Education for "spearmint" and find an article by Otis S. Rothenberger. Print out and attach. Keep a copy for yourself.
2. Background-the following articles provide some background from which the procedure above was derived. You do not have to print these out, but might want to save them as pdfs for future reference.

via <http://pubs.acs.org/action/showPublications?display=journals>

Journal of Organic Chemistry 23, 2038 (1958)

Journal of the American Chemical Society 73, 5856 (1951)

Chemical Reviews 48, 329 (1951)

via <http://um9mh3ku7s.search.serialssolutions.com/>

Russian Chemical Reviews 37(7), 543 (1968)

via <http://www.pat2pdf.org/>

US Patent #3,293,301

The Synth Project WS11 link on 228 web page provides links to various aspects of the synthesis and compounds involved.

3. MSDS info
for limonene, limonene nitrosochloride, carvoxime, carvone and reagents used to produce them from J. Chem. Ed. article above.
You will need MWt, MP, BP, structures, CAS# for all reagents and products. Also IR, NMR, MS spectra for the products.

Recommended sources: Synth Project WS11 link, SciFinder, SDBS, Merck Index

4. Summary of reactions:
step 1 R(+)-Limonene is converted to R(+)-limonene nitrosochloride
step 2 R(+)-limonene nitrosochloride is converted to R(-)-carvoxime
step 3 R(-)-carvoxime is converted to R(-)-carvone

Attach your balanced reactions to this sheet and keep another copy for yourself.

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

Expect a quiz over step 1. Gloves are recommended for all steps.

You should come prepared to do step 1 of the procedure from JChemEd above.

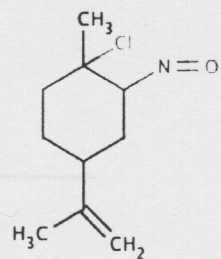
You should also have the usual prelab property table prepared in your lab book and an MSDS form covering chemicals to be used in the first step.

5. ChemBioDraw: Go to the chemistry CLC in rm G-34 of chemistry.

Rm G-34 PCs: Follow this path:

Start → Programs → ChemBioOffice 2010 → ChemBioDraw Ultra 12.0

In Chem Draw, draw the structure of limonene nitrosochloride shown here →



Select the structure and from the **Structure** menu, select "**Clean Up Structure**"

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.

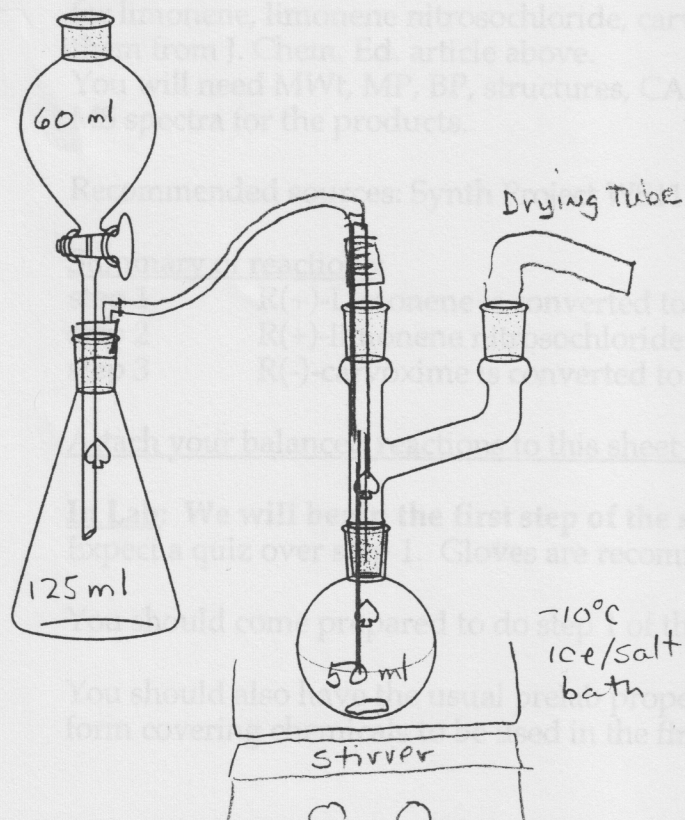
Copy and paste the formula and MWt from the previous page on the NMR.

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 limonene nitrosochloride.

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

6. Sketch of Apparatus setup for step 1.
Generation of EtONO



Generation of NOCl

