

General Notes

1) Lab Report Due for Experiment #8 (Grignard) Nov. 29/30

- Note: i) the ^1H NMR spectrum of the final product is provided on the website for comparison (only NMR provided and will need to be interpreted – along with your experimentally determined NMR spectrum)
- ii) NMR solvent is DMSO (peak at 2.49 ppm)

2) Lab Report Due for Experiment #9 (Unknown): Dec. 6/7

3) No lecture next week (Friday, November 19), Extra Office Hour

4) Review Session: Friday, December 3 in W 151 PBB

5) Exam 2, Friday December 10, will be held in W 151 PBB

6) Regrades of Exam 1 available in Chemistry Center and regrades of labs will be handed back by your TA

Experiment #9: Identification of Unknown Sample

- purpose of the experiment is to determine the structure of an unknown compound using chemical tests and spectroscopic data normally available in an organic laboratory

Advice

- it is crucial to maintain a good written account of each step
- beware of false positives and false negatives
- be ready to make decisions between contrary information

Functional Groups

$\text{R}-\text{OH}$ alcohol



phenol



carboxylic acid



ester



aldehyde

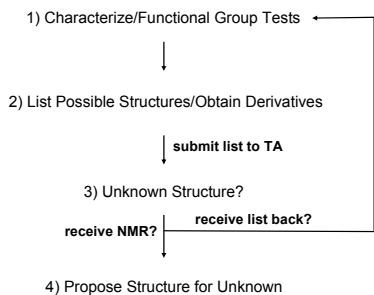


amine



ketone

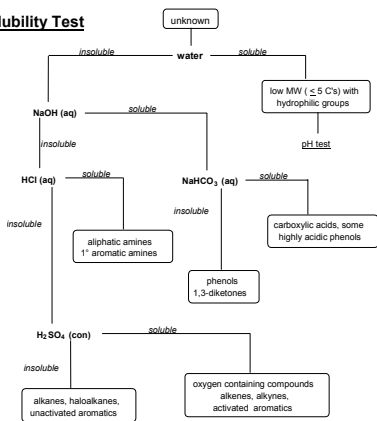
Four General Steps



Characterize/Functional Group Tests

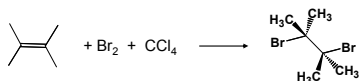
- 1) Obtain unknown from TA
- 2) Note appearance (solid, liquid, color, crystalline, etc.)
- 3) Determine solubility (organic, aqueous, aq. NaHCO_3 , aq. NaOH , aq. HCl)*
- 4) Prepare unknown sample for NMR analysis
- 5) Obtain melting point or boiling point (repeat for accuracy)
- 6) Burn small amount of sample (aromatic flame test)
- 7) Obtain IR [liq.(neat), solid (nujol, CCl_4 , CHCl_3)]
- 8) Conduct at least three functional group tests (should run positive controls)*

Step 1 - Solubility Test

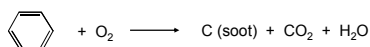


Functional Group Tests

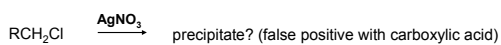
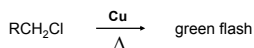
Alkenes (Bromination)



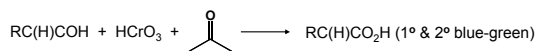
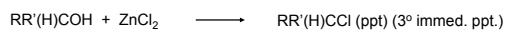
Aromatics (Flame Test)



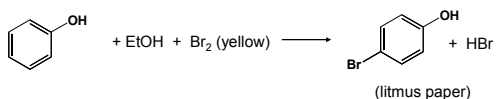
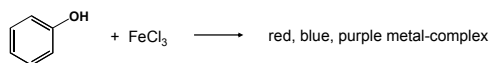
Alkyl Halides (Beilstein Test, Silver Nitrate Test)



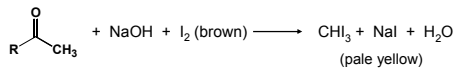
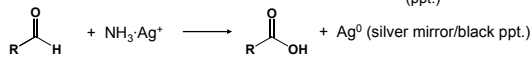
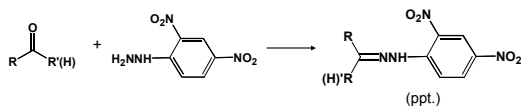
Alcohols (Lucas Test, Chromic Acid Test)



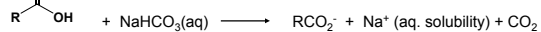
Phenols (Ferric Chloride Test, Bromine Test)



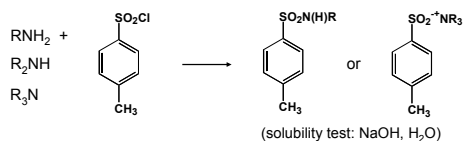
Aldehydes & Ketones (Dinitrophenylhydrazine, Tollens, Iodoform)



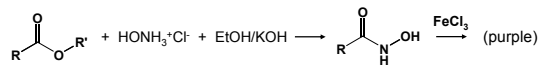
Carboxylic Acids (Acidity, Bicarbonate)



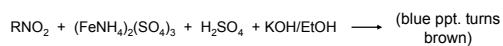
Amines (Basicity, Hinsberg Test)



Esters (Hydroxyl Amine/Ferric Chloride)



Nitro Compounds (Iron(II) Hydroxide Test)

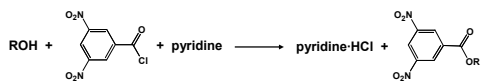
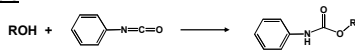


List Possible Structures/Obtain Derivatives

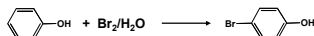
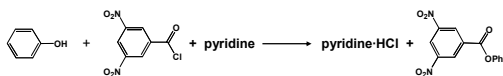
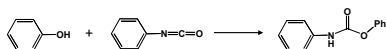
- 1) Develop a list of possibilities from the chart in the lab (and on web)
- 2) Propose 3-5 structures based on properties and derivatives
- 3) Submit your list of possible structures to TA (use datasheet)
 - a) give chemical names
 - b) give chemical structures
 - c) give known mp range of derivatives
- 4) Synthesize appropriate derivative and obtain melting point (sharp)

Derivatives

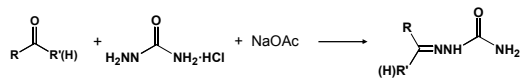
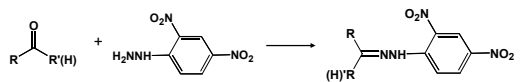
Alcohols



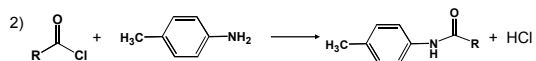
Phenols



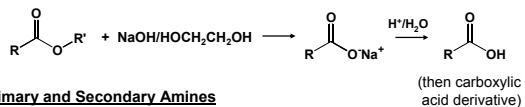
Aldehydes and Ketones



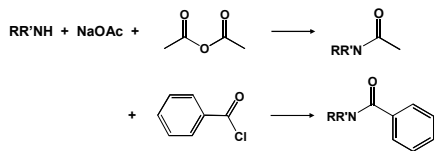
Carboxylic Acids



Esters



Primary and Secondary Amines



Tertiary Amines



Unknown Structure?

- 1) If you receive list back – more experimental work is needed
- three total attempts (attempts #2 and #3 result in a 5 pt. deduction each)
- 2) If you receive spectrum – interpret, synthesize derivative, and define unknown

Propose Structure of Unknown

- 1) Propose a single structure for unknown
- 2) Prepare unknown report

Procedures and Precautions

- 1) beware of false positives and false negatives (outlined in course pack)
 - example: FeCl_3 test does not work with all phenols (e.g. steric effects)
- 2) use fresh sample for each solubility test
- 3) use 'clean' glassware
 - Note: acetone is a ketone (e.g. participates in iodoform test)
- 4) have material ready for NMR analysis on Day 1
- 5) crucial to maintain a good written account of each step
- 6) provide logical account of decision making in lab report
- 7) do not overuse all of your sample (5 pt. deduction)
