Lab Report: Synthesis of Zyban

NAME:_____

PARTNER'S NAME:_____

LAB SECTION:_____

DATE:_____

% SCORE:

	Pts possible	Pts received
Lab Notebook		
Abstract		
Spectroscopy		
Questions		
Discussion (Rxn Success)		
Total		

A. ABSTRACT:

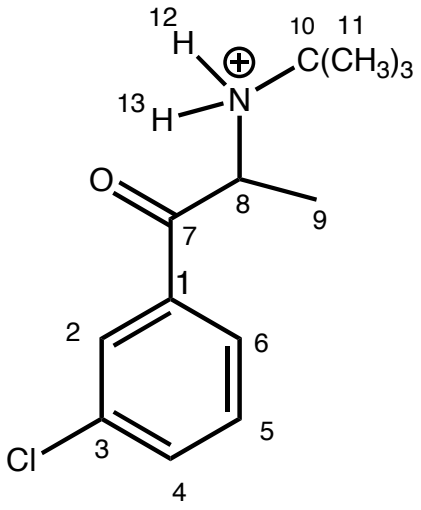
B. SPECTROSCOPIC ANALYSIS

1. IR SPECTROSCOPY

Staple your product IR to your report. Label with your name and sample. Assign the absorptions in the IR spectrum that correspond to the "important" functional groups present: N-H+, C=O, and Aromatic.

2. ^{3}CNMR SPECTRUM

Attach a Carbon-13 NMR (provided) to your report with the peaks labeled with the appropriate numbers shown on the structure at right. (numbers 12 and 13 refer to protons only) The appendix contains substituents tables for approximating the chemical shift of substituted benzene rings. An example for Zyban is given in the appendix.
Label peaks on the expanded section also.



3. HNMR SPECTRUM

a. Report the chemical shifts for H12 and H13,
H12_____ H13_____

b. Are protons H12 and H13 homotopic, enantiotopic or diastereotopic? (Hint:A quarternary nitrogen with 4 unique substituents is chiral). Explain your response using structures.

b. Report the chemical shift observed for protons at H2, H4, H5 H6. For each response explain how you identified the proton using the observed signal splitting and substituent tables.

H2

H4

H5

H6

C. QUESTIONS

1. Alpha Bromination of ketones is acid catalyzed. What acid is formed as the reaction in Step 1 progresses? Show an acid catalyzed mechanism for the Bromination of m-chloropropiophenone.

2. Step 2 is a substitution of bromine by t-butylamine. What type of substitution is occurring Sn1 or Sn2? Explain based on the reaction's substrate and solvent.

3. The reaction mixture from the substitution step 2 is extracted with MTBE to isolate the product. State layer (organic or aqueous) in which each of the following compounds will be present in the highest concentration.

    a. NMP_____
    b. Excess t-butylamine_____
    c. HBr_____
    d. Zyban free base (compound 3a)_____

4. Why are the ether extracts combined then extracted with water at the end of Step 2?

5. The HCl solution used in the final step was made by mixing 20 mL concentrated HCl (12.0 M) with 100 mL isopropyl alcohol. Why wasn't an aqueous HCL solution used for this part of the synthesis?

D. DISCUSSION

This experiment consisted of three steps and multiple extractions- a substantial amount of chemistry for a 3 hour lab. Was your experiment successful? What data confirms that you produced Zyban? (Note an IR of commercial Zyban is in your manual). If you obtained no product, offer an explanation as to where procedural errors were made.