Oxidations and Reductions

Name_____

Partner_____

Technique __ /5 Report ___ /50 Total ____/55

Section I: Introduction

Statement of Purpose: (1 pt)

Write balanced chemical equations for the oxidation and reduction reactions of each substrate.

Oxidations: (1 pt)

Reductions: (1 pt)

Section II: Experimental.

Week I: Oxidation (5 pts): Provide the experimental for the reaction you ran, don't worry about your partner's experimental.

To a solution of _____(alcohol) (____ g, ____ mol) and glacial acetic acid (____ mL, ____ mol) in acetone (____ mL) was added an excess of aqueous solution of NaOCl (0.75 M) dropwise over 15 minutes. The

mixture was stirred vigorously for ____ min until TLC analysis show the starting material was consumed.

Observations: (1 pt)

Workup: (5 pts)

Saturated $NaHSO_3$ (___ mL) was added to the reaction mixture, and complete reduction of excess NaOCl was confirmed by a negative starch-iodide test. The reaction mixture was extracted with ether (___ mL), and the organic layer was washed successively with H_2O (____ mL), saturated aqueous $NaHCO_3$ (____ mL), and H_2O (___ mL). The organic layer was dried ($MgSO_4$) and concentrated to give a white waxy solid (____ g).

Observations: (Include any spectral data that helped you decide that the reaction had or had not gone they way you expected.) (1 pts)

Week II: Reduction (4 pts)

To a solution of _____ (ketone) (___g, ____ mol) in EtOH (___ mL) was added $NaBH_4$ (____ g, ____ mol) in portions such that the temperature remained between ___ and ___° C. After ___ minutes, TLC indicated that the reaction was complete.

Observations: (1 pts)

Workup: (5 pts)

H_2O (____ mL) was added followed by the careful addition of HCl (3 M, ____ mL). After hydrogen evolution ceased, the mixture was heated to reflux and then cooled to room temperature. The mixture was extracted with ether (__ portions of ___ mL), and the combined organic phases were washed with H_2O (___ portions of ____ mL). The organic phases were dried ($MgSO_4$) and concentrated to give a white solid (____ g).

Observations: (Include any spectral data that helped you decide that the reaction had or had not gone they way you expected.) (1 pt)

Section III: Analysis

Calculation of yields:

Ketone: (1 pt)

Alcohol: (1 pt)

Section IV: Questions

(5 pts each)

1) Attach the IR spectrum of your reduction product. Assign the major absorbances to the structural feature that generated them.

2) How can you tell that the reaction was or was not complete (be very specific).

3) One observation that was made throughout all of the laboratory sections is that both the oxidation of borneol and the reduction of camphor were considerably slower than for the cyclohexanol derivative. Consider the mechanisms shown in your course pack and the structures of each of the substrates. How do you explain this observations?

4) Based upon the NMR analysis of the alcohol products from the $NaBH_4$ reduction of the ketones, what is the ratio of diastereomers in each product mixture? Which was more selective? Why (this one will take some thought)?