Names: _____ *Sec. _____*
Chem 227/ Dr. Rusay

Postlab : Chiral Compounds and Green Chemistry: Reduction of a ketone by sodium borohydride and baker's yeast

PART I: Optical rotation, optical purity, enantiomeric excess

Determine the rotation of the product from Method 3 using the polarimeter and complete the following Data Table:

Cell path length = 100. mm	*Temperature* = 25 °C	λ = *589 nm* (sodium D)	*solvent* = H_2O/ethanol	$\alpha_{solvent}$ = 0°

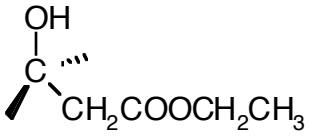
	Mass (g)	Volume (mL)	α	$[\alpha]$ (calc.)
Method 1	4.80	25.00	0°	0°
Method 2	4.14	25.00	7.3°	
Method 3	3.82	25.00		

Calculations:

$[\alpha]_2$ = $[\alpha]_3$ =

Optical Purity (Enantiomeric Excess) Method 3 [Use the calculated $[\alpha]$ value for Method 2 for your calculation in Method 3.]

Complete the drawing for the correct configuration of the enantiomer that is in excess:

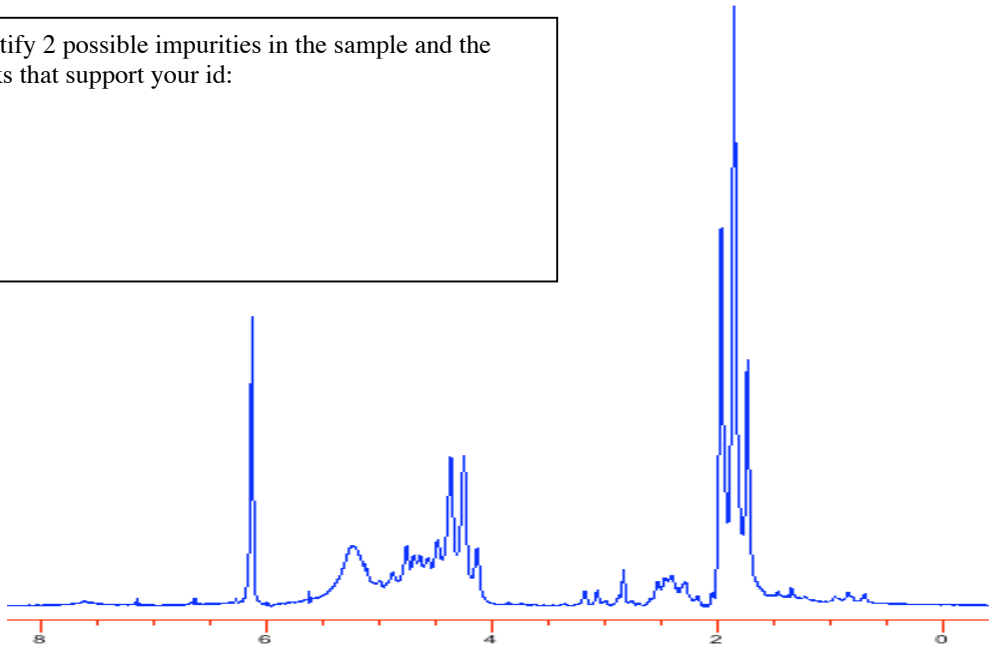


	$[\alpha]$	Total % R-	Total % S-	Enantiomeric Excess: (%)	Abs. Config.
Product Method 3					

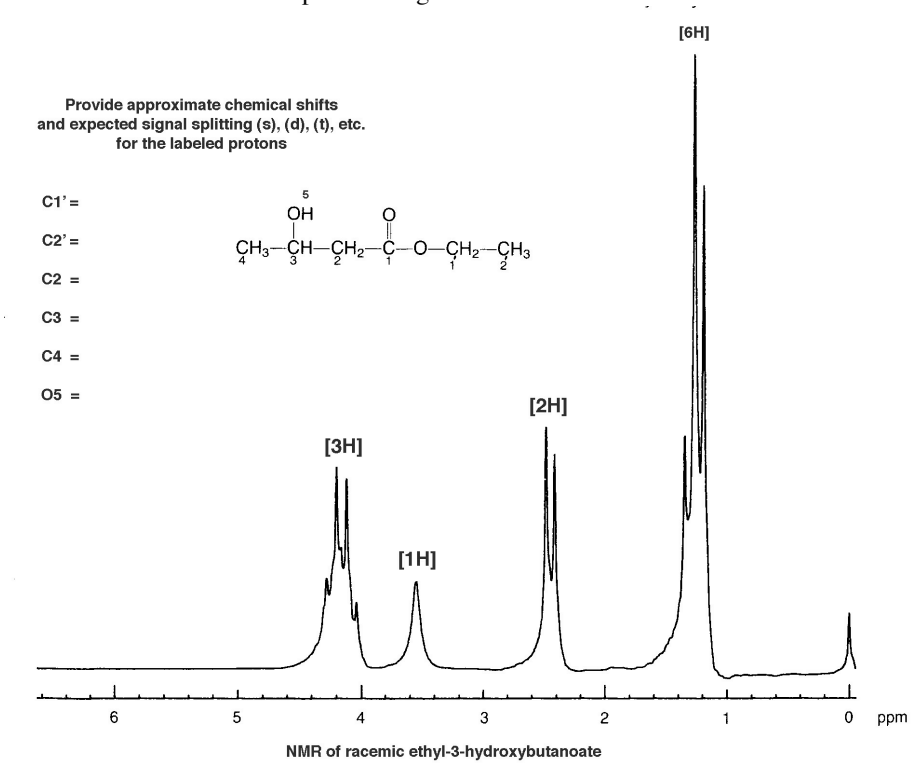
PART II: NMR, optical purity, enantiomeric excess
*(*Refer to the handouts and the following spectroscopy data.)

Crude product Method 1:

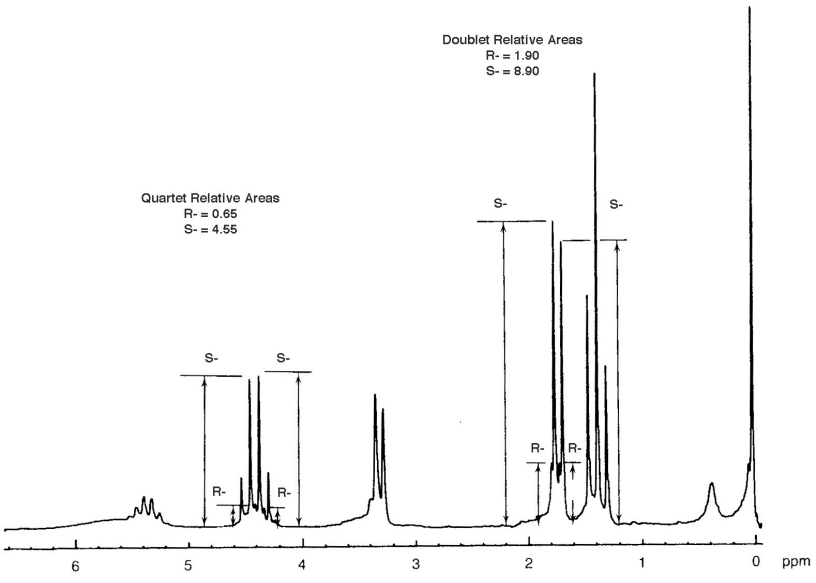
Identify 2 possible impurities in the sample and the peaks that support your id:



Purified product Method 1. Provide the proton assignments:



Using the following NMR data complete the Table below.



NMR of the enantiomeric excess product, plus chiral shift reagent: Method #3, Sodium Borohydride / Tartaric Acid
Chiral shift reagent = Tris[3-(heptafluoropropylhydroxymethylene)-(+)-camphorato] europium III

	[α]	Total % R-	Total % S-	Enantiomeric Excess: (%)	Abs. Config.
Product Method 3 (Quartet)					
Product Method 3 (Doublet)					
Product Method 3 AVERAGE					

Compare the two methods of determing the enantiomeric excess. Which do you think is a better experimental method? Briefly explain your choice.

PART III:

Compare and contrast the sodium borohydride/tartaric acid reduction with the yeast reduction. Which process is more "green"? Briefly explain the reasons for your choice. Include an evaluation of all waste (including aqueous), chemical yields, yield of a single enantiomer, safety, and energy efficiency.

What methods of chemical characterization can you use to characterize the presence of an enantiomerically pure compound? Do the ^{1}H NMR and ^{13}C NMR spectra of racemic mixtures look the same as those of an enantiomerically pure compound found in the mixture? Optical rotations? IR?

PART IV:
The following attached ^{1}H NMR was developed from a compiled sample of DVC student products. (Method 3) Complete the Table below showing yor calculations beneath the Table, and your measurements on the NMR.

	Total % R-	Total % S-	Enantiomeric Excess: (%)
Product (Quartet)			
Product (Triplet)			
Product AVERAGE			

Compare your results with those in Part II. Briefly explain what could account for any difference in the enantiomeric excess between the two results.