Experiment 5: Molecular Modeling and the Isomerization of (-)-Menthone

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Tuesday, March 27, 2007
7:42 PM

Purpose: To perform an acid-catalyzed isomerization and measure optical rotation of (-)-menthone and (+)isomenthone to calculate the energies of (-)-menthone and (+)-isomenthone.

Procedure:

1. Syringe 2.0mL of (-)menthone and pour into 50mL round bottom flask
2. Add 1-2 boiling stones
3. Add 10mL of glacial acetic acid
4. Add 10mL 1M HCL
5. Attack reflux condenser that is connected to circulating cold water. Place assembly in heating mantle
6. Heat mixture at reflux for 30 min.
7. Pour into 125mL Erlenmyer flask.
8. Cool in ice bath
9. Add NaOH until pH is 10 or more.
10. Pour into 125mL separatory funnel.
11. Extract with 10mL dichloromethane.
12. Save dichloromethane(bottom) layer and extract top layer with 10mL more dichloromethane
13. Put the 2 dichloromethane layers in 50mL Erlenmeyer flask and dry with CaCl
14. Measure weight of 125mL filter flask
15. Gravity filter solution into filter flask
16. Rinse CaCl with 1-2mL dichloromethane
17. Evaporate to remove dichloromethane.

18. Weigh filter flask again
19. Dilute 1 drop of product with 1-2mL of methanol.
20. Weigh 10mL volumetric flask
21. Transfer product to to flask and weigh
22. Bring volume to 10mL by adding ethanol
23. Stopper
24. Use polarimeter to measure optical rotation

25. Perform GC or HPLC

26. Calculate energies of (-)-menthone and (+)-isomenthone using Wavefunction Spartan or Chem3D.

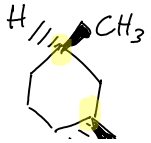
Prelab:

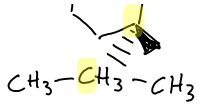
Compound	Hazards
Glacial acetic acid	Corrosive
(-)-menthone	Do not breath, avoid contact with skin/eyes
(+)-isomenthone	Do not breath, avoid contact with skin/eyes
Ethanol	Flammable

27. Specific rotation is the observed angle of optical rotation when plane-polarized light is passed through a sample with a path length of 1 decimeter and a sample concentration of 1 gram per 1 milliliter. Observed rotation depends on pathlength (l) of the cell, the concentration of the sample (c) and the specific rotation for the pure enantiomer of the compound. To summarize, specific rotation is defined by specific units while observed rotation defined by the experimentally used units. Observed rotation can be calculated into specific rotation.

28. (-)-menthone = (2S,5R-2)-Isopropyl-5-menthylcyclohexanone
(+)-isomenthone =(2R,5S)-2-Isopropyl-5-methylcycloexanone

I have highlighted the stereocenters that change their R/S notation during the reaction below:





This will not result in a racemization because there is no internal plane of symmetry.

29. (-)-menthone and (+)-isomenthone are diastereomers.