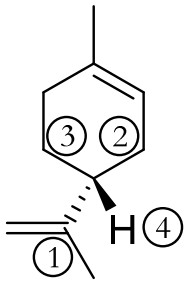
Experiment 9

Isolation of Limonene: Using Liquid Carbon Dioxide as a Solvent

Study Questions

1) (+)-Limonene is the (R)-enantiomer. Draw the structure of this enantiomer indicating the proper stereochemistry at the stereocenter of the molecule.
Answer:



2) You isolate 5 g of limonene and place it in a polarimeter cell with a total volume of 30 mL and a path length of 1.2 dm. You observe a rotation of 2.48º. The sample's NMR spectrum shows pure limonene with no other contaminants.
   a) What is the specific rotation of this sample?

      Answer:

$$[\alpha] = \frac{\alpha}{l \cdot c}$$

$$[\alpha] = \frac{2.48}{(1.2\ \text{dm}) \cdot \left({}^{5\text{g}}/_{30\ \text{mL}}\right)}$$

$$[\alpha] = 12.4º$$

   b) What is the enantiomeric excess of this sample, and which enantiomer is in excess? What percent of each enantiomer makes up this sample?

      Answer: Since the sample's rotation is positive, the dominant enantiomer must be (+)-limonene.

$$[\alpha]_{sample} = (ee \cdot [\alpha]_{dominant\ enantiomer})$$

$$12.4º = (ee \cdot 124º)$$

$$0.1 \text{ or } 10\% = ee$$

      The sample has 10% ee, so it is 45% (-)-limonene and 55% (+)-limonene.

3) What are the major diagnostic IR bands that you would expect to see in limonene? **Answer:** 3100-3000 (C–H stretch of an alkene), 3000-2850 (C–H stretch of an alkane), and 1680-1640 (C=C stretch).