EXPERIMENT #6: Dehydration of 2-Methylcyclohexanol

Background

By the time you do this experiment we will have covered elimination reactions in class. Please review your class notes and chapter seven of your text before coming to lab. You should already be able to predict the results of these experiments based on your knowledge of the concepts.

In the first week of this experiment you will dehydrate 2-methylcyclohexanol under acidic (i.e., E1) conditions and perform a distillation to isolate your cyclohexene products. The relative amounts of the formed products will be determined by gas chromatography. In the second week you will perform calculations using Spartan to help you design a reaction coordinate diagram and to further analyze the product ratios that you experimentally obtained.

Experimental

Week #1

1. Make a table of reactants and products in your lab notebook, include all weights, volumes, moles, and everything you had in your prelab table.
2. Measure 10.0 mL of 2-methylcyclohexanol and add it to a 50 mL round bottom flask.
3. CAREFULLY measure 2.5 mL of 85% phosphoric acid and add it to flask CAUTION: 85% phosphoric acid is a very strong, corrosive acid. Be very careful handling it.
4. Add a boiling stone, and set up a simple distillation using a sand bath to heat the round bottom flask.
5. Find your instructor or TA to approve your setup before you begin to heat your flask.
6. Heat the sand bath to ca. 170 ºC but do not let the temperature of the distillate to rise above 95 ºC.
7. Collect the product until the distillation becomes very slow and you have collected ca. 10 mL of product.
8. Transfer your product to a separatory funnel and wash the product first with water, then a 10% solution of sodium bicarbonate, and then with water once again.
9. Dry the product over sodium sulfate.
10. Obtain a percent yield and run gas chromatography on your product.
Week #2
1. In the Schupf Lab, using any programs that you wish, make a reaction coordinate diagram of the transformation of trans-2-methylcyclohexanol into all of the cyclohexene products that you observed in your product mixture. Include all relevant intermediates along your reaction pathways.
2. Obtain energies for the species in your reaction coordinate diagram using semi-empirical/AM1 calculations in Spartan [you may ignore the catalyst (acid) and the solvent in your calculations].
3. Obtain the energy of the transition state for the hydride shift.²
4. Have the instructor approve your diagram before you leave lab.

Week #1 Prelab
1. Read how to perform a simple distillation¹ and draw a sketch of a simple distillation setup.
2. Make a table of reactants and products (hint: there are three products). Be sure to include structure, name, molecular weight, boiling point, and density for each compound.
3. Draw all of the chair structures of 2-methylcyclohexanol

Report
In constructing your laboratory report, make sure to address the following:

Include a complete reaction mechanism. Be sure to use ChemDraw, and you probably will want to attach this as an appendix (i.e., on its own sheet of paper).

Based on your calculations would you have predicted that your reaction would have provided the observed the products and ratios? Why or why not?

References