Hydration of Cyclohexene

Pre-Lab Questions: (1) Orange colored bromine solution is added to a clear liquid compound and the resulting color is clear. Judging by this result, has a reaction occurred? (2) What does hydration mean? (3) Draw reactions for cyclohexene similar to Eq.1 & 2 on p65-66 in manual.

Procedure: ***Caution:*** Corrosive sulfuric acid. Use gloves and handle with care. If you get acid on skin, wash with abundant quantity of water and notify TA immediately.

Students should be familiar with background information in *742 Hydration 1-Hexene* in lab manual p65-76. Work in pairs and do all procedures in fume hoods as much as possible. In pre-lab discussion, TA should review all standard distillation and sep funnel procedures.

Reaction: To a 100 mL round bottom flask add a small stir bar and 5.0 mLs of 67% sulfuric acid solution which has been prepared by Lab Services*. Add 5 mL (4.1g; 0.05 mol) of cyclohexene. Obtain a stir plate, properly secure the flask, and place a distilling head (adapter) on top of the flask for added protection in case of 'bumping'. Stir the mixture vigorously for at least 5 minutes. Use no heat. Solution should change to a reddish-brown color.

Distillation: Add 60 mLs distilled water to the rb flask. Since the stir bar remains in the flask you don't need boiling chips. Get a flask heater and prepare a simple distillation apparatus (see figure in book), but do not start until your set-up has been personally checked by your TA for proper and secure connections. The distillation seems to work most efficiently when it reaches approximately 97°C. Start with a setting no higher than 6 on the flask heater and closely monitor the temperature to avoid overheating. Collect 40 mLs of distillate. This will possibly take ½ to 1 hour after the temperature has reached 97°C, but for safety you must not ignore the distillation. Do not let it go dry. ***When finished, remove the stir bar from rb flask and return it to your TA.***

Boiling Points: Cyclohexene: 83 °C
Cyclohexanol: 161 °C

Extraction: First, refer to previous experiments and ask TA if you are not sure how to do extraction safely, including how to hold, shake, and vent the funnel. Transfer distillate to a 125 mL separatory funnel. Wash the distillate with 20 mL of brine (saturated NaCl) solution by adding it to the distillate in the sep funnel. This is called 'salting out'. Extract your product twice using 10 mL portions of diethyl ether. ***Caution; ether is very flammable.*** Save all your solutions until you are finished. Collect both organic layers in a small beaker, dry with anhydrous sodium sulfate, and then gravity filter or decant into a 125 mL erlenmeyer flask to remove remaining solids. In the fume hood, heat organic layer in a low temperature (<60 °C) water bath to evaporate solvent from product. Place aqueous layer in properly labeled waste.

Confirmation of cyclohexanol: Students should do Bromine Test for saturation on small amounts of both product and starting material (see pg. 103 in lab manual).

Post Lab Questions: (1) Explain results of bromine tests. (2) Why does product distill at 97 °C if boiling points of cyclohexene and cyclohexanol are 83 °C and 161 °C respectively?

Reagents: Cyclohexene (extremely flammable)
Cyclohexanol (product)
67% Sulfuric Acid (corrosive)
Sodium Sulfate
Diethyl Ether (extremely flammable)
2% Bromine (corrosive) in cyclohexane