Chem 224 Exp. # 6 Alcohol Dehydration FS2017

Dehydration of cyclohexanol to cyclohexene. Separation of the product of a reaction mixture by fractional distillation; boiling point and refractive index determination of the liquid component.*
Reading Assignment: MTOL, pp. 72-85 (distillation theory), 49-52 (refractive index).
Also read **CER TECH-700, 701, 702**

This experiment demonstrates the dehydration of an alcohol to an alkene using a strong acid catalyst. The product is recovered by fractional distillation from the reaction mix. The product is characterized by GC, FTIR, RI and chemical tests.

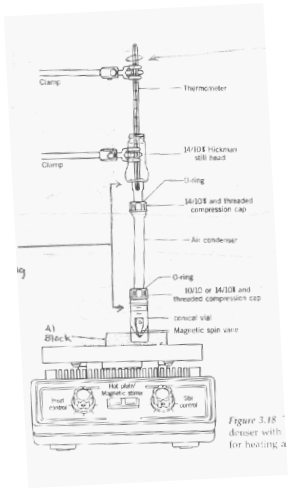

Figure 3.18 denser with for heating a

1. Preheat the hot plate and aluminum block at a heat setting of ~130-145 °C while you assemble your glassware.

2. Put together a fractional distillation set-up. Transfer the reactants to a 5-ml conical vial. The cyclohexanol should be weighed and the mass recorded to the nearest mg, since yield is based on its amount and small volumes cannot be measured accurately. Add your large magnetic stir bar (to regulate boiling and avoid bumping), and attach the air condenser and Hickman still head and thermometer as shown, applying grease to the joints. The column should be covered with Al foil then covered with cotton batting held in place with test tube holders. It is important that no glass be exposed from the conical vial to the base of the Hickman still. Regulate the heat so that a smooth slow distillation is attained.

Collect the cyclohexene in the BP range of 80-85 C corresponding to the cyclohexene product, BP=83 C. Record the temperature when distillate starts collecting. Also record highest temperature reached.

Continue the distillation until the temperature at the still head drops, indicating that no more product is distilling over and record the boiling range. Avoid excessive heating afterwards, which may cause decomposition and contamination of the distillate. Disassemble the apparatus while hot and store the components under the hood as they will smell bad. Brown still pot residue (strong acid!) goes in the mineral acid waste container.

3. If there are two layers to the distillate, first remove the lower water layer with a pipet and discard. If only a single milky layer is present, add Na2SO4 to dry your product. Decant the clear dry liquid into a preweighed (tared) vial and weigh to determine yield.

4. Run GC of your product to determine the amount of cyclohexanol & cyclohexene present. Comment on the outcome.

5. Run FTIR of the product (TA will run). Label peaks corresponding to cyclohexene and cyclohexanol or water (if visible) in your product.

6. Run chemical tests on standards and the product: 1% Br2 in CH2Cl2, 1% aq KMnO4 and Ce(NH4)2(NO3)6 using white ceramic well plates noting color changes for cyclohexanol, cyclohexene std. and product. Tabulate the results. Comment on the outcome.

7. Determine the refractive index of the product using a refractometer **(TECH 702, fig. 6)** and record your results. Record the temperature of the refractometer in order to correct the RI reading for temperature. Consult your handouts for proper operation of the refractometers in lab. Tabulate your results in your lab book. Record brand names of instruments used & any applicable operating parameters. Determine % error for corrected RI.