

Experimental Procedure:

A sand bath was set to just under 40 upon entering the lab. The round-bottomed (RB) flask was placed in 30 mL beaker and zeroed out on the mass balance scale. 2.0 grams (± 0.05 grams) of cyclohexanol was pipetted into RB flask on mass balance scale and about 0.5 mL of 85% phosphoric acid was then added to RB flask in the fume hood, thus creating heat from the reaction between the two. The RB flask was clamped securely to the ring stand with a small three-necked clamp as low on the neck of the flask as possible and a few boiling chips were added to the flask. A black plastic connector was attached to the flask and connected to the fractional distilling column with copper sponge packed not too loosely, but not too tightly. A white plastic connector was attached to connect the fractional distillation column to the distilling head and a white plastic thermometer adaptor was attached to the top of the distilling head. A thermometer was gently placed through the thermometer adaptor, lining up the top of the mercury in the thermometer with the bottom of the distilling pipe, and aluminum foil was gently wrapped around the fractional distillation column. A collection vial was held at a 45° angle pushed up under the distilling head using copper wire and immersed in a 100 mL beaker of ice water. The RB flask was lowered into the sand bath, and rapid distillation (drop every 1-2 seconds) took place with the temperature range being recorded. The placement of the collection vial pushed up on the distilling head and ensuring it was immersed in the ice water was crucial for this distillation as the formation of cyclohexene and water from cyclohexanol with phosphoric acid as the catalyst occurred in the form of steam. Once the distillation slowed, the RB flask was raised out of the sand bath and cooled. A “chaser” of 2 mL of toluene was added to the flask dropwise by removing the thermometer and pipetting in on top of the fractional distillation column. The thermometer was put back in its same spot and the RB flask was lowered into the sand bath again to ensure that all the cyclohexene was distilled. The solution was distilled until it reached 90 °C and then after 25 drops at that temperature, the RB flask was raised out of the sand bath and the collection vial was capped. To ensure that the distillation apparatus was cleaned and dried for the distillation later in the lab, the apparatus was rinsed with water, ethanol, and then acetone and dried with air in the hood. The fractional distillation column was dried using the vacuum in the hood. The contents of the vial was pipetted into a test tube (reaction tube was too small) and the vial was transferred rinsed with about 10 drops of toluene. Equal volume of the amount in the test tube of saturated aqueous sodium chloride was added to the test tube to create two layers (aqueous and organic). The two layers were mixed by pipetting the contents up and down vigorously numerous times. Once the layers were defined, the bottom layer (aqueous) was removed into a waste beaker and the organic layer was pipetted into a clean vial using a clean, dry pipet and a transfer rinse was done with 5 drops of toluene. A few spheres of calcium chloride were added to the vial with the organic layer and swirled until the spheres of calcium chloride didn't clump. After letting it sit for about 5 minutes, the dry organic layer was pipetted into the clean 5 mL RB flask and a transfer rinse again was done with toluene in the vial. The distillation apparatus was set up again the same as before except with the clean vial labeled 'waste'. Once the temperature reached the BP of cyclohexene (± 3 °C), the 'waste' vial was replaced with a new, clean, pre-weighed vial and distillate was collected until the temperature rose sharply, where the 'waste' was placed on again and stayed on until the distillation was complete. The vial with cyclohexene was capped and weighed on the mass balance and

the mass was recorded. The sample then underwent gas chromatography with help of the TA using 0.0002 mL of the cyclohexene to analyze and the purity and percent yield was calculated from the chromatograph. Chemical tests of bromine in dichloromethane and potassium permanganate were conducted using between 0.3-0.5 mL of cyclohexene and cyclohexane in each test tube and observing what happened in each test tube.