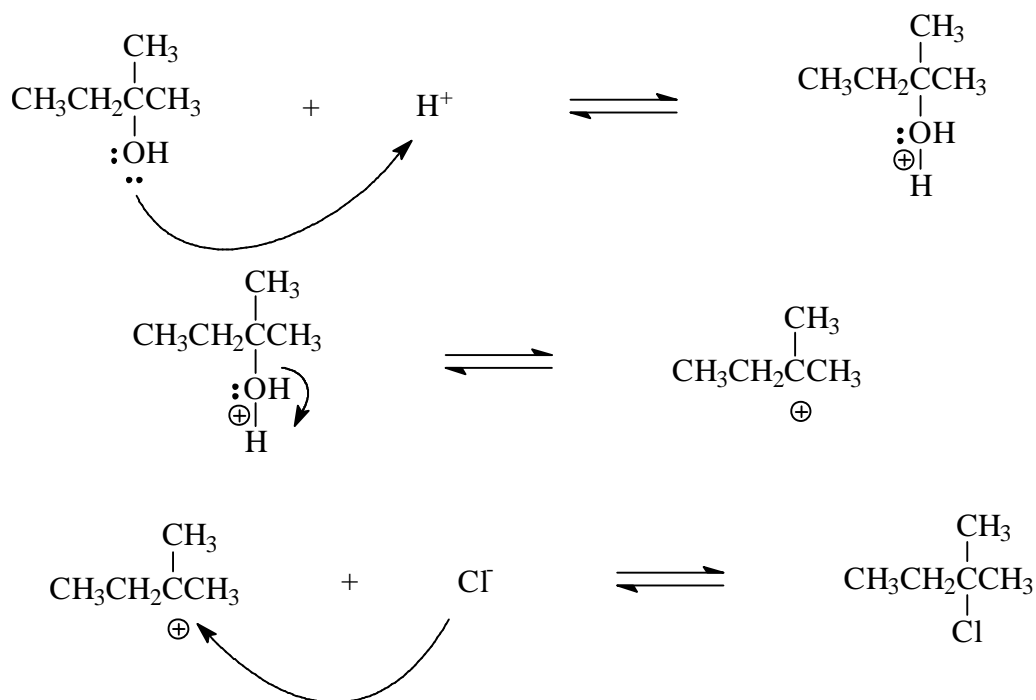


Organic Chemistry Laboratory #5 - Preparation of *t*-Pentyl Chloride

Alkyl chlorides and bromides can be readily prepared from the alcohol by reaction with hydrochloric acid and hydrobromic acid respectively. The reaction proceeds as shown below.



	<i>t</i> -Pentyl Alcohol	HCl	<i>t</i> -Pentyl Chloride
MW:	?? g/mol		?? g/mol
density:	?? g/mL		?? g/mL
mols:	?? mol		?? mol
amount:	?? mL	?? mL	?? mL
properties	b.p. ?? °C		b.p. ?? °C

Pre-lab:

This pre-lab assignment is to be done before lab and turned in to your instructor before beginning. Using a CRC Handbook or an Aldrich catalog, look up the missing information from the table above. A table like this one should appear in your lab notebook. Read over the procedure and calculate the theoretical yield of *t*-pentyl chloride. Provide the IUPAC names of *t*-pentyl alcohol and *t*-pentyl chloride

Procedure:

In a 125-mL separatory funnel, place 22 mL of *t*-pentyl alcohol and 50 mL of concentrated hydrochloric acid. Do not stopper the funnel. Gently swirl the mixture in the separatory funnel for about 1 minute. After this period of swirling, stopper the separatory funnel and carefully invert it. Without shaking the separatory funnel, immediately open the stopcock to release the pressure. Close the stopcock, shake the funnel several times, and again release the pressure through the stopcock. Shake the funnel for 2-3 minutes, with occasional venting. Allow the mixture to stand in the separatory funnel until the two layers have completely separated. Which layer contains the alkyl halide? Separate the layers.

The operations in this paragraph should be done as rapidly as possible since the *t*-pentyl chloride is unstable in water and sodium bicarbonate solution. Wash (swirl and shake) the organic layer with one 25-mL portion of water. Again, separate the layers and discard the aqueous phase after making certain that the proper layer has been saved. Wash the organic layer with a 25-mL portion of 5% aqueous sodium bicarbonate. Gently swirl the funnel (unstoppered) until the contents are thoroughly mixed. Stopper the funnel, and carefully invert it. Release the excess pressure through the stopcock. Gently shake the separatory funnel, with frequent release of pressure. Following this, vigorously shake the funnel, again with release of pressure, for about 1 minute. Allow the layers to separate, and drain the lower aqueous bicarbonate layer. Wash the organic layer with one 25-mL portion of water, and drain the lower aqueous layer.

Transfer the organic layer to a small dry Erlenmeyer flask. Pour it from the top of the separatory funnel. Dry the crude *t*-pentyl chloride over anhydrous calcium chloride until it is clear. Swirl the alkyl halide and the drying agent to aid the drying. Decant the **clear** material into a small **dry** distilling flask. Add a boiling stone and distill the crude *t*-pentyl chloride in a **dry** apparatus using a heating mantle. Collect the pure *t*-pentyl chloride in a receiver, cooled in ice water. Collect the material that boils between 79 and 84°C. Weigh the product and calculate the percentage yield. You should also obtain the refractive index for your sample. Submit the sample in a labeled vial to your instructor.

Questions:

1. Aqueous sodium bicarbonate was used to wash the crude *t*-pentyl chloride. Why would it be undesirable to wash with sodium hydroxide?
2. Some 2-methyl-2-butene may be produced in the reaction as a byproduct. Give a mechanism for its production. How can it be removed during purification?
3. How is the unreacted *t*-pentyl alcohol removed in this experiment? Look up the solubility of the alcohol and the alkyl halide in water.
4. Why must the crude alkyl halide be dried carefully with calcium chloride before the final distillation?

Preparation of *t*-Pentyl Chloride

Name: _____

Lab section: _____

Date: _____

DATA

t-pentyl alcohol. _____ mL

t-pentyl chloride _____ mL

Theoretical yield _____ mL

Percent yield _____ %

Refractive index _____

Temperature _____ °C