# Running a Reaction

Once you have mastered the techniques of organic chemistry, you will use these to run reactions in which one organic compound is converted to another. Sometimes in the rush to follow the instructions and finish the reaction, it is easy to lose sight of the overall process. It is important to recognize what you are trying to accomplish with each procedure, and breaking up the process into constituent steps can be helpful.

Step 1 – Plan the reaction.

Although the reactions you will run have already been planned, you should be aware of what decisions have to be made when you start from scratch. First, you must decided what the starting material(s) and all reagents will be. Then you must choose the scale of the reaction – how much of the starting material will be used (reactions can be run on a 10 mg scale, 50 mg scale, 100 mg scale, 200 mg scale, etc). I have tried to avoid wasting chemicals by choosing the smallest scale that would give enough product to be easily handled. The equivalents of other reagents are then determined, and the needed amounts are calculated.

The conditions of the reaction, such as solvent, temperature, and protection from moisture must then be decided. This process involves some educated guesses. The reactions that you will run have been optimized (hopefully) to give good results – if you were starting from scratch, it would probably take some trial and error to hit on the best set of conditions for the reaction. *You should have read the lab, written out your introduction, and filled out the reagents table before you come to lab.* 

Step 2 – Obtain and purify the reagents; prepare glassware.

All of the reagents that you will need have been ordered from chemical companies, and should be available for your use. Most of them are pure enough to use straight from the bottle – a few I have already purified before the lab starts. Count you blessings! In the real world, this can add quite a bit of time to the process.

With some water-sensitive reagents, glassware must be dried before using it.

Step 3 – Set up and run the reaction.

This is generally where you will start when you arrive in the lab. You will need to set up the reaction equipment and measure the chemicals into a reaction vessel (usually a conical vial or a round bottom flask). It is important to get an accurate mass of the organic starting material (how much you actually used, not how much you were told to use) so that you can calculate the % yield at the end – this can usually be accomplished by adding the limiting reagent first, on a balance.

All of reactions that we will run are quite rapid, relatively speaking – it is common for organic reactions to require up to 24 hours or more to go to completion. While the reaction is running, review the rest of the procedure and work on the questions that go with each lab. You will usually be told how long to let the reaction run, but it you didn't know, you could usually monitor the amount of starting material and product by TLC. Very few reactions go all of the way to completion, so when no more product seems to be forming, you're done.

#### Step 4 – Quench the reaction.

If you are using highly reactive reagents, it is a good idea to quench them (inactivate them) before proceeding further. This is often done by adding water. Not all reactions have a quenching step.

### Step 5 – Isolate the product.

When the reaction is complete (and quenched if necessary), the next task is to separate or isolate the desired product from left over starting material, excess reagents, byproducts, and finally the solvent. This step may also be referred to as "working up the reaction." A number of different techniques may be useful:

- extraction removal of the product from the reaction solution into a new solution
- washing removal of an impurity from a solution containing the product
- filtration removal of a solid from a solution, either the product or an impurity
- drying removal of water from a solution containing the product
- rotary evaporation removal of the solvent from a solution containing the product
- column chromatography may be used to both isolate and purify a compound

# Step 6 – Purify the product

Once you have separated the product from the reaction mixture, it will usually still have small amounts of other chemicals mixed in. Purification techniques can be used to remove these unwanted impurities, although in some cases, products will be characterized without a purification step. The following techniques are common:

- recrystallization the most common procedure for purifying solids
- distillation often used to purify liquids
- column chromatography a very effective purification technique, but it takes a lot of time and solvents, so we will only use it a few times during the course

# Step 7 – Characterize the product.

Once you have purified the product, the final step is to confirm its identity, determine the purity, and calculate the yield. All characterization information should be included in the conclusion.

In order to calculate the yield, you will need an accurate final weight of the product. You should obtain this before removing any of the material for other characterization procedures. Use a scale which is accurate to 0.001 grams, and make sure the product is completely dry and cool before weighing it.

To determine the identity and purity of the compound, you can use a variety of techniques.

- The physical characteristics of the product, such as its state of matter, appearance, color, and odor, can be compared to those of the authentic compound.
- If the compound is UV active, you can use TLC to show whether the compound is pure by looking for other spots. If you have an authentic sample to use for comparison, you can also verify the identity; if not, at least you can verify that it is different from the starting materials and has the expected polarity.
- Melting points give information about both purity and identity, while boiling points are more useful for identity.
- IR spectroscopy gives important information about the functional groups present in the sample, which can again provide information about both purity and identity.
- NMR spectroscopy is very useful in establishing the identity of a product, and may also detect impurities if they are present in large amounts.

Note: If your reaction fails (if you determine that you have not obtained any product, or if you lose your reaction due to a major spill, etc) you will not be penalized. However, you must still complete all of the steps of the lab. Ask permission to work with another student whose reaction did give the product in order to isolate and purify the product. Use that student's product mass to calculate a % yield. Then use a portion of their product to do all of the characterization steps on your own (IR, mp, etc). In your notebook, record what really happened and any explanation that you have, then what you learned about the other student's product.