4.2: WHY DID MY EXPERIMENT FAIL TO WORk THE WAY I EXPECTED?

Experiments fail all the time to work in the ways we expect, especially in a teaching lab. Countless students each week in numerous colleges and universities do not get their product.[13] Why not?

13 There are no overall statistics as far as I am aware, and this is simply based on my experience, but usually in a class of 20 students, anywhere from 1-5 of them have experiments fail in any given week, this often happens for unknown reasons. Happily, only once in a while is a student so unlucky to have two failed experiments in a given quarter.

The answer is complex, but we will try to go through some common reasons experiments fail in the teaching labs.

Previously, we said that the outcome of the experiment is set in phase 1, and that very often the purity of the product is set in phase 2. Let us examine these statements closer. What can be the reason for a failed experiment, an experiment in which no product is obtained?

Is it because the chemical theories underlying the experiment are fundamentally wrong? Or is it because the procedure given to the students contains a fatal error? If either of these possibilities were true, we would expect the experiment to fail for the entire class because these are fundamental flaws in the design of the experiment and should apply to everyone equally. If, however, we see that the experiment works for some students and not others, we must seek an explanation at the level of the individual, i.e., we must assume that the different outcomes reflect different choices that individual students made.

If absolutely no product is obtained, the mistake or error can usually be traced back to stage 1. Very typical mistakes (that the student might not have been aware of) are calculation errors and/or errors in measuring out reactants. A misplaced decimal point, e.g. 0.1 g versus 0.01 g is easy to overlook. Other common errors are improper heating during the reaction stage, and letting the reaction continue for a wrong period of time. Another error that we see quite often, is use of the wrong reagents. Chemical names are strange, and it is easy to overlook subtle differences such as “conc.” versus “6M” sulfuric acid, or acetic anhydride versus acetic acid. The reagents in each pair sound very similar, but may react completely differently. Of course, with so many possibilities for error, it can be very difficult to backtrack and find an error after the experiment is complete.

Simple misunderstandings often occur, even with a very carefully written procedure, and a very careful TA. To give you an idea of the level of involvement required in designing lab manuals, all of the PSU organic chemistry labs have been piloted at least three times, by three different people. The manual is constantly updated and refined, and even after several terms, minor changes are still implemented. This means that people read and interpret differently. Something that might seem very clearly worded to you might be very confusing to someone else. In this case your TA will aid you in figuring out what has gone wrong.

Sadly, even when a reaction has been performed properly, it is possible for things to go wrong in stage 2, the work-up and purification. In fact, this is often a greater source of trouble because purification, by its very nature, produces two things: the desired (pure) material and the undesired contaminants. Unless one pays careful attention, one can easily toss out the purified product and hold on to the contaminants. Also, as you probably noticed in chapter 2, purification procedures can be complicated and there are many ways for them to go astray.

Getting two phases confused during a liquid-liquid extraction, for example, or failing to get crystal formation during recrystallization, are examples of mistakes that can prevent you from obtaining product.