

## GUIDELINES FOR PREPARING 'SCHEMATIC DIAGRAM' OF WORK-UP AND PURIFICATION.

### Background

If you find that the procedure for carrying out the synthesis of an organic compound seems like a blur to you and you don't understand what is happening at every stage in the process, welcome to the club. This is normal at this time. Most novices in the laboratory are unclear about what the various ingredients in the procedure are for, and whether they should be called reactants, catalysts, solvents, etc. In addition, they have a shaky concept of what might be called 'material flow', which refers to where the different things end up when certain operations are carried out, such as separation of layers in a separatory funnel, distillation, filtration, etc.

The first page of the report form, previously discussed, is designed to gradually strengthen your understanding of the basic stages in most syntheses, as well as the names and functions of the various ingredients. To get you thinking about material flow, there will be a question for every synthesis report that asks you for a schematic diagram of the work-up and purification procedure. This is a type of flow chart that starts with all of the things present in the crude reaction mixture at the beginning of work-up, and shows where everything eventually ends up during work-up and purification. After you have done this a few times, you will begin to feel a lot more confident that you understand what you are doing in the lab.

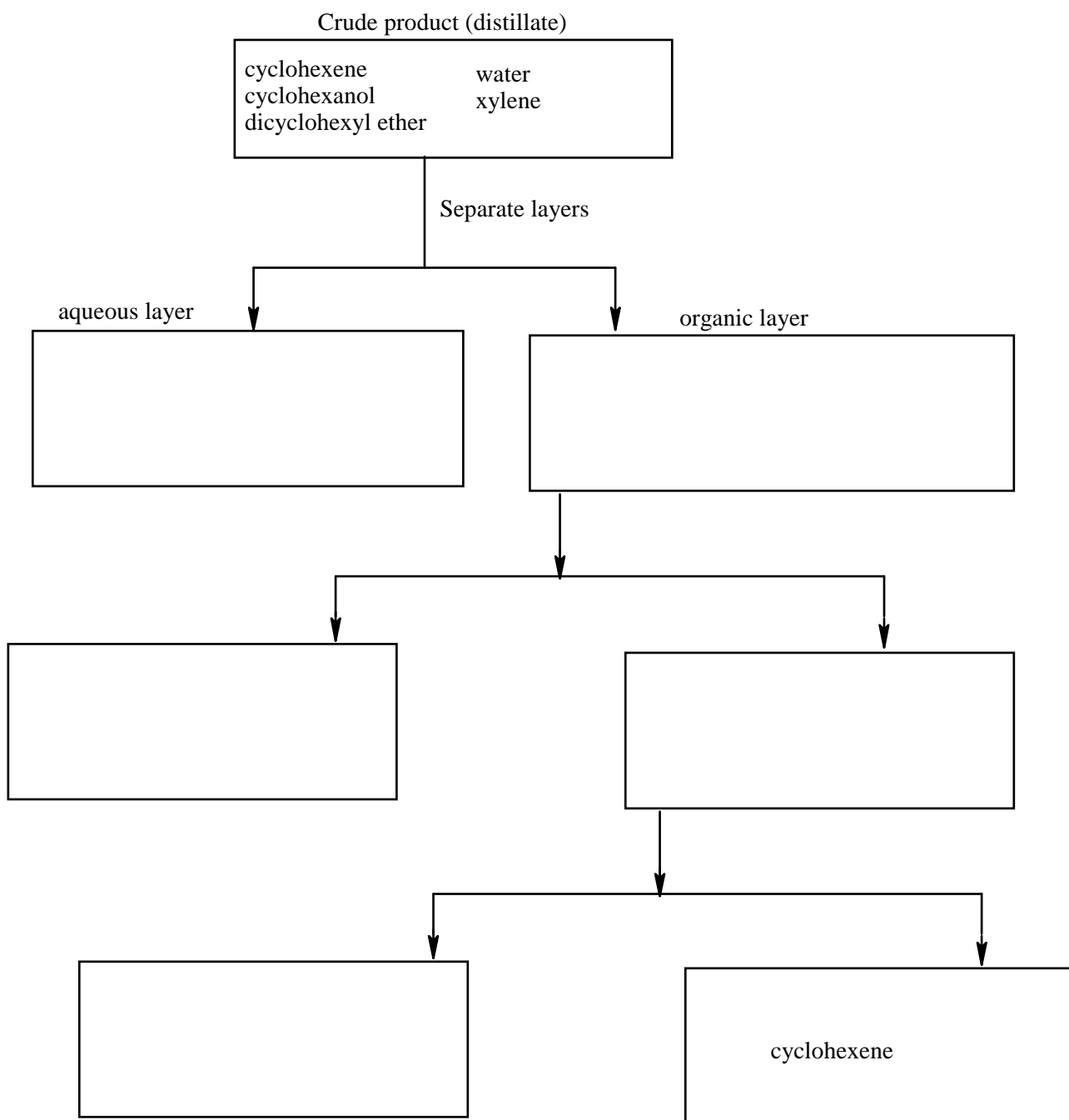
### Preparing the diagram (flow chart)

One straightforward way of preparing the diagram is illustrated on the next page with the sequence of connected boxes. This particular diagram is appropriate for the cyclohexene synthesis, and you can simply fill in the information and turn it in with your report. The first steps are done for you. Note that the top box contains a list of the materials present, and at the line to the next box, you indicate some operation that you carried out. Sometimes the operation involves a separation process, such as filtration or separation of layers with a separatory funnel; then the connecting line will branch to two boxes, which are identified and the contents noted. At the end of the whole process will be a box representing the compound you are preparing. An inspection of the diagram should reveal what happened to everything else that was there at the start.

An important consideration in deciding where things go during a separation process is **solubility differences**. The concept of '**like dissolves like**' applies, which means that polar compounds tend to dissolve in polar solvents, and non-polar compounds in non-polar solvents. **Most organic compounds are relatively insoluble in water and soluble in organic solvents.** Exceptions are low-molecular-weight polar compounds such as alcohols, ketones, etc., which tend to be water soluble. **Ionic compounds tend to be soluble in water and insoluble in organic solvents.** Keep this in mind when you are deciding where things go during a separation process. Ionic compounds are also relatively non-volatile, which is the reason that drinking water can be prepared from sea water by distilling off the water.

Also keep in mind that chemical changes are sometimes involved during work-up. The reactions should be shown in the diagram. You then have to deal with these reaction products (and usually an excess of some reagent used) and show where they end up.

**Schematic diagram for cyclohexene work-up and purification**



Balanced equations for any reactions occurring during work-up: