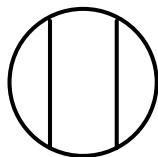


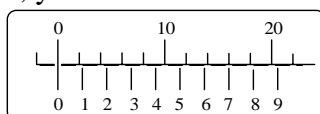
POLARIMETRY FOR CHEMISTRY 355:

Use of the manual polarimeters.

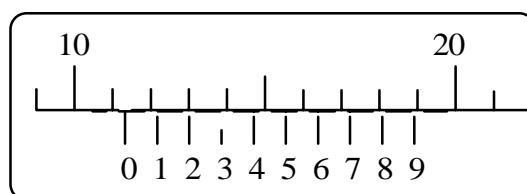
1. Turn on the switch on the right side of the polarimeter. Allow the lamp to warm up for 10 minutes. The light should be seen to be very bright inside. Do this and then prepare your sample.
2. Dissolve your sample in an appropriate solvent. The concentration you will use will depend on the specific rotation of your compound. For most samples, a range of 0.1-0.4 g/mL is appropriate, with the lower end for high rotating samples and the upper end for low rotators. As a general rule, one would prepare a higher concentration sample first and then dilute it by half for a second determination. This will give you better confidence in your rotation values. For very low rotating samples ($|\alpha| < 10^\circ$) a concentration of 0.5 g/mL or higher may be prepared, but it is generally quite difficult to reach these concentrations without saturating the solvent. It is easiest to prepare 10 mL of sample in a graduated cylinder, so that you can fill the cell and easily prepare a 1:2 dilution.
3. Pour the sample into the polarimeter cell. Use the 5.5 mL, 1 decimeter cells and fill to the point that there is solvent in the necks. Place the Teflon plugs in place. Check to see that there are no bubbles in the light path. Check this by tilting the cell lengthwise to see if bubbles come out. Then look down the light path and inspect for bubbles. You should be able to see clearly through the cell.
4. Look into the polarimeter eyepiece without a sample inside. Looking into the eyepiece horizontally with the bench, you should see a circle with a bar in the center that looks like the following.



5. Looking downward into the eyepiece, you should see a dial that looks like the following.



6. First set the dial to zero by turning the knob on the right until the zeros on the dial line up as above. From here, as you turn the dial, the circle and bar will rotate and the relative brightness of the bar and edges of the circle will change. As you rotate positive, the bar will dim relative to the edges and vice versa when you rotate negative. Observe this and try to get the bar and edges to match. Then observe the dial. It should be at or very near zero. Make sure you can do this before observing the sample.
7. Open the cover of the polarimeter and place the sample in the compartment so that the cell cleanly seats at the bottom. Close the cover. Turn the dial until you can get the bar and edges to match. The integer rotation is read from the top side of the dial where the lower zero line crosses. The tenths are read from the lower side where one of the lower lines meets an upper line. For example:



8. This dial reads 11.2 degrees. The zero on the bottom is between 11 and 12 degrees and the lower line at 2 lines up with a line on the upper scale (13). You notice that this agrees with what might be estimated from the position of the zero mark. You can use this to check yourself. Again, if you are unsure, ask.
9. When you have finished with your measurement, turn off the polarimeter if everyone is finished with it.
10. Clean out the cell by rinsing twice with the same solvent you used for your sample, then twice with acetone and blow dry nitrogen (not air) through the cell until it is dry. Don't forget to clean off the stoppers as well.

Instructions for the Perkin-Elmer digital polarimeter.

1. Turn on the instrument by pressing the red power button.
2. Turn on the sodium lamp by pressing the Na button. Do not turn on the Hg lamp unless you need it – the bulbs are rather expensive.
3. Make sure the yellow dial is set to Na 589, and the slit is set to the large (o) width.
4. Set the integration time to 20 sec.
5. Allow the lamp to warm up for 15 minutes or so. Prepare your sample as described above. You will need to use the cells that have the wooden or plastic adapter rings on the ends.
6. Zero the instrument by pressing and holding the Zero button for five seconds. Wait for the instrument to read 0.000.
7. Open the cover, place your sample in the instrument and close the cover. Wait for the reading to stabilize. Give it at least 30 seconds. If the reading will not stabilize, it is usually due to light scattering from particulates or solubility problems. Filter your sample through a pipet packed with glass wool and try again. If the reading still won't stabilize, ask your instructor or the TA.
8. Remove your sample and turn off the instrument if everyone is finished with it.
9. Clean out the cell by rinsing twice with the same solvent you used for your sample, then twice with acetone and blow dry nitrogen (not air) through the cell until it is dry. Don't forget to clean off the stoppers as well.