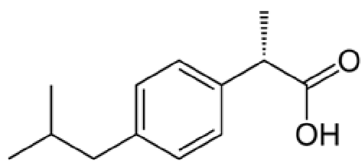
CHEM 333L
Organic Chemistry Laboratory
Revision 1.3

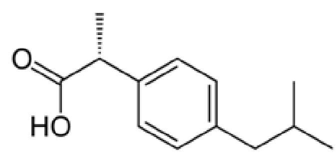
Polarimetry

In this short exercise we will measure the Optical Rotation of a solution of Sucrose, which will allow us to determine Sucrose's Specific Rotation [α]. The specific rotation is a characteristic physical property associated with optically active molecules. We compare our measured value with the accepted value as reported in the literature. Nest, we will determine the specific rotation of the S-(+)-Ibuprofen isolated in last week's laboratory. However, this sample may not be optically pure. The comparison of our "observed" specific rotation with the accepted value will allow us to determine the sample's Optical Purity.

One of the few physical properties that distinguish a pair of enantiomers, be they the enantiomers of Ibuprofen:

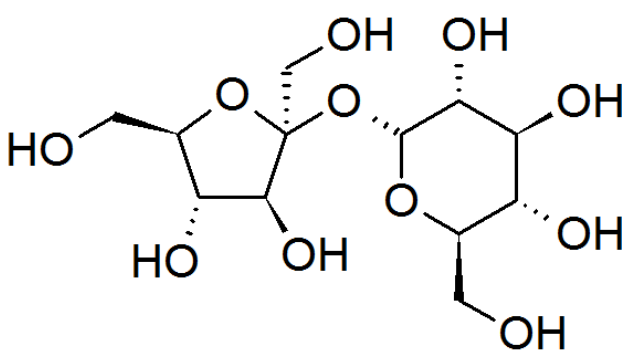


(S)-Ibuprofen



(R)-Ibuprofen

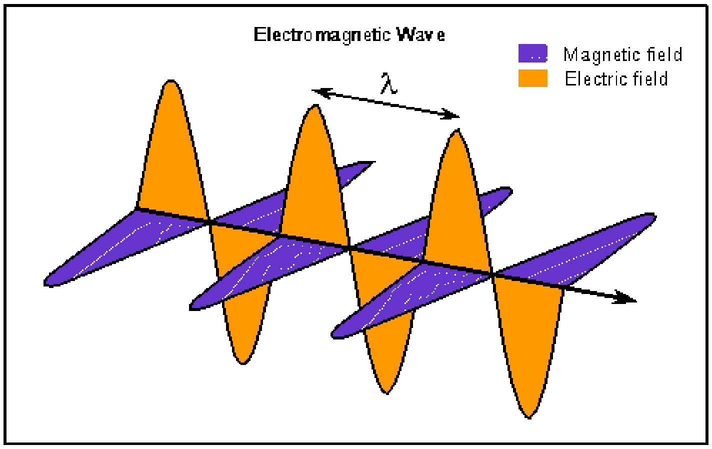
or the enantiomers of Sucrose:



β-(2*S*,3*S*,4*S*,5*R*)-fructofuranosyl-α-(1*R*,2*R*,3*S*,4*S*,5*R*)-glucopyranoside

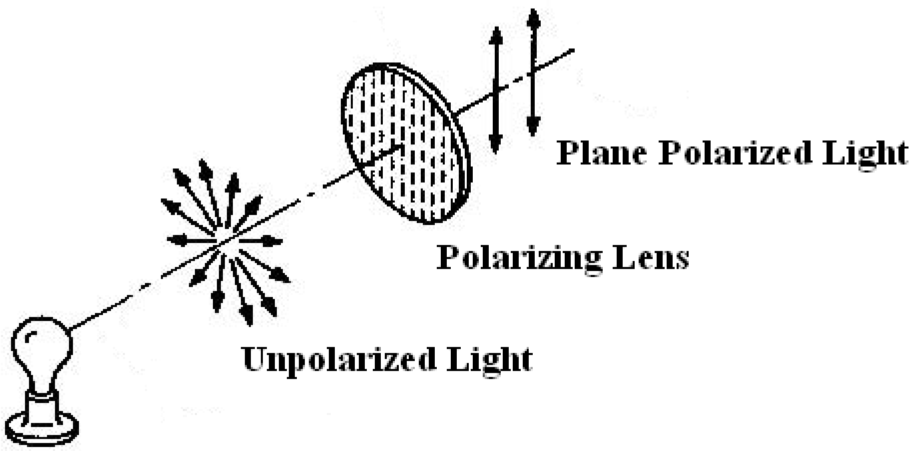
is the rotation of plane polarized light. With one enantiomer, plane polarized light will be rotated in a positive direction, while with the other, the rotation will be negative.

Electromagnetic radiation can be viewed as a self-propagating electromagnetic wave, with electric and magnetic fields oscillating perpendicular to each other:



Nick Strobel
http://www.astronomynotes.com/light/s3.htm

Plane polarized light is produced by passing the electromagnetic radiation through a polarizing lens. After passing through a polarizer, all the electric field vectors of the electromagnetic waves from a given light source lie in the same plane.

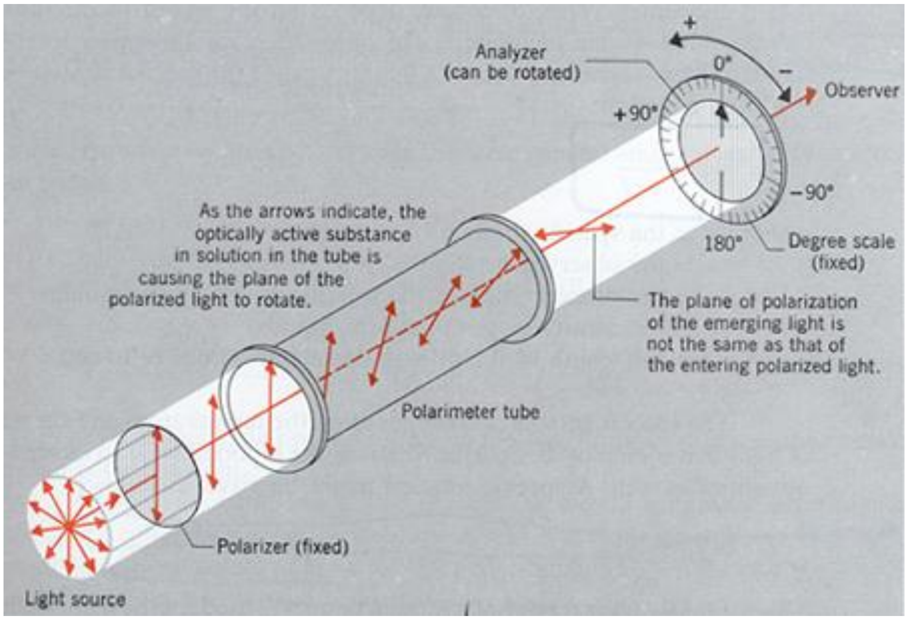


This plane polarized light can then be passed through a solution of an optically active compound.

> In a molecule, an electron is not free to oscillate equally in all directions; that is, its polarizability is anisotropic, which means different in different directions. When electrons in molecules oscillate in response to plane-polarized light, they generally tend, because of their anisotropic polarizability, to oscillate out of the plane of polarization. Because of its interaction with the oscillating electrons, the light has its electric and magnetic fields changed. Thus, when plane-polarized light interacts with a molecule, the plane of polarization rotates slightly. In a large collection of achiral molecules, however, for any orientation of a molecule that changes the plane of polarization of the light, there is apt to be another molecule with a mirror-image orientation which has the opposite effect. Consequently, when a beam of plane-polarized light is passed through such a compound, it emerges with the plane of polarization unchanged.

> For molecules such as one of the enantiomers of [an optically active compound], however, no such mirror-image orientation exist, and the plane of polarization of the light is usually measurably altered in it passage through the sample.
>
> Introduction to Organic Chemistry
> Andrew Steitwiesser, Jr. – Clayton H. Heathcock

The angle of rotation of the light α can then be measured by passing the light through a second polarizer.



Organic Chemistry, 2nd Ed.
T.W. Graham Solomons

The angle of rotation will be proportional to the ability of the compound to rotate plane polarized light, or Specific Rotation [α], the Length of the polarimeter cell l, and the Concentration of the optically active compound c:

$$\alpha = [\alpha]\, l\, c$$

The specific rotation is a characteristic property of a given optically active molecule. If the specific rotation of a compound is positive, the compound is said to be dextroratory, if negative, it is referred to as levorotatory. Formally, specific rotation has units of deg dm^{-1} cm^{3} g^{-1}. However, it is frequently reported simply in units of degrees.

Finally, for a sample that may be contaminated with the opposite enantiomer, the Optical Purity can be defined as:

$$\text{Optical Purity} = \frac{Obs.[\alpha]}{[\alpha]} \times 100$$

Thus, in this exercise, we will measure the specific rotation of an optically pure sample; Sucrose. Then, we will measure the optical purity of our previously isolated S-(+)-Ibuprofen. These measurements will be made with a Autopol III polarimeter manufactured by Rudolph Analytical.

This polarimeter automatically rotates the polarizing lenses and detects the point at which they are inline with the angle of rotation caused by the sample. It requires nothing more than to load the sample into the sample cell and to place the sample cell in the sample compartment. Everything else is done automatically.

Pre-Lab Questions

1. How many assymetric carbons are there in a molecule of naturally occurring Sucrose? What is the maximum number of stereoisomers that can exist for this compound?

2. Camphor has a specific rotation of $+44.26^{o}$. How many grams of Camphor are required to produce an angle of rotation of 35^{o} using a 10mL cell with a path length of 1 dm?

Procedure

Optical Activity of a Sucrose Solution

1. Each person will be assigned a Sucrose solution that is to be prepared between 5% and 20%. Prepare 100g of this solution by accurately weighing out both the Sucrose and the Water. Using a magnetic stir bar, allow this solution to mix for at least 15 minutes. Use the tabulated density values (see Appendix) to convert the concentration in wt% to a concentration in g/cm^3.

2. **Carefully** load a polarimeter cell with this solution (your instructor will demonstrate this) and measure the optical rotation of the solution. Be sure to determine the length of the polarimeter cell. Be sure to thoroughly rinse out the cell when you are done. **(The end plates for the cell are very sensitive and should only be cleaned with Kimwipes.)**

3. Determine the specific rotation of Sucrose.

4. $[\alpha]_D^{20} = +66.37^o$ for Sucrose. Calculate the percentage error in your determination.

Optical Purity of S-(+)-Ibuprofen Sample

1. Dissolve your sample of resolve Ibuprofen in 2 mL of Ethanol. Transfer this to a clean 5 mL Volumetric Flask. Bring the solution up to the flask's mark with additional Ethanol. Mix the solution until it is homogeneous. (You may need to collaborate with another student in order to have sufficient Ibuprofen to make a meaningful measurement. Consult with your laboratory instructor concerning this point.)

2. Load the sample into a polarimeter cell and measure the optical rotation of the solution. Be sure to determine the length of the polarimeter cell.

3. Determine the "observed" specific rotation of the sample.

4. Determine the optical purity of the sample.

Post Lab Questions

1. Is it possible to determine if a sample is (+) or (-) from a single angle of rotation measurement? Explain. If not, how might this determination be made?

2. In the discussion above, it is implied the analyzing, or second polarizing lens, in a polarimeter is rotated to match the rotation of the plane polarized light due to the sample, and thus produce a maximum intensity in the light passing through the polarimeter. In actual fact, things are arranged so that a minimum of light passes through the polarimeter. Why is this?

Appendix - Density of Aqueous Sugar Water Solutions

Wt%	density (g/mL) at 20^oC
0.0	0.998234
1.0	1.002120
2.0	1.006015
3.0	1.009934
4.0	1.013881
5.0	**1.017854**
6.0	1.021855
7.0	1.025885
8.0	1.029942
9.0	1.034029
10.0	**1.038143**
11.0	1.042288
12.0	1.046462
13.0	1.050665
14.0	1.054900
15.0	**1.059165**
16.0	1.063460
17.0	1.067789
18.0	1.072147
19.0	1.076537
20.0	**1.080959**