## Polarimeter Experiment:

What is the aim of polarimeter experiment?

A polarimeter is a device that measures the rotation of linearly polarized light by an optically active sample. This is of interest to organic chemists because it enables differentiation between optically active stereoisomers, i.e., enantiomers.

What are the applications of polarimeter?

Polarimeters are used in a wide range of applications, from the determination of the purity and concentration of ingredients in pharmaceuticals to the maturity testing of agri cultural products to the measurement of the sugar con tent in beverages and candies.

Why do we use sodium light in polarimeter?

A polarimeter is defined as a scientific instrument that is used for measuring the angle of rotation caused by the passing of polarized light through an optically active substance. For this sodium light is used because it produces monochromatic light and the energy output is high.

What is angle of rotation in polarimeter?

The point at which it tilts is called the "angle of rotation." It is the basic value measured with a polarimeter. The angle of rotation changes depending on the concentration of the liquid sample, the length of the observation tube, the temperature, and the measurement wavelength.

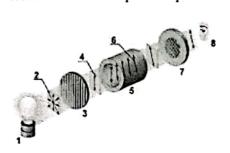
What is the function of a polarizer in a polarimeter?

A polarizer is a device through which only light waves oscillating in a single plane may pass. A polarimeter is an instrument used to determine the angle through which plane-polarized light has been rotated by a given sample. You will have the opportunity to use a polarimeter in the laboratory component of the course.

Is optical rotation a physical property?

Fortunately, there is one physical property in which enantiomers differ: their ability to rotate plane-polarized light. ... The ability of a solution to rotate plane-polarized light in this fashion is called optical activity, and solutions which have this ability are said to be optically active.

What are the main parts of polarimeter?

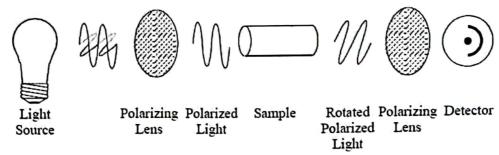


The polarimeter is made up of two Nicol prisms (the polarizer and analyzer). The polarizer is fixed and the analyzer can be rotated. The prisms may be thought of as slits S1 and S2. The light waves may be considered to correspond to waves in the string.

What is the principle of polarimeter?

Polarimetry measures the rotation of polarized light as it passes through an optically active fluid. The measured rotation can be used to calculate the value of solution concentrations; especially substances such as sugars, peptides and volatile oils.

### **Optical Polarimetry**



In a typical polarimetry experiment, monochromatic light is passed through the sample. A sodium lamp is usually used as the light source and the wavelength of its D line is 589.3 nm. The light provided by the source is not polarized so its electromagnetic waves oscillate in all planes perpendicular to the transverse axis. After passing through a polarizing lens (prism), the only light remaining is oscillating in one plane (plane polarized), whose angle is determined by the angle of the lens itself. This light is then passed through a solution of an optically active compound, which results in a rotation of the plane of oscillation. A second polarizing lens (prism) is used in conjunction with a detector to find the angle of rotation.

The magnitude of the rotation is not only determined by the intrinsic properties of the molecule, but also on the concentration, path length, and wavelength of light. These parameters should be familiar from use of a UV/Vis spectrometer. In order to standardize the optical rotations reported in the literature, a parameter has been defined as the specific rotation  $[\alpha]_1$ :

$$[\alpha]_{\lambda} = \frac{\alpha_{meas}}{1c}$$

The parameters are

 $\alpha_{meas}$  the rotation measured by the polarimeter in degrees

 $\lambda$  the wavelength used to measure the rotation in nanometers

l the path length in decimeters (usually this is 2 dm, note that 1 dm = 0.1 m or 10 cm)

c the concentration measured in g/mL

Normally  $[\alpha]_{\lambda}$  values are quoted in the literature at 20 °C. There is a slight temperature dependence on optical rotation, which can be corrected using:

$$[\alpha]_{\lambda}(20 \text{ °C}) = [\alpha]_{\lambda}(T)[1 + 0.0001(T - 20)]$$

The percent optical purity (x) of a material is the ratio of the measured specific rotation,  $[\alpha]_{\lambda(meas)}$ , to the standard pure specific rotation,  $[\alpha]_{\lambda(std)}$ , where  $x = [\alpha]_{\lambda(meas)}/[\alpha]_{\lambda(std)} *100$ . The percentage of the major enantiomer in a mixture of enantiomers can be calculated as:

% (major enantiomer) = 
$$x + (100 - x)/2$$

Thus, if a sample is an equal mixture of (+) and (-) enantiomers, the measured rotation is zero and x = 0, so the % (major enantiomer) = 50 %.

# **Polarimetry**

Polarimetry is an instrumental analytical method using rotation of polarized light by some substances as a measure of their concentration in a solution. The instrument used is called a polarimeter. When it is adapted for measuring quality of sugar the name saccharimeter is used. In both instruments it is the rotation of polarized light by a substance in a solution which is measured. Usually, it is only one instrument which has two interchangeable scales, one labelled in angular degrees <sup>0</sup>, the other in units <sup>0</sup>Z, named International Sugar Scale (I.S.S.).

### **Polarimeter**

Figure 1 shows a principle of a polarimeter set up and its main components together with their function. Unpolarized light from the light source is first polarized. This polarized light passes through a sample cell. If an optical active substance is in a sample tube, the plane of the polarized light waves is rotated. The rotation is noticed by looking through the analyser as a change in intensity of illumination. To reach the same illumination as was without an optical active sample the analyser must be turned around for an angle. Readings are taken in degrees (angle)  $\alpha$  or sugar degrees  ${}^{0}Z$ .

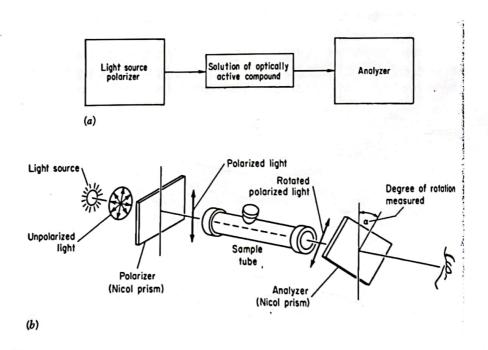


Figure 1: a) principle of a polarimeter set up

b) components of a polarimeter

Figure 2 shows polarizer (P) and analyzer (A) in a perpendicular position with one another. Both have the structure on a molecular level that polarize an unpolarized light. If they are in a position shown no light passes through analyzer. Polaroid films for example have such a molecular structure that filters (block) all planes of light vibrations except one. Nicol prisms are made of calcite (CaCO<sub>3</sub>) crystals, which show double refraction phenomenon. Both beams are polarized but only one is enough to be used for polarizing unpolarized light.

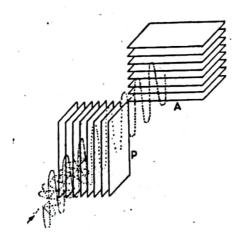


Figure 2: One of mutual positions of a polarizer and an analyzer

Figure 3 shows the passage of polarized light through a sample tube, which is positioned between polarizer and analyzer. The length of the tube is one of important parameters to be fixed if the measurements were comparable.

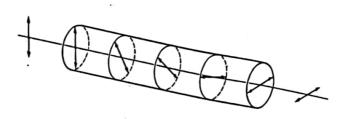


Figure 3: Rotation of polarized light plane by a sample with an optical active substance and the influence of the length of sample tube

### **Optical Rotation**

Certain compounds, mostly organic (notably those containing asymmetric carbon atoms) rotate the plane of polarized light. The phenomenon is called optical rotation and such substances optically active compounds.

Measuring angle of rotation the concentration of a substance in a solution is determined.

The measured angle of rotation depends upon many variables:

- The type or nature of sample (example: sugar solution)
- Concentration of the optical active components
- The length of the sample tube
- · The wavelength of the light source
- Temperature of the sample

We describe the nature of a sample by introducing the specific optical rotatory power (or specific rotation) of a substance, defined as

$$[\alpha]^{\Theta}_{\lambda} = \frac{\alpha}{\gamma J}$$

in SI units: rad m2 kg-1

(Notice:  $2\pi \text{ rad} = 360^{\circ} \text{ (deg of angle)}$ )

where  $\alpha$  is the angle of rotation in rad,  $\gamma$  is the mass concentration in kg/m³, and I is the length of the sample tube in m. Specific rotation is determined at a specified temperature  $\Theta$  (usually 20 °C) and a wavelength of light source (usually sodium lamp with its D line at 589 nm).

Some substances rotate the light to the right (or clockwise) as viewed looking towards the light source, we sign this rotation and  $\alpha$  as +, some to the left (or anticlockwise), signing  $\alpha$  as -.

In practical measurements readings are taken at different units:  $\alpha$  in  $^{0}$  (deg),  $\gamma$  in g/cm<sup>3</sup>, / in dm and so  $[\alpha]_{D}^{20^{\circ}}$  is usually tabulated in  $^{\circ}$  cm<sup>3</sup>/g dm.

Substance in a solution H <sub>2</sub> O solvent	Specific rotation
	$[\alpha]_D^{20^0}$
	[ 0 cm <sup>3</sup> /g dm ]
sucrose	+ 66.54
glucose	+ 52.74
fructose	- 93.78
maltose	+ 137.5
lactose	+ 55.3
dextrose	+ 194.8

For example:

Sucrose (cane sugar) solution  $[\alpha]_D^{20^0} = +66.54^{-0}/\text{dm}$  at a concentration of 1 g/cm<sup>3</sup>.

The Influence of the wavelength of a light source for sugar solutions is seen from the following table:

Description of the	Wavelength	Specific rotation
light source	[nm]	$[lpha]_D^{20^\circ}$ [ $^0$ cm $^3$ /g dm ]
Mercury, green	546.23	+ 78.4178
Sodium, yellow	589.44	+ 66.5885
HeNe Laser	632.99	+ 57.2144
Near Infrared (NIR)	882.60	+ 28.5462

Notice the high precision of specific rotation determined with modern polarimeters.

Temperature dependence of specific rotation is for sugar solutions as follows:

$$\alpha$$
 (t) =  $\alpha$  (20.0 °C)[1.0 - 0.000471 (t - 20.0)]

or calculated for some temperatures of a sucrose solution at some concentration:

ſ	Temperature	Rotation of a sucrose solution
	°C	$\alpha$
1		[ oangle deg ]
	20	40.000
L	21	39.981
	25	39.906

Notice the decrease of the rotation of sucrose solution with rising temperature. Also the effect of temperature is relatively small.

# Polarimetry of sugar solutions

Polarimetry is frequently used for determining the quality of sugar products. Measurements are made by polarimeters or saccharimeters with the scale in angle degrees (°) and sugar degrees (°Z). Angle of rotation depends linearly on concentration of sugar in the solution other parameters (temperature, light source, length of the tube) being the same.

Sugar industry with its International Commission for Uniform Methods of Sugar Analysis (ICUMSA) introduces International Sugar Scale (ISS) in  $^{\circ}$ Z units.

100.00 °Z units (sugar degrees) belong to Normal Sucrose Solution prepared from exactly 26.000 g of sucrose dissolved in pure water to 100 cm³. At 20 °C and D sodium lamp rotation for this solution in a tube of 200 mm will be

 $\alpha$  = +34.626 °. The ISS is linearly divided, i.e. a rotation of +17.313 °

(13 g/100 cm $^3$ ) equals to a reading of 50.00  $^{\circ}$ Z.

The 0 °Z point in ISS is fixed by the indication given by the saccharimeter for pure water.

Normal Sucrose Solution was used to calibrate and standardize polarimetric methods and instruments. Sugar solutions are not very stable and have to be renewed regularly.

Today quartz control plates are used as a standard for the calibration of polarimeters.

Interrelation between both scales is defined from a straight line (y = a.x) equation:

$$^{0}Z = \frac{100.00}{34.626}$$
.  $^{0}$  (deg) = 2.889.  $^{0}$  (deg)

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For further information see:

http://micro.magnet.fsu.edu/optics/lightandcolor/polarization.html (animation)

http://micro.magnet.fsu.edu/primer/java/scienceopticsu/polarizedlight/filters (animation)

http://www.oiml.org/publications/R/R014-e95.pdf (ICUMSA Int. Sugar Scale)

http://www.schmidt-haensch.com/v1/datenblatt\_gb/polarimetereng.pdf

(overview polarimeters, polarimetry for sugar industry)

http://www.topac.com/saccharomat.pdf (short overview technique)

http://www.opticalactivity.com/sugar.htm (polarimeters)

http://hyperphysics.phy-astr.gsu.edu/hbase/hframe.html (on polarization)

http://www.glenbrook.k12.il.us/gbssci/phys/Class/light/u1211e.html (polarization)

http://ts.nist.gov/ts/htdocs/230/232/232.htm (then SRM No. 917b and View Certificate of Analysis)

http://mineral.galleries.com/minerals/carbonat/calcite/calcite.htm

http://en.wikipedia.org/wiki/Calcite (double refraction crystal picture)

http://dl.clackamas.cc.or.us/ch106-07/optical.htm

#### Reference text books

 G.J. Shugar, J.T. Ballinger, Chemical Technicians' Ready Reference Handbook, McGraw-Hill, Inc. 1996, p. 448-454

2. D.P. Shomaker, Experimental Physical Chemistry, McGraw-Hill, 1989 p 728-729

 R.H. Petrucci, General Chemistry, Prentice Hall, International, Simon &Schuster/A Viacon Company, 1997 p. 875

 Official Journal of the European Communities, 1979, No 239/52, Method for determining quality of sugar p. 309