M9. Optical rotation of solutions. Measurement of the concentration of optically active substances by refractometric method and by using a polarimeter.

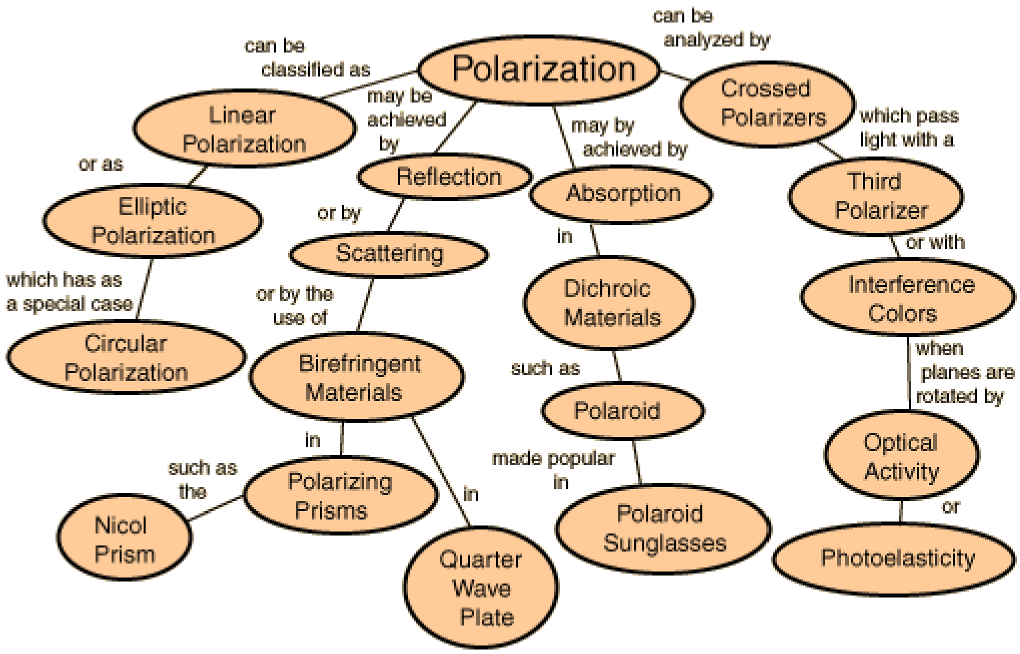
Problems to be prepared:

- Electric field [1] – Chap. 22-2
- Magnetic field [1] – Chap. 28-3
- Maxwell's equation [1] – Chap. 32-5
- Wave theory of light [1] - Chap. 33
- The phenomenon of polarization [1] – Chap 33-7. Ways to polarize the light.
- Polarizers. Prism Nicola. Phenomena occurring during the passage of light through a prism [1] – Chaps. 33-8 through 33-10
- Optically active substances.
- Rotation of the plane of polarization. Specific optical rotation.
- Reflection and refraction of light.
- Total internal reflection.
- Abbe Refractometer.

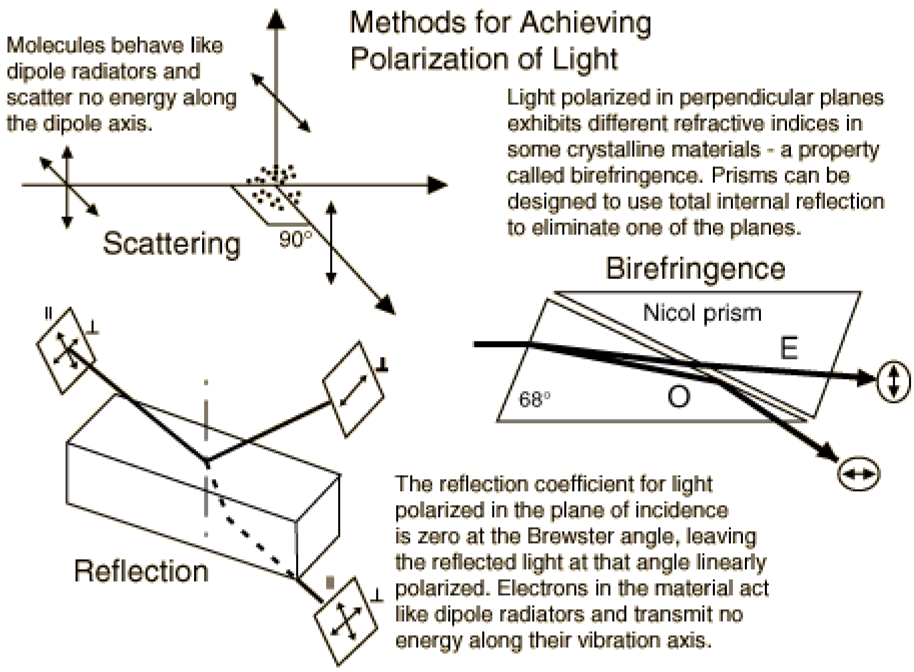
REFRACTOMETRY. Using a refractometer one can measure the refractive index of the tested liquid. Since the refractive index is directly proportional to the concentration of the substance, these two quantities can be interconnected and read simultaneously. The Abbe refractometer used during the exercises has two scales, one for reading the refractive index and second for reading the concentration of the test substance in %. This refractometer is rated for sucrose solutions.

ON-LINE laboratory. The aim of the refractometry animation is to determine the concentrations of a series of sugar solutions (Animation, part1), which will then be further polarimetrically tested (Animation, part2).

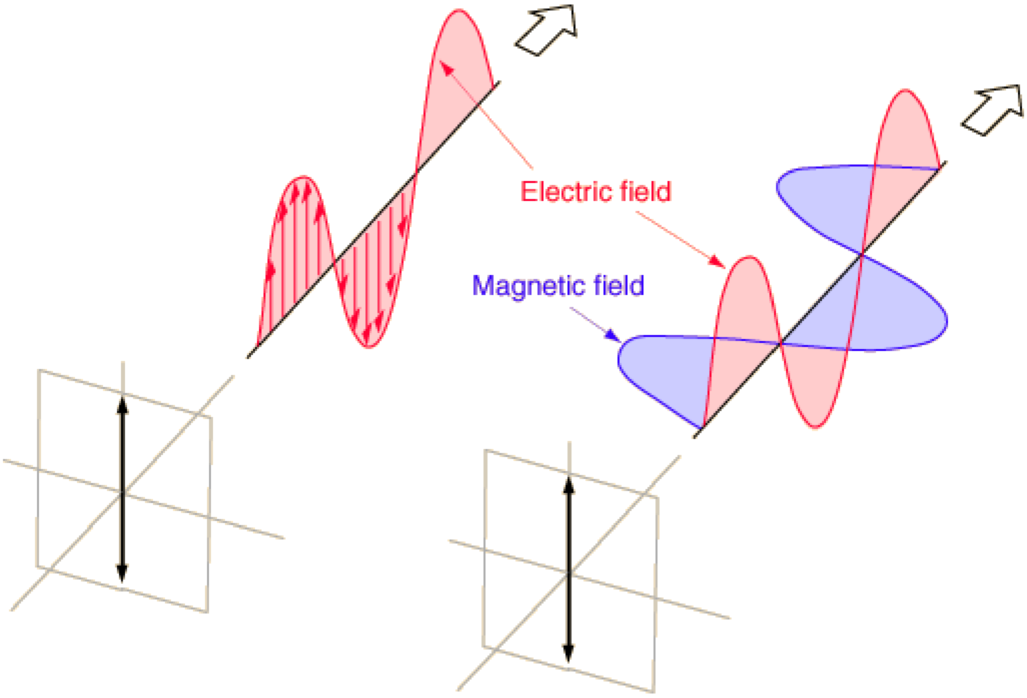
POLARTMETRY. Polarimetry is a sensitive, nondestructive technique for measuring the optical activity exhibited by inorganic and organic compounds. A compound is considered to be optically active if linearly polarized light is rotated when passing through it. The amount of optical rotation is determined by the molecular structure and concentration of chiral molecules in the substance.



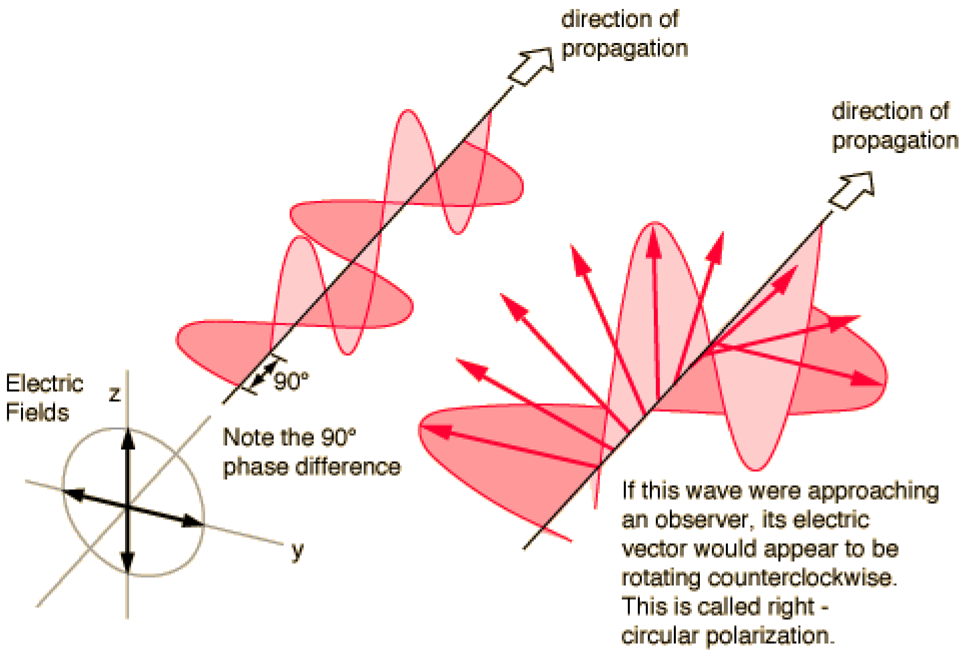
Normal monochromatic light contains light that possesses oscillations of the electrical field in all possible planes perpendicular to the direction of propagation. When it is passed through a polarizer (e.g. Nicol prism, Polaroid film), only light oscillating in one plane will leave the polarizer.



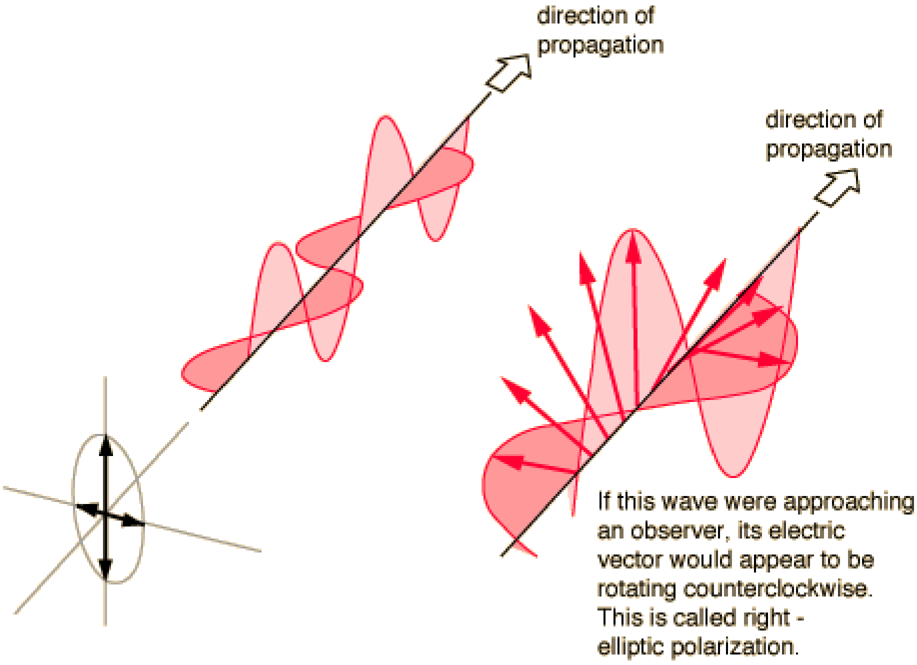
Linear Polarization. A plane electromagnetic wave is said to be linearly polarized. The transverse electric field wave is accompanied by a magnetic field wave as illustrated.



Circular Polarization Circularly polarized light consists of two perpendicular electromagnetic plane waves of equal amplitude and 90° difference in phase. The light illustrated is right-circularly polarized.



Elliptical Polarization. Elliptically polarized light consists of two perpendicular waves of unequal amplitude which differ in phase by 90°. The illustration shows right- elliptically polarized light.

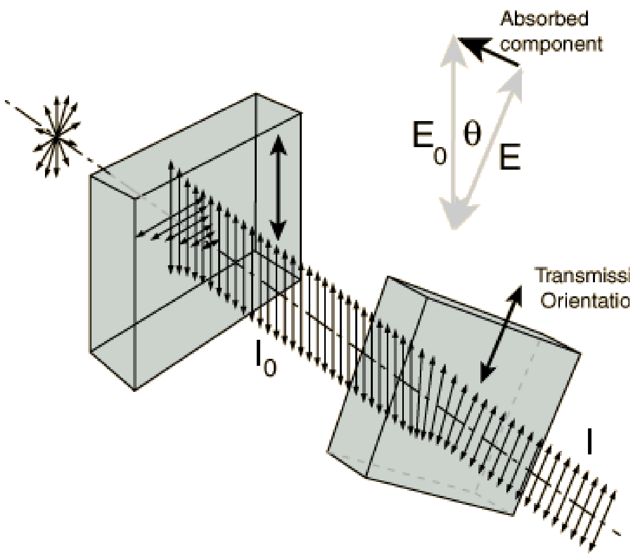


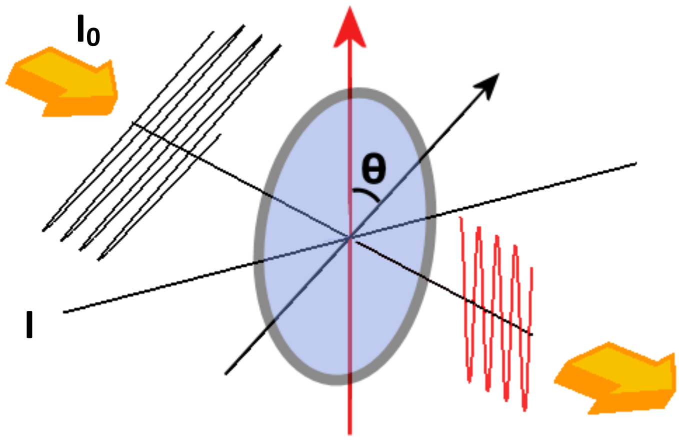
In a polarimeter, plane-polarized light is introduced to a tube (typically 10 cm=1 dm in length) containing a solution with the substance to be measured. If the substance is optical inactive, the plane of the polarized light will not change in orientation. The observer will read an angle of $\alpha=0^{o}$. If the compound in the tube is active, the plane of the light will be rotated on its way through the tube. In order to observe the maximum brightness, the observer (person or instrument) will have to rotate the axis of the analyzer back, either clockwise or counterclockwise direction depending on the nature of the compound. For clockwise direction, the rotation (in degrees) is defined as positive ("+") and called *dextrorotatory* (from the Latin: dexter=right). In contrast, the counterclock-wise direction is defined as negative ("-") and called *levorotatory* (from the Latin laevus=left). Unfortunately, there is no obvious correlation between the configuration [(D/L) in Rosanoff, (R/S) in Cahn-Ingold-Prelog nomenclature] of an enantiomer and the direction [(+) or (-)] in which they rotate plane-polarized light.

Polaroid filters absorb one component of polarization while transmitting the perpendicular components. The intensity of transmitted light depends on the relative orientation between the polarization direction of the incoming light and the polarization axis of the filter and is described quantitatively by Malus' law. The law stating that the intensity of a beam of plane-polarized light after passing through a rotatable polarizer (analyzer) varies as the square of the cosine of the angle through which the polarizer is rotated from the position that gives maximum intensity.

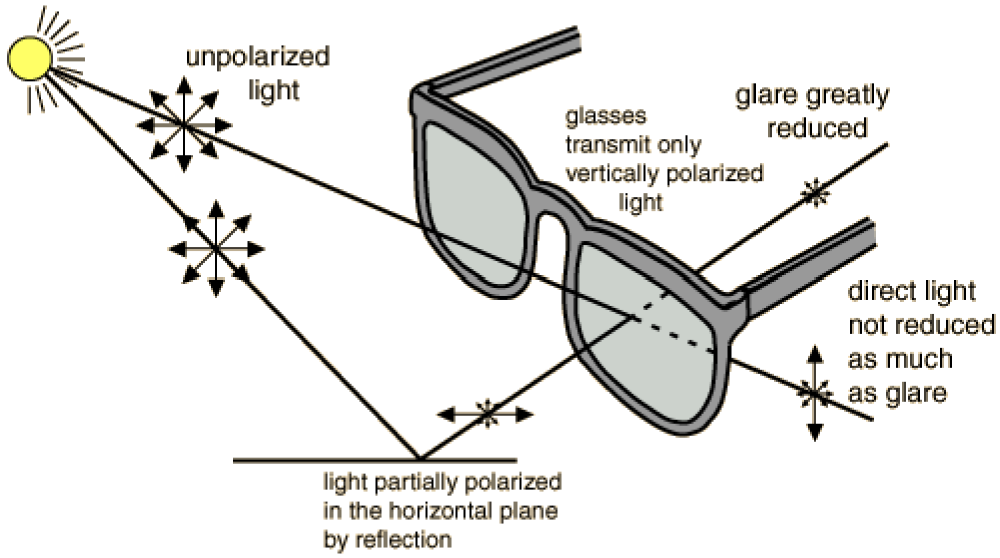
$$I = I_0 \cos^2 \theta$$

where I_0 is the incoming light intensity, I is the light intensity detected by the analyzer, and θ is the included angle between the polarizer's transmission axes and the analyzer's transmission axes.



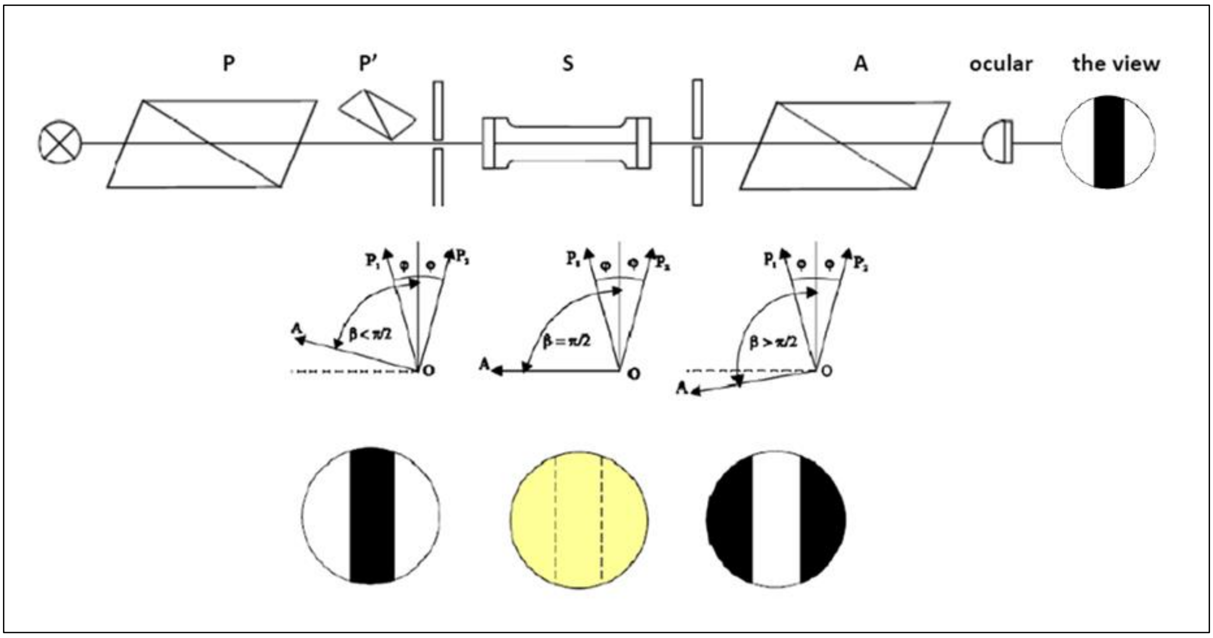


The polaroid material used in sunglasses makes use of dichroism, or selective absorption, to achieve polarization.



The experimental equipment - polarimetry

Lippich's polarimeter, which allows measurement of rotation angle of the plane of polarization, is shown in figure below. Monochromatic light from the sodium lamp ⊗ passes through a polarizer (P) (Nicol prism). Polarized light beam passes through the cuvette (sample) with optically active substance (S), Nicol prism, analyzer (A), and finally to the ocular. Nicol prism (P') serves as the half-shadow polarizer. If the analyzer A is perpendicular to the polarizer P, the free half of the field of view is dark, and if it is perpendicular to the half-shadow polarizer, the covered half is dark. If the analyzer is set to a middle position, the stripe and outer parts of the field of view are equally bright. This zero position can be adjusted very precisely because deviations from it are clearly visible due to the opposite changes in brightness of stripe and outer parts of the picture. In the experiment the rotation of the plane of polarization by a sugar solution is measured as a function of the concentration, whereby the concentration is changed in small steps. The angle of rotation strongly depends on the wavelength of the light. Therefore a yellow colour filter is used in the experiment.

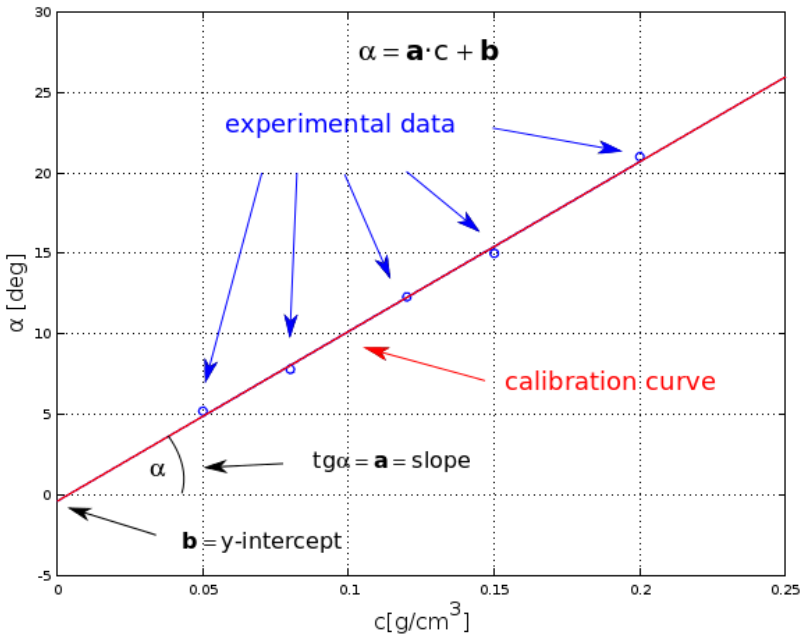


Overview and scope of the experiment

The theory predicts that rotation of polarization plane (α) depends linearly on concentration of optically active substance (c)

$$\boldsymbol{\alpha(c) = a \cdot c + b} \quad (1)$$

where: $\boldsymbol{a}$ is a slope of a straight line and $\boldsymbol{b}$ is its intersection point with *y*-axis. This straight line is called calibration curve and it can be used for determination of unknown concentration of investigated substance. An example of calibration curve fitted to experimental data is shown in the figure below.



The experiment with polarimeter is divided into three parts:

- ***Determination of calibration curve***. In this part of the experiment rotation angles of polarization plane are determined for solutions with known concentrations. Then the straight line (eq. 1) is fitted to the experimental data with linear regression method. As a result a and b coefficients, which define the calibration curve, are obtained.
- ***Determination of unknown concentration*** ($\boldsymbol{c_x}$). The measure the rotation angle of polarization plane (α_x) for a solution with unknown concentration is performed. Then eq. 1 and parameters a and b, obtained in the first part of the experiment, are used to calculate unknown concentration (c_x).
- ***Determination of specific optical rotation parameter*** ($\boldsymbol{\alpha_0}$). This parameter describes the tendency of investigated substance to rotate the polarization plane. Numerically it is the slope of the calibration curve (a) divided by the thickness of the layer of investigated solution (l).

Instruction

REFRACTOMETRY (Animation, part1)

1. The Abbe refractometer used in the Animation has two scales, one for reading the refractive index and second for reading the concentration of the test substance in %. This refractometer is rated for sucrose solutions. By clicking on the arrows move the separation line "white area - yellow area" to the center of the cross and read the concentration of the solution on the green scale (in %). Repeat this procedure at least three times for each six independent concentrations.
2. Convert the concentrations expressed in % into g / cm3. For the sake of simplicity, we assume that the solution density does not change and amounts to 1000 kg / m3. Enter the obtained concentrations in the appropriate place in the table.

POLARYMETRY (Animation, part2)

1. Starting with concentration 1 set the analyzer (by using the arrows under the figure) to see the two halves of the field of view are equally bright. Read the angle α. Set the analyzer to zero position and repeat all operations twice.
2. Follow the same procedure with the other solutions, including the solution of unknown concentration c_x.
3. Calculate the average angle of rotation of the polarization plane α for each of the solutions. Present the results in table.
4. Plot the data ($f(\alpha) = c$) and perform linear fit. Print the graph.
5. Knowing the parameters obtained from linear fit calculate C_x. Mark C_x on the printed graph.
6. Calculate the specific optical rotation α_0 of this substance.

$$\alpha_0 = \frac{slope\ of\ linear\ fit}{length\ of\ the\ path\ traveled\ by\ light\ in\ a\ solution}$$

   where:

   b – the slope calculated by computer,

   l - length of the path travelede by light In a solution = 2 dm.

7. The results present in the table, fill in the missing units. Make the conclusions.

No.	c (%)	c(g/cm^3)	α (°)	α_{av}(°)	α_0(.........)
1					
2					
3					
4					
5					
c_x	–				

[1] Walker J., Halliday and Resnick, *Principles of physics: international student version*, 9 th ed., extended, Hoboken : John Wiley & Sons, Inc., 2011. , ISBN 978-0-470-56158-4

[…] or other books on physics

Detailed instruction

8. Prepare the following *Table of Results*:

<table>
<tr><td></td><td>$c(g/cm^3)$</td><td>$\alpha(\text{deg})$</td><td>$\overline{\alpha}(\text{deg})$</td></tr>
<tr><td rowspan="3">1</td><td rowspan="3"></td><td></td><td rowspan="3"></td></tr>
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<tr><td></td></tr>
<tr><td></td></tr>
<tr><td></td><td>$c_x(g/cm^3)$</td><td>$\alpha_x(deg)$</td><td>$\overline{\alpha_x}(\text{deg})$</td></tr>
<tr><td rowspan="3">6</td><td rowspan="3"></td><td></td><td rowspan="3"></td></tr>
<tr><td></td></tr>
<tr><td></td></tr>
</table>

9. Using the last page of this instruction learn how to read the angle from the scale of polarimeter. Set an angle on the polarimeter to a random value, read the angle and write it down. <u>Ask the teacher to check if it is correct</u>.
10. Using the knob on the right hand side of the instrument set the analyzer (A) to zero degrees. Adjust the sharpness of the picture by turning the eyepiece. Gently rotate the knob away and towards yourself to learn how the picture changes. Set the analyzer in

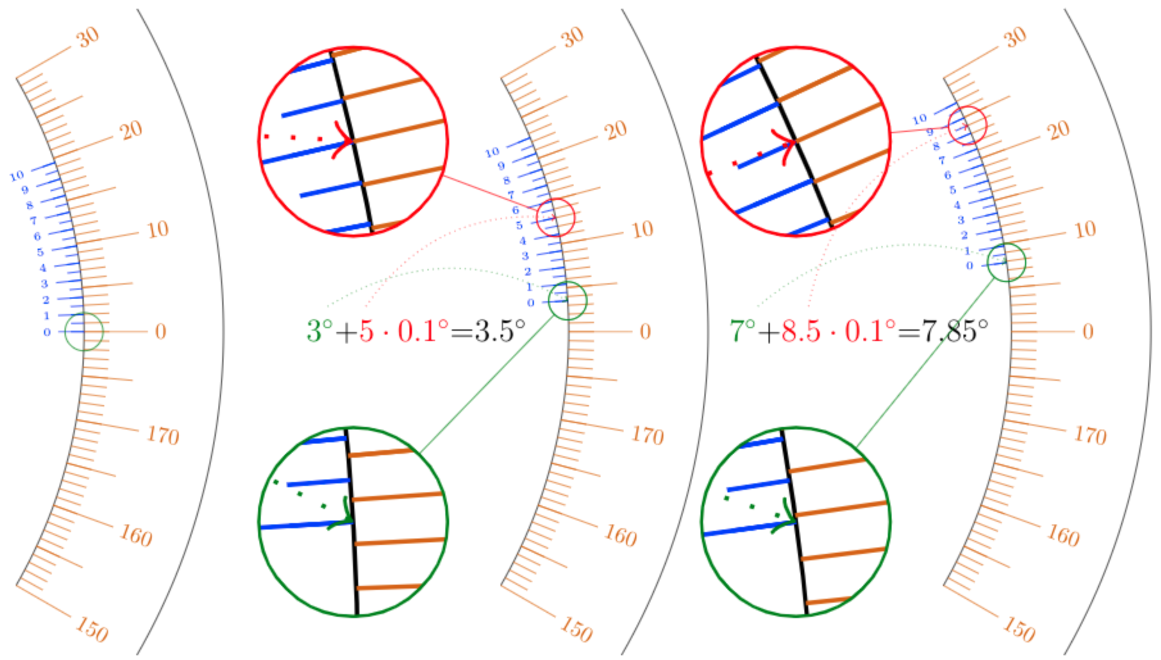
the position in which the picture is uniformly bright and the stripe is almost invisible. The reading from scale should be close to zero.

11. Fill the cuvette with the solution number 1. Write the concentration of the substance to *Table of Results*. The cuvette must be completely filled with a solution (no air bubble). Place the cuvette into the holder. Adjust the analyzer in order to obtain uniformly bright picture. Read the angle α and place it in *Table of Results*. Reset the analyzer to zero position and repeat the measurement twice. Write down results in *Table of Results*.

    IMPORTANT NOTICE! The angle of rotation should not exceed 40^o for any measurement.
12. Follow the same procedure with four remaining solutions.
13. Make three measurements of angle of rotation of polarization plane (α_x) for the solution with unknown concentration (c_x) and place them in the last row of *Table of Results*.
14. Calculate the average angle of rotation of the polarization plane α for each solution and put the results in the last column of *Table of Results*.
15. Enter the values of rotation of polarization plane α as a function of concentration of solutions *c* to the *GraphPad Prism* application. The data should obey the following linear formula $\alpha(c) = \boldsymbol{a} \cdot c + \boldsymbol{b}$ in which ***a*** – *slope* of the line and ***b*** – *y-axis intercept*. Perform linear regression transformation to fit the straight line. The algorithm will find best possible ***a*** and ***b*** coefficients. Print the graph.
16. Use values of α_x, parameters ***a*** and ***b*** and the equation of a straight line to determine the value of c_x.
17. Determine the specific optical rotation $\alpha_0 = \boldsymbol{a}/d$ of this substance, where ***a*** is the *slope* of the straight line obtained from linear regression and $d = 2dm$ is the thickness of investigated layer of solution.
18. What type of sugar was in solutions? Write down the conclusions.

[1] Walker J., Halliday and Resnick, *Principles of physics: international student version*, 9 th ed., extended, Hoboken : John Wiley & Sons, Inc., 2011. , ISBN 978-0-470-56158-4

How to read the scale of the polarimeter



(a) Reading 0° (b) Reading 3.5° (c) Reading 7.85°

The scale of polarimeter is built of two subscales: the main scale with range $2 \times (1 - 180^0)$ (big scale which does not move, marked with orange color) and Vernier scale (small moving scale, marked with blue color). The Vernier scale is divided into 20 intervals and every second mark has integer number from 0 to 10. The main scale is used for reading of full degrees. For this purpose a user should find a mark on the main scale which is the nearest mark below zero from Vernier scale (see green circles). In order to read tenth or hundredth of degree find two marks on both scales, which overlap best (see red circles). The number on Vernier scale multiplied by 0.1 is a part of the number after decimal dot. Add this number to integer part to obtain the angle of rotation of polarization plane.