Demonstrating Basic Properties and Application of Polarimetry Using a Self-Constructed Polarimeter

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INSTRUCTOR/STUDENT NOTES

1. Reagents and Materials

General materials for the polarimeter:

- 4 × Lego brick 2×1 with hole (grey: Element ID: 4211440) to hold the LEDs and the polarizing film in place (these are essential Lego bricks)
- 2 × Lego brick 2×1 with hole (grey: Element ID: 4211440) + connector peg/bush (grey: Element ID: 4211807) to attach the plastic dial to the protractor (these are essential Lego bricks, but can be replaced by others)
- 3 × Lego plate 6×16 (grey: Element ID: 4211733) as the base for the polarimeter
- 1 × Lego brick 2×2 (yellow: Element ID: 3003247/3003) to mount the brick holding the light source LED (yellow)
- 1 × Lego brick 2×2 (red: Element ID: 300321/3003) to mount the brick holding the detector LED (red)
- 1 × Lego brick 2×2 (grey: Element ID: 4211387/3003) to mount the Lego holding the polarizing film
- 2 × Lego plate 2×3 (grey: Element ID: 4211396) to hold the base together
- Lego bricks to hold the protractor in place (e.g. Lego Brick 2×4 - grey: Element ID: 4211385)

To acquire Lego parts, go to: www.lego.com and search for "Pick a Brick"

- Polarizing film; American Polarizers Inc.: AP42-007T-12X19 Visible Light Linear Polarizer – 42% Transmission – 12 in. × 19 in. x 0.007 in.
- Thick, sturdy plastic film to make the dial (e. g. laminated laminating pouch)
- 2 × 1.5 V (3 V) or 3 × 1.5 V (4.5 V) batteries and a battery holder or an appropriate power adaptor
- Wires with a crocodile clip at each end
- 1 × Yellow LED 5 mm: (ID: L5-Y14ER) as light source

- 1 × Red LED 5 mm: (ID: 333-HRC) as light detector
- A suitable resistor [typically in range 10-60 ohm (Ω)]
- Optical glass cuvettes with path lengths 10, 20, 40 and 100 mm (Amazon)
- Multimeter/Voltmeter (with internal resistance of at least 10 MΩ)
- Laminating pouches or cardboard for the protractor
- Extra LEDs for experiments on optical rotation as function of wavelength:

405 nm: UV-5TZ-405-15	525 nm: L5-G81N
428 nm: 383UBC/C430	592 nm: L5-Y14ER
470 nm: L5-B5SC	626 nm: HLMP-3750
480 nm: LC503FBL1-15P-A3-00001	635 nm: 826-442
500 nm: L5BG1N	660 nm: 333-HRC

Reagents and solutions

- D-(-)-Fructose (Sigma)
- D-(+)-Sucrose (VWR)
- (*R*)-(+)- and (*S*)-(-)-Limonene (Fluka)
- Citrus solvent ski wax remover (Swix)
- D-(-)- and L-(+)-Arabinose (Fluka)
- Syrups and Honeys (local stores)
- Essential oils (local or online stores)
- Ethanol (absolute)

Solutions with 0.50 g/mL fructose or sucrose were used in most measurements. Such high concentrations were a deliberate choice in order to improve the accuracy of the optical rotation readings. The amount of sugar was weighed, transferred to a measuring flask, dissolved in water and diluted to the mark. Dissolving of sugar at this concentration may take some time, and gentle heating will speed up the process. The solution should then be cooled to room temperature before dilution. Solutions of arabinose were made in the same way.

Solutions of limonene (*R* and *S*) in different concentrations were made with absolute ethanol. The amount of limonene was weighed directly in a measuring flask on a scale placed in a fume hood. The sample of citrus ski wax remover was made in the same manner.

Honeys and syrups were either used neat, or diluted to 40 or 50% with distilled water. Essential oils were used neat.

2. Hazards

There are no hazards involved in constructing the polarimeter from Lego bricks and the additional pieces. Make sure that the holes in the protractor and the plastic dial are made in a safe manner. Use an awl, a nail, a leather hole puncher (revolving punch plier) or some other suitable tool. If wanted, holders for the 1 and 4 cm cuvettes can be made by cutting the top off a Lego brick, either 2×2 or 2×6. The brick should be held with pliers and the top cut with a saw (i.e. small hacksaw). It is however easy to align the cuvette with the knobs on the Lego plate, making a holder unnecessary.

None of the LEDs used here are in a wavelength range harmful to the eyes. However, the contrast between the light source and the environment can be strong in dim lighting, especially for high intensity LEDs. We advise building extra walls of Lego-bricks or covering with small pieces of material to shield the light. This should not be a problem with the 592 nm yellow LED used in most experiments. The LEDs are cold-running and will not get warm. The voltage used here is low and the risk associated with this voltage is minimal.

With respect to chemicals the following apply: The carbohydrates are non-hazardous. Limonene, ethanol and essential oils are both flammable and irritating. Preparing solutions of limonene and citrus ski wax remover in ethanol and measurements of these solutions and essential oils require a properly ventilated location, preferably a fume hood. Goggles, gloves and lab coat should be used.

Extra Lego-bricks can be used to build walls on both sides of the cuvette to prevent this from falling over. If essential oils or limonene in ethanol are spilled on the Lego-bricks, dry off with a paper and leave the paper in the fume-hood. Lego bricks can be cleaned with water or ethanol. Do not use acetone as this will destroy the bricks. A lid or something similar like a glass microscope slide on top of the cuvette can be used to avoid evaporation.

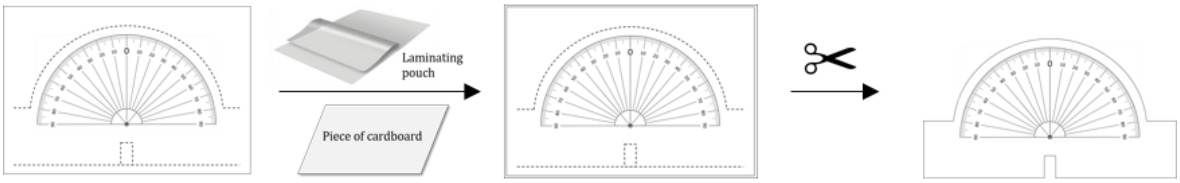
3. Patterns: plastic dial, polarizing films, protractor

Take a look at the diagram shown in section 4 before proceeding to make the protractor, the dial and the polarizing films. This will make it easier to understand the making of these parts.

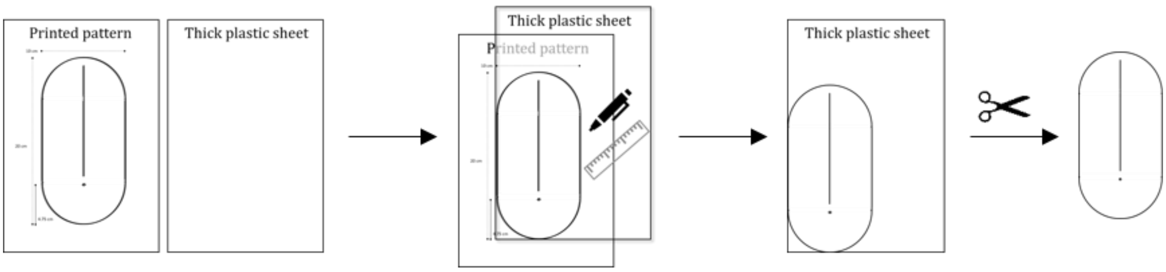
The attachments contain:

- A protractor (attachment 1).
- A pattern for the plastic dial (attachment 2).
- Patterns for the fixed and the rotating polarizing films (attachment 3).

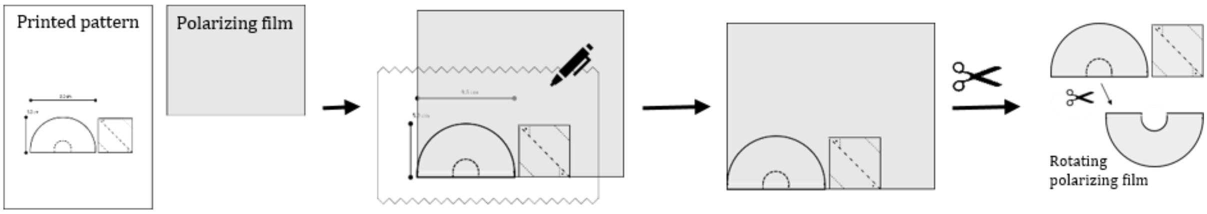
1) Print the protractor on paper (attachment 1). Laminate or fix the protractor onto a piece of cardboard. Cut along the dotted lines with a pair of scissors.



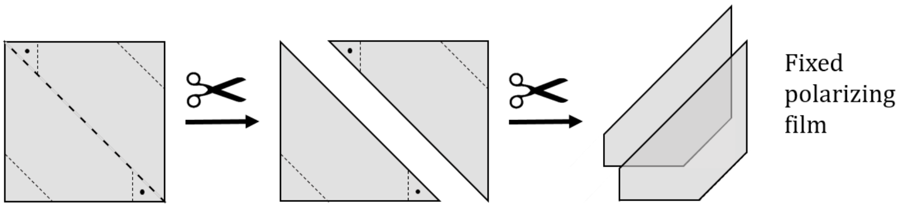
2) Print the paper pattern for the plastic dial (attachment 2). Use a thick plastic sheet (e.g. laminated laminating pouch) and place it on top of the pattern. Copy the lines onto the plastic sheet with a marker. Use a ruler to ensure the reading line is straight. Cut along the outer line with a pair of scissors. The paper pattern is not used further.



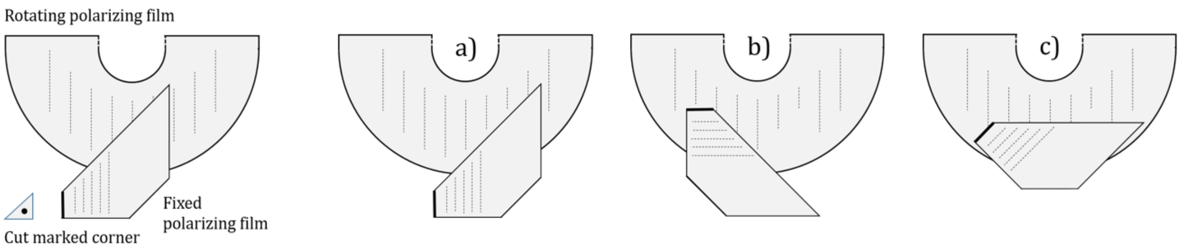
3) Print the paper patterns of the rotating and fixed polarizing films (attachment 3). Place the polarizing film on top of the patterns and copy the patterns with a marker. Cut the polarizing films around the outer line of the patterns with a pair of scissors.



Cut the square polarizing film along the dotted diagonal line (as shown under) and the cut off the two corners along the dotted lines. This makes two fixed polarizing films. Only one is needed for a single polarimeter.

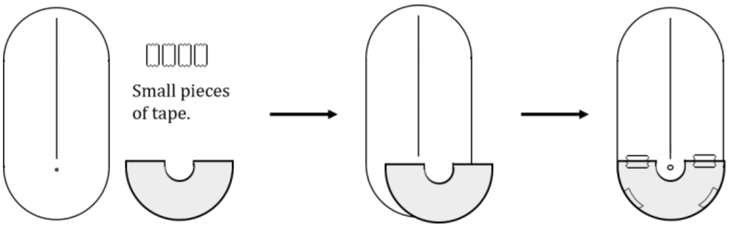


Note that the fixed polarizing film can be used in different positions for reading of maximum (a) and minimum (b) light transmittance, and for easy reading of high optical rotations (c). The cut of the marked corner shows the alignment between the fixed and the rotating films. Note that the lines shown on the polarizing films only show the alignment between them as long as the films have been cut in accordance with the attached pattern. They don't show the polarizing of the film.



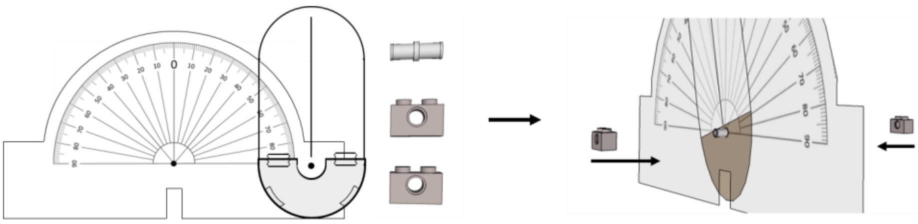
a) *Fixed and rotating films are aligned. Maximum transmittance will be at 0° on the protractor.*
b) *Fixed and rotating films are 90° to each other. Minimum transmittance will be at 0° on the protractor (alignment used for measurements here).*
c) *Fixed and rotating films are 45° to each other. Minimum and maximum transmittance will be at 45° to each side on the protractor.*

4) Fix the rotating polarizing film on the plastic dial with tape as shown. Note that the layer of plastic film is <u>behind</u> the polarizing film.



5) Make holes through the plastic dial and the protractor at the circles using an awl, a nail, a leather hole puncher (revolving punch plier) or some other suitable tool. Make sure the hole is small enough, otherwise the plastic dial may become too loose.

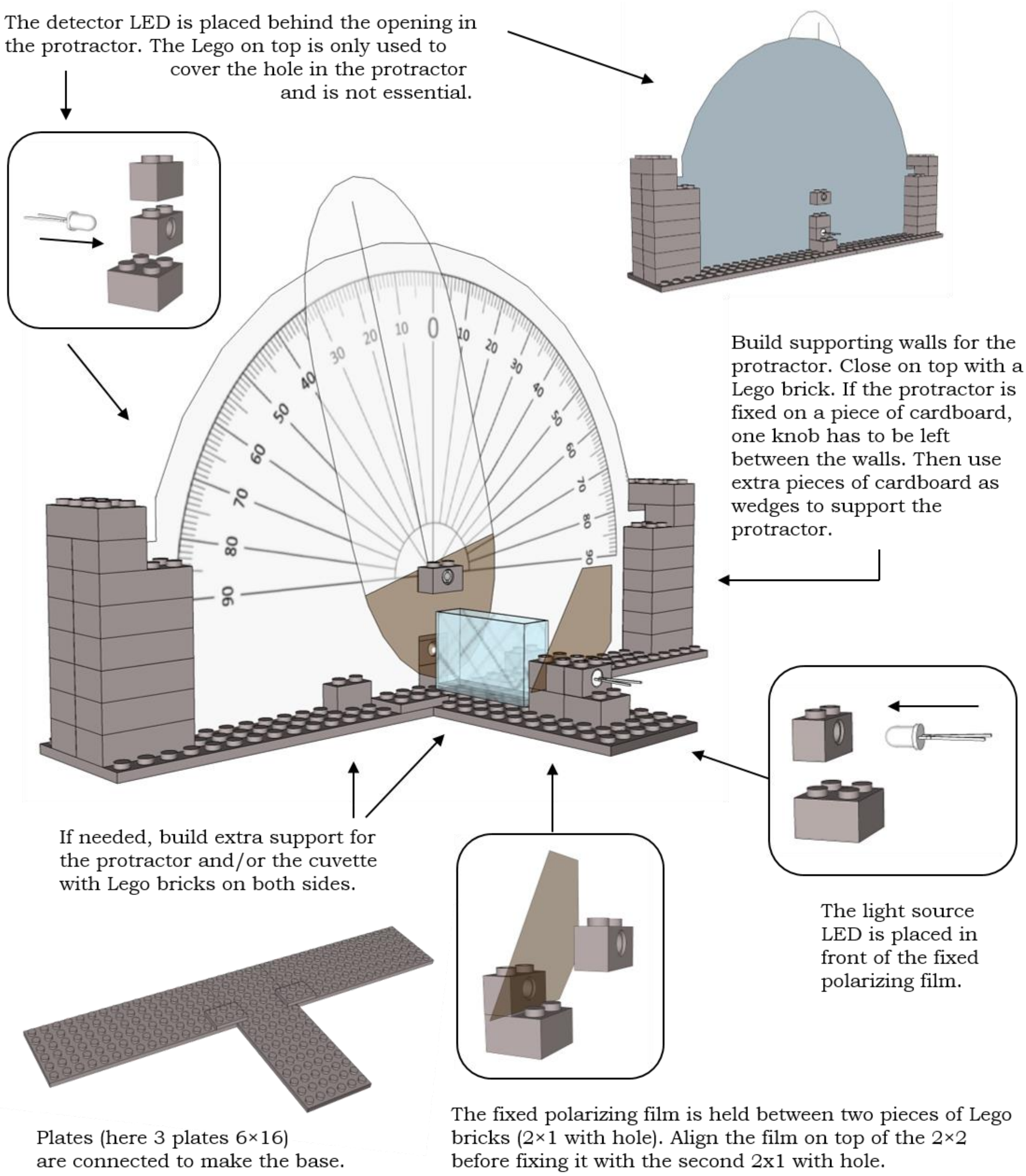
   Place the protractor and the plastic dial together, align the holes and push a Lego connector peg (or similar) through the hole. Keep the protractor and the plastic dial together by adding a Lego brick 1×2 with one hole on each side.



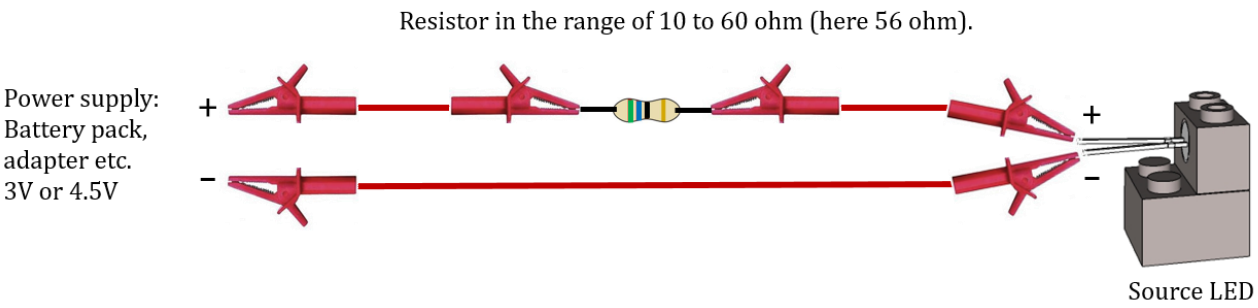
   Measurements can be done with the protractor either facing the light source LED or the detector LED (see later). As plastic films can depolarize plane polarized light, it is important to have the plastic dial with the polarizing film attached in the correct way. If the protractor is facing the light source LED, the plastic part of the dial should be next to the protractor. If the protractor is facing the detector LED, the polarizing film should be next to the protractor. The incident light should always hit the polarizing film before the plastic part of the dial.

4. How to assemble the polarimeter

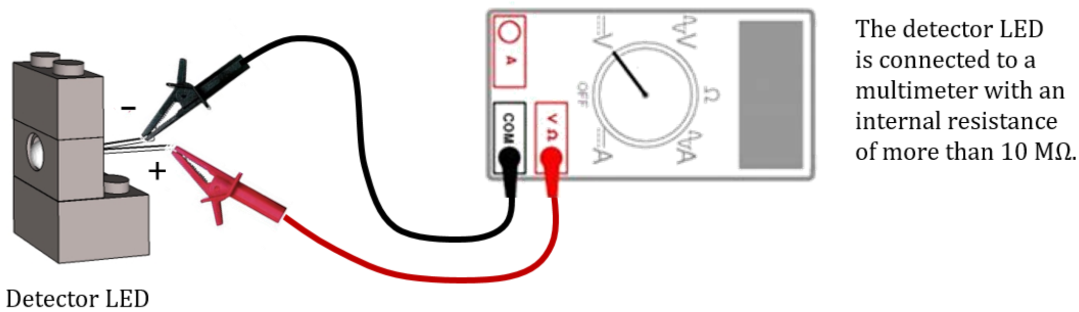
The polarimeter is assembled as shown in the diagram below. It is easily assembled and this can be done either by students or instructor. If time is restricted, the protractor, the fixed polarizing film and the plastic dial with the polarizing film can be prepared by the instructor in advance.



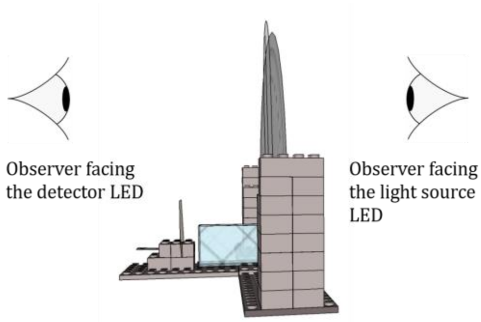
The source LED is connected to a power supply and a resistor. The power supply can be either a battery pack, a mobile phone charger[13] or a power adapter. The wire of the charger/adapter has to be cut to expose the two wires inside which are then connected to the circuit. The LED's negative pole (cathode, flat side of the LED, short leg) must be connected to the negative pole of the power source, and the LED's positive pole (anode, long leg) to the positive pole. If you are unsure, and the LED doesn't light, exchange the connections to the LED.



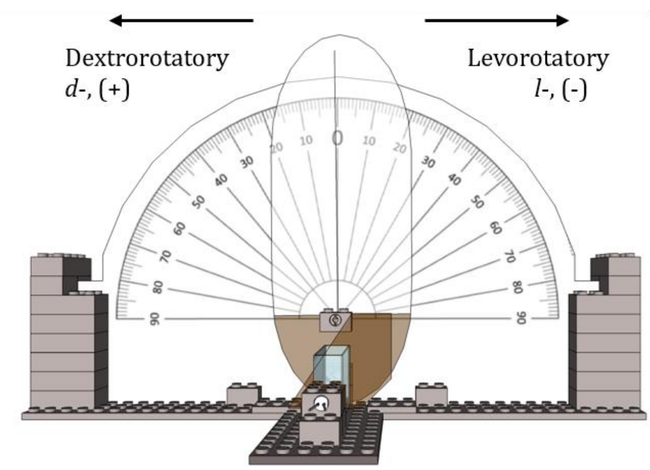
The detector LED is connected to a multimeter with an internal resistance of more than 10 MΩ. Depending on how the multimeter is connected to the detector LED, the output voltage can be plus or minus. This doesn't matter since we are only concerned with the absolute value. If the output is in minus and you would like to have it in plus, swap the connections to the detector LED.



The optical rotation can be recorded with the observer facing the detector (protractor facing the light source) or facing the light source (protractor facing the detector), as shown.



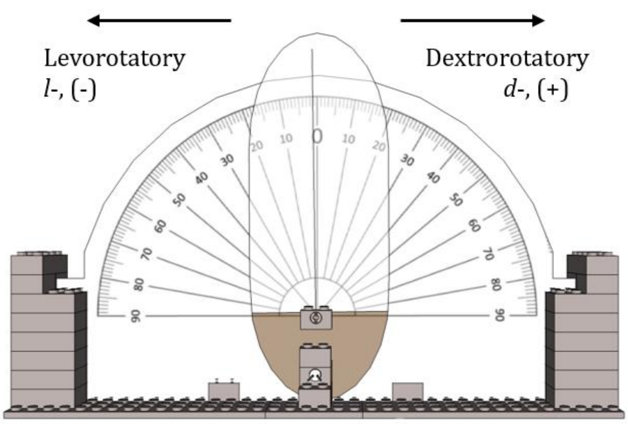
Observing the rotation facing the detector is actually opposite to the conventional rules for defining rotation of plane polarized light as dextrorotatory (right, (+), *d*-) and levorotatory (left, (-), *l*-). Therefore, a conventionally levorotatory solution will rotate to the right and a conventionally dextrorotatory solution to the left as shown below.



Observer facing the detector LED

Observer placed opposite normal convention.

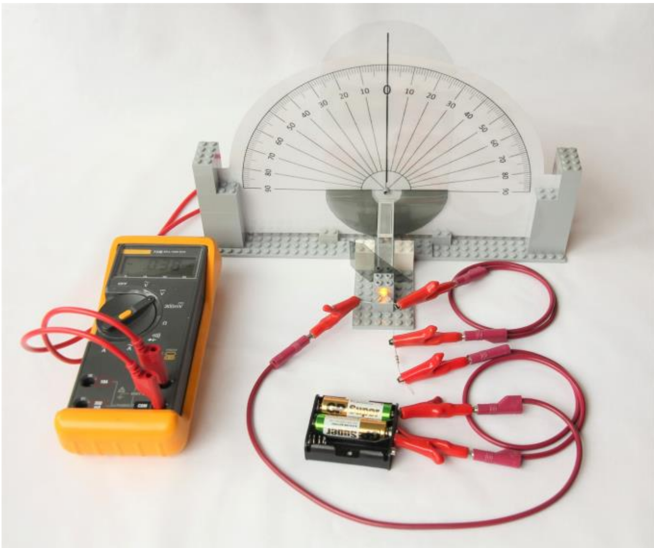
By definition optical rotation is to be observed facing the light source. With this set-up (observer facing the detector) a conventionally levorotatory solution will rotate the plane polarized light to the right and vice versa.



Observer facing the light source LED

With this set-up the rotation will follow normal conventions.

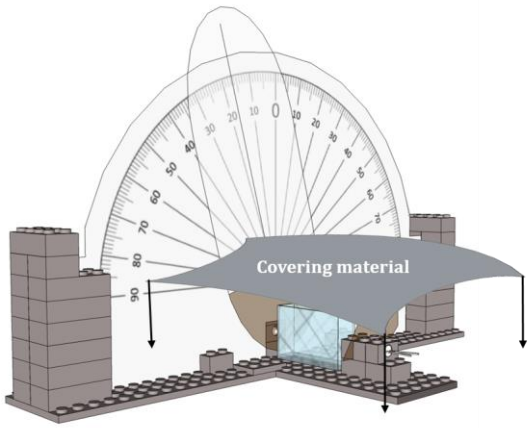
In this set-up the dial with the polarizing film must be turned around so that the polarizing film touches the protractor. The incident light from the light source must hit the polarizing film before the plastic film of the dial.



We prefer viewing the protractor facing the detector LED as shown. This makes replacing of cuvettes easier and less light from the LED reaches the eyes of the observer. We use long cables from the detector LED to the multimeter so we can observe this at the same time as we move the dial.

If you wish to face the light source LED, the protractor has to have its front towards the detector LED and the dial mounted so that the polarizing film is facing the light source through the opening in the protractor.

Readings should be undertaken in dim lighting, otherwise some covering will be needed.



5. Measurements

5.1 Optical rotation curves at maximum and minimum light transmittance

Optical rotation can be measured either at minimum or maximum light transmittance. As shown in Figure 1, the minimum point was smoother and easier to interpret. Similar results were obtained for two types of polarizing films.

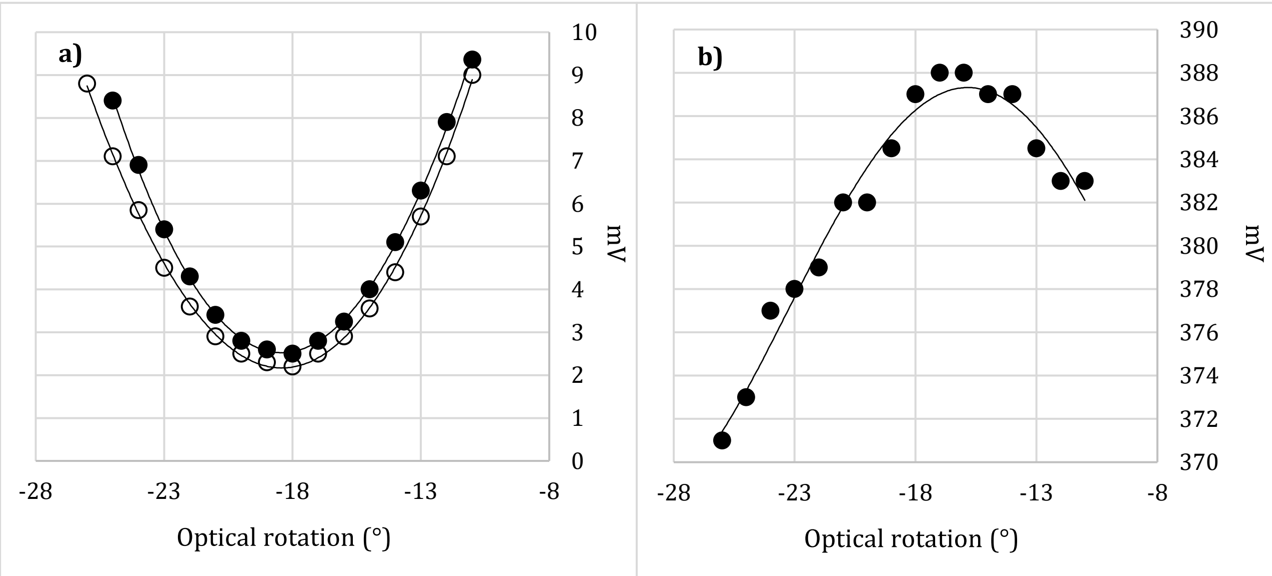


Figure 1: Reading of optical rotation of D-fructose (0.50 g/mL, 4 cm) at minimum transmittance (a) and maximum transmittance (b). Two types of polarizing films were tested: A cheaper polarizing film without specifications from Amazon intended for physics experiments in schools (open symbols, only shown for minimum transmittance) and a more expensive polarizing film from American Polarizer (filled symbols).

All readings have been made at minimum light transmittance, and many have been determined from plotted graphs like the ones shown in Figure 1 (a). A yellow diode (L5-Y14ER, λ_{max} 592 nm) was used as a light source in all experiments, unless otherwise stated, and a red diode (333-HRC) as detector. The light source was powered with 2 × 1.5V batteries or an appropriate power adapter. The latter provides a stable voltage (batteries get weaker). The circuit included a 56 ohm resistor. Measurements were made at ambient temperature (20 – 23°C), unless otherwise stated.

5.2 How to perform a measurement

Ideally, minimum light transmittance should be at zero. However, the precision in mounting the instrument will vary, and a calibration has to be done to find the optical zero. This is normally done also for commercial instruments. We have nulled the instrument without any cuvette.

Calibrated zero reading: Note the voltage at zero. Move the dial in steps of 0.5° to each side while observing the voltage on the multimeter attached to the detector LED. The lowest reading represents minimum light transmittance. Move back and forth several times and allow the system to stabilize between each movement. Note the degree for minimum transmittance which is the calibrated zero.

When a cuvette with an optical active solution is placed in the instrument, the optical zero will change because the plane of the incident light will be rotated. Move the dial slowly to each side to determine if the rotation is dextro- or levorotatory, and to locate the new optical zero. Move the dial in steps of 0.5° back and forth around the new optical zero for a precise reading. Note the degree for minimum transmittance. We do recommend to make graphs like in Figure 1 a). The net rotation is the difference between the new reading and the calibrated zero.

Be aware that the actual voltage at the new optical zero most often will be somewhat higher than at the calibration. The liquid in the cuvette refract the light and the intensity towards the detector will increase.

5.3 Identification of unknown sugars

The polarimeter was tested with 30 first year bachelor students (bioengineering) taking an organic chemistry course. The polarimeter was constructed in advance and was ready to use. Groups of two students each were given a short demo on how to use the polarimeter before they measured the optical rotation of three sugar solutions (0.50 g/mL, 4 cm cuvette) marked A, B and C. They calculated specific rotation from observed optical rotation, and identified the sugar solutions as D-fructose, D-sucrose and D-glucose. The identifications of the solutions were correct for all 15 groups. Student results (average and standard deviation for 15 measurements), measurements done by the teacher both with the self-constructed polarimeter and with a scientific instruments (Anton Paar) together with literature values from Sigma-Aldrich are given in Table 1 in the manuscript.

5.4 Varying concentrations (Figure 2 in the manuscript) and quantification of (*R*)-limonene in citrus ski wax remover

Optical rotation as a function of concentration was determined for both (*R*)-(+)- and (*S*)-(-)-limonene. Neat (*R*)-limonene has a density of 0.842 g/mL at 20°C while (*S*) has a density of 0.844 g/mL at 25°C according to Sigma-Aldrich. The concentrations made are given below together with measured rotation on the self-constructed polarimeter and on a commercial instrument (Anton Paar).

Compound	Amount (g)	Volume (mL)	Concentration g/mL	α (°) 592 nm (4 cm)	α (°) 589 nm Anton Paar
(*R*)-(+)-Limonene	1.0005	10	0.100	+5	+4.04
	5.000	25	0.200	+10	
	7.520	25	0.301	+14.5	
	10.030	25	0.401	+19.5	
			0.500		+23.4
	15.00	25	0.600	+28	
	neat		0.842	+41	+40.5
(*S*)-(-)-Limonene	1.0058	10	0.101	-3	-3.25
	2.9800	10	0.298	-10	-9.65

	5.0830	10	0.508	-17.5	-17.67
	0.5994	10	0.599	-21	-21.35
	neat		0.844	-30.5	-32.62

The calibration curves are shown in Figure 2 in the article. To determine the content of (R)-limonene in citrus ski wax remover, 10.00 g was diluted to 25 mL giving a concentration of 0.400 g Citrus solvent per mL. The measured optical rotation was +5°. The correlation between concentration and optical rotation is given by the equation for the linear regression of the calibration curve for (R)-limonene (Figure 2 in the article):

$$y = 47.847x + 0.1929$$

Where y is the observed optical rotation α and x is the concentration of (R)-limonene in g/mL. Using this equation to calculate the concentration for an optical rotation of +5 gives:

$$g/_{mL} = \frac{\alpha - 0.1929}{47.847} = \frac{5 - 0.1929}{47.847} = 0.100$$

A concentration of 0.100 g/mL of (R)-limonene in a solution of 0.400 g/mL of citrus solvent gives a content of 25% limonene.

5.5 Varying path length (Figure 3 in the manuscript)

Optical rotation as a function of path length was determined using a D-(-)-fructose solution with a concentration of 0.50 g/mL. Cuvettes with path length 1, 2, 4 and 10 cm were used. The results are given below.

Path length:	2 cm	2 cm	4 cm	10 cm
α (°) 592 nm	-4.5	-9.5	-19.5	-47

5.6 Varying temperature (Figure 4 in the manuscript)

Optical rotation as a function of temperature was determined using a D-(-)-fructose solution with a concentration of 0.50 g/mL in a 4 cm cuvette. For the two lower temperatures of 9.5 and 15.5°C, a cuvette with the fructose solution was placed in the fridge to cool down for two hours. The cuvette was then placed in the polarimeter and optical rotation measured during the time the solution heated up. Measurements were made at 9.5 and 15.5°C. For temperatures above room temperature, the cuvette with the solution was placed in a heated water bath for 10 minutes, then placed in the polarimeter and optical rotation measured during the time the solution cooled down. Measurements were made at 52, 43, 37, 30, 24 and 22°C. The results are given below.

Temperature °C	9.5	15.5	22	24	30	37	43	52
α (°) 592 nm	-21	-20	-19	-18.5	-18	-17	-16	-14.5

5.7 Varying wave length (Figure 5 in the manuscript)

Specific optical rotation as a function of wavelength was determined for D-(+)-sucrose and D-(-)-fructose both at 0.50 g/mL with a 4 cm cuvette. Details for the equipment used are given below.

λ (nm)	Source ID	Color	Voltage (V)	Resistor (Ω)	Detector	Detector ID
405	UV-5TZ-405-15		4.5	56	480 nm - blue	LC503FBL1-15P-A3-00001
428	383UBC/C430	Blue	4.5	56	480 nm – blue	LC503FBL1-15P-A3-00001
470	L5-B5SC	Blue	4.5	56	480 nm - blue	LC503FBL1-15P-A3-00001
500	L5BG1N	Blue/ green	4.5	56	660 nm - red	333-HRC
525	L5-G81N	Green	4.5	56	660 nm – red	333-HRC
592	L5-Y14ER	Yellow	3	56	660 nm – red	333-HRC
626	HLMP-3750	Red	3	56	660 nm – red	333-HRC
635	826-442	Red	3	56	660 nm – red	333-HRC
660	333-HRC	Red	3	56	660 nm - red	333-HRC

The measured rotations and the calculated specific rotations for the self-constructed polarimeter are given below together with results from a commercial instrument (Anton Paar).

λ (nm)	Self-constructed polarimeter (4 cm)				Anton Paar	
	D-Sucrose (0.5g/mL)		D-Fructose (0.5g/mL)		D-Sucrose	D-Fructose
	α (°) 592 nm	$[\alpha]_{592}$	α (°) 592 nm	$[\alpha]_{592}$	$[\alpha]_{589}$	$[\alpha]_{589}$
405	+29	+145	-43.5	-217.5		
428	+27	+135	-40	-200		
436					+127.94	-176.12
470	+21	+105	-30	-150		
500	+17	+85	-25	-125		
525	+15	+75	-22.5	-112.5		
546					+77.84	-106.92
579					+68.67	-94.3
589					+66.09	-90.8
592	+13	+65	-18.5	-92.5		
626	+10.5	+52.5	-16.5	-82.5		
635	+11	+55	-16.5	-82.5		
660	+10	+50	-15.5	-77.5		

It is also possible to use D-(+)-glucose as long as the solution is made in good time before the measurements. When α-D-glucose is dissolved in water an equilibrium will form between the α– and the β–anomers. The optical rotation will change (mutarotation) until equilibrium is reached. This process can be monitored with a polarimeter. At equilibrium (~36% α with a specific rotation of +112° and ~64% β with a specific rotation of +18.7°) the optical rotation will be constant and the specific rotation about +52 - 53° at 589 nm.

5.8 Enantiomeric excess (ee)

Enantiomeric excess was determined using L-(+) and D-(-)-arabinose. Solutions with 0.200 g/mL (5.00 g in 25 mL) for each enantiomer were made. A mixture with L:D = 3:1 having a theoretical enantiomeric excess of 50% were made by mixing 10.5 mL L- and 3.5 mL D-arabinose.

5.9 Syrups and honeys

Some of the syrups and honeys were too dark to be measured neat. These were diluted with distilled water to 40 or 50% in order to increase light transmission to a level giving reliable measurements.

Type	Optical rotation α (°) 592 nm (4 cm)	Calculated optical rotation for neat for comparison
Kjartan’s Honey Dew/ Forest Honey - 50 %	- 4	-8
Liquid mountain honey European Acacia - neat	- 6	-6
Kjartan’s Raspberry honey - 50%	- 2	-4
Serbian Forest honey - 50%	+ 2	+4
Lyle Golden Syrup - 40%	+ 5	+12.5
Maple Syrup Winnitou - neat	+ 22	+22

5.10 Essential oils

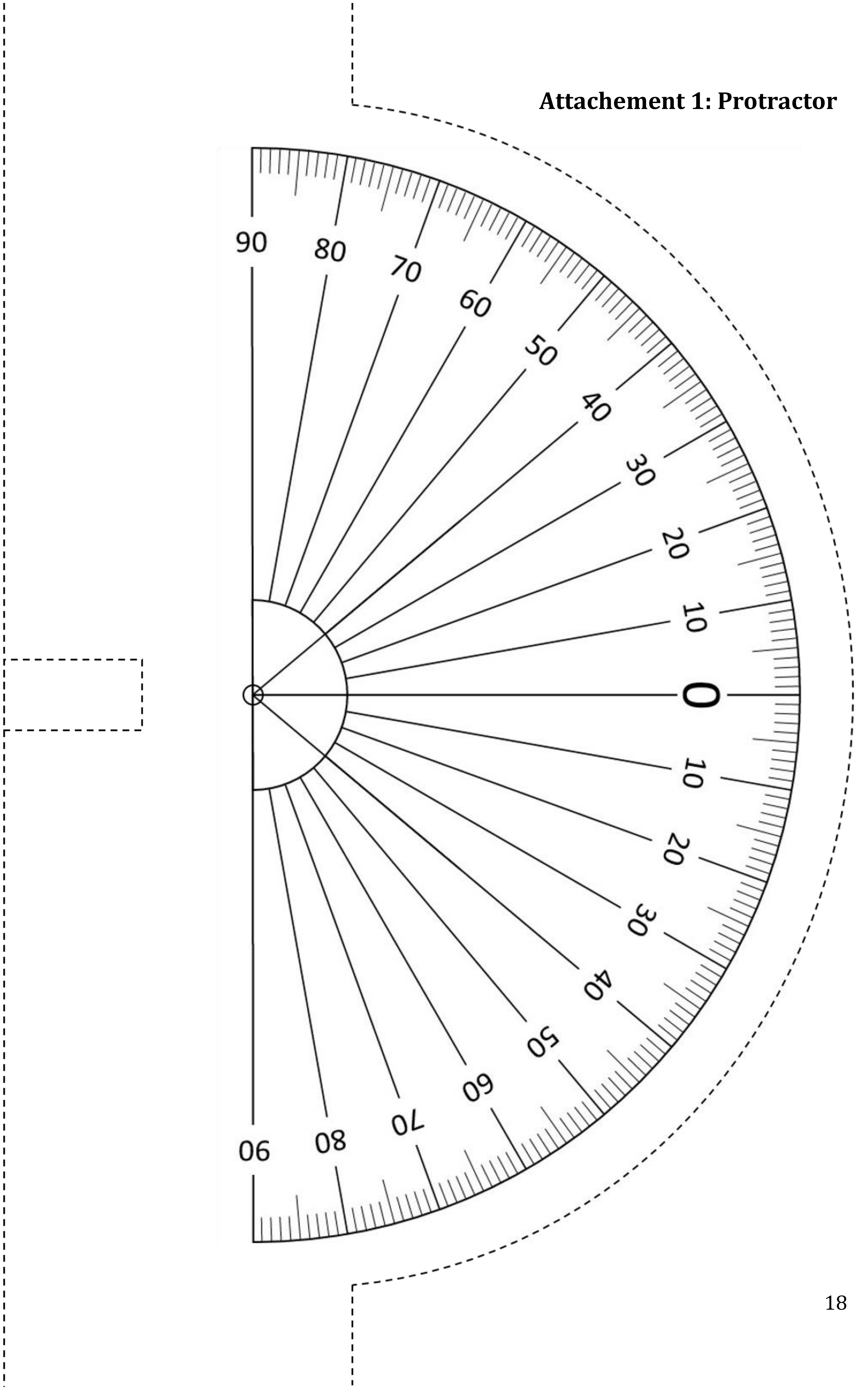
Essential oils were purchased at local stores in Norway and France, or ordered on-line. All the essential oils were measured neat.

6. Polarimeters in the classroom

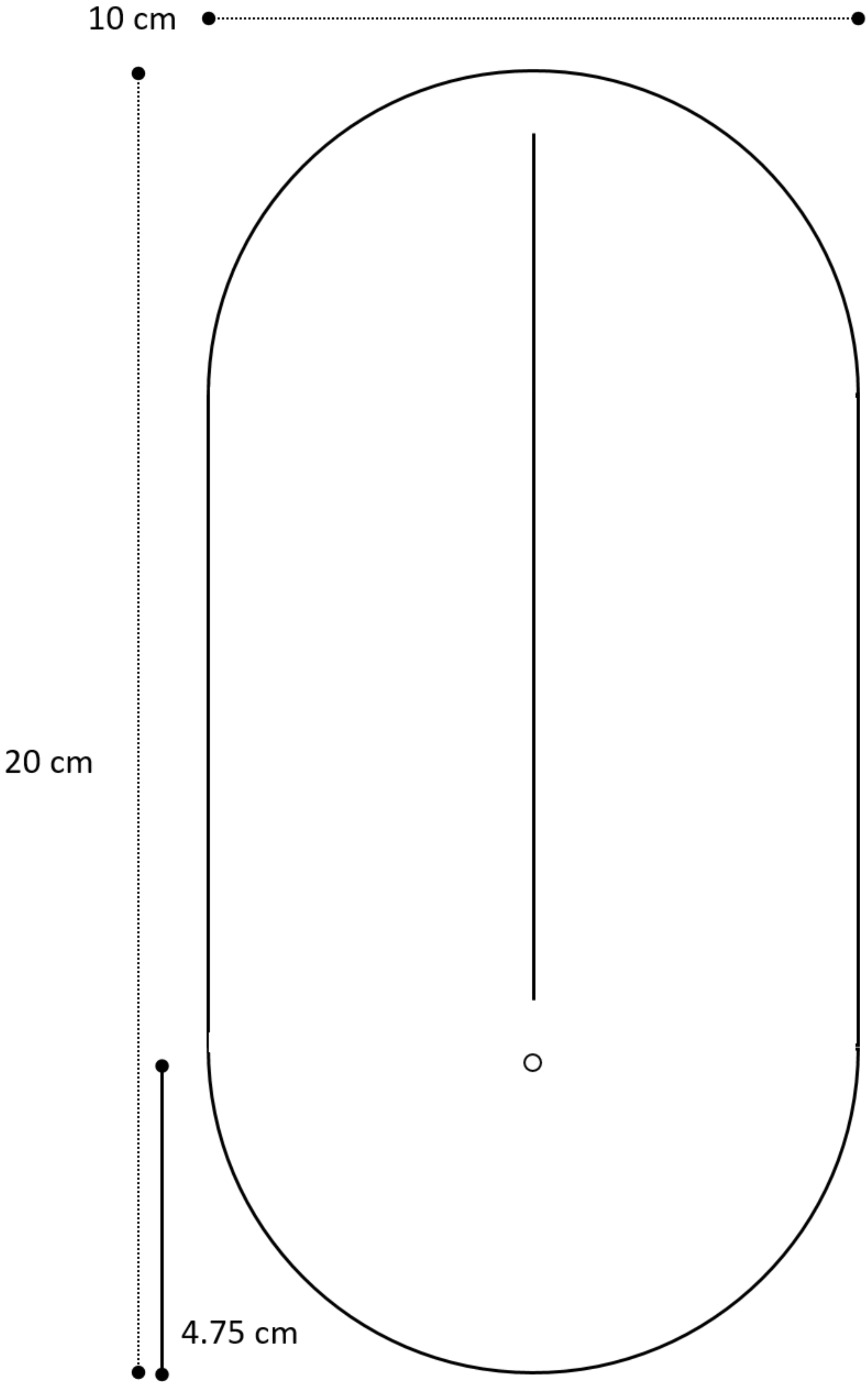
The Lego-parts of the polarimeter are very robust and can hardly break. They are not affected by ethanol, although by acetone (which is not used here). The students should be careful with the cuvettes, but this goes for all handling of cuvettes. The only mistake we suspect students can make, is that of not including a resistance in the circuit. This will damage the LED after a while, but it can easily be replaced. We have tested the polarimeter with many students without any damage so far.

As these polarimeters are inexpensive and simple to make it is easy for the teacher to make many before classes. We have used several pre-made ones, with maximum 4 students around each when optical rotation was the main focus and time restricted. In courses with in-service teachers, we have let the students make their own. The choice is up to the instructor, depending on the specific learning situation.

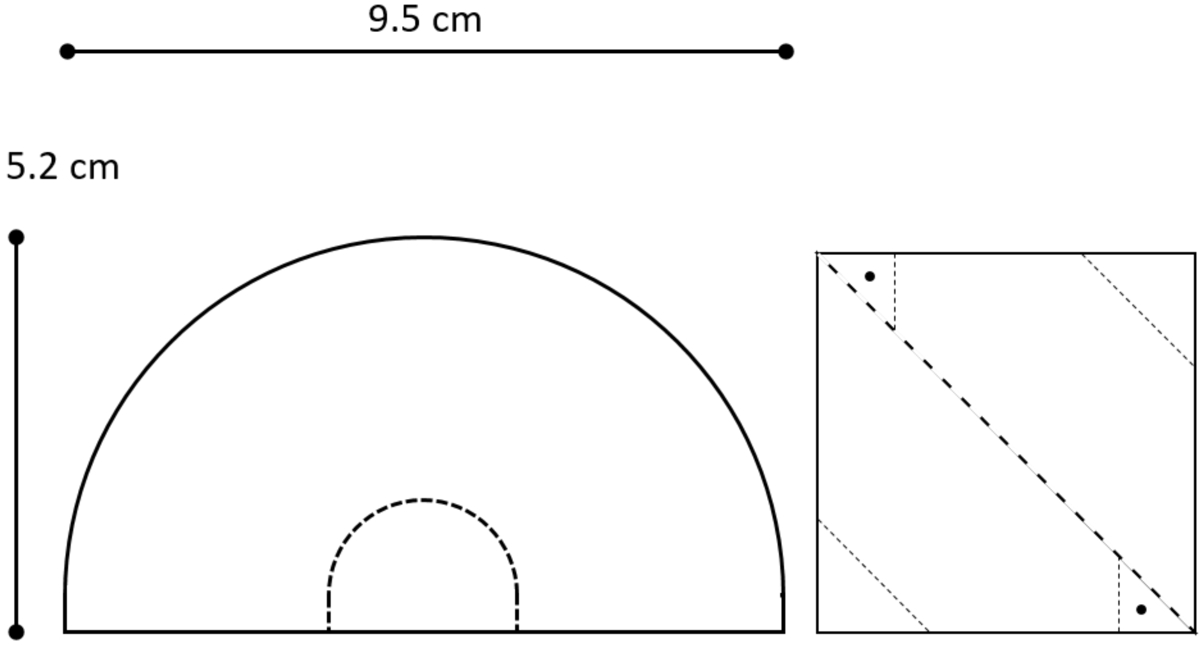
Attachement 1: Protractor



Attachment 2: Pattern for the plastic dial



Attachment 3: Patterns for the rotating and fixed polarizing films



Rotating polarizing film

Fixed polarizing film