

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

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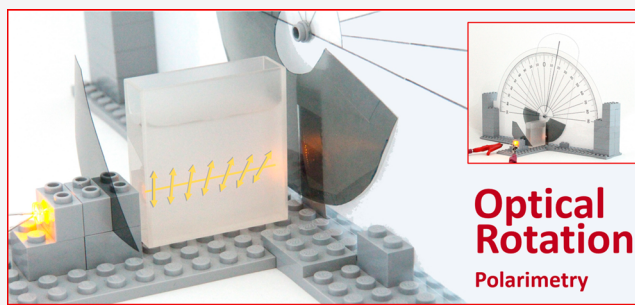
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Supporting Information

ABSTRACT: An easily constructed and inexpensive polarimeter with an optical rotation angle resolution of about 0.5° is presented. It is made from small pieces of polarizing film, 2 LEDs, a protractor, and a few wires, all held in place with plastic interlocking toy bricks, such as Lego bricks. The instrument was used to demonstrate the optical rotation of plane polarized light as a function of concentration, path length, temperature, and wavelength, and to determine enantiomeric excess in solutions of arabinose, the amount of limonene in citrus ski wax remover, and optical rotations of various types of honeys and essential oils. Results were comparable to values obtained on a commercial scientific instrument, and with literature values.

KEYWORDS: High School/Introductory Chemistry, First-Year Undergraduate/General, Second-Year Undergraduate, Upper-Division Undergraduate, Analytical Chemistry, Organic Chemistry, Laboratory Instructions, Hands-On Learning/Manipulatives, Chirality/Optical Activity, Spectroscopy



Optical
Rotation
Polarimetry

INTRODUCTION

Students encounter polarimetry in high school in connection with stereochemistry. The classical schematic representation of a polarimeter is illustrative and easy to understand, an asset not shared by the modern polarimeter, where basic features are hidden. Many simple, self-constructed polarimeters have been reported for use in demonstrations and student experiments.^{1–11} Overheads have earlier been used as a light source.^{2–5} These have now been replaced by LEDs,^{6,7} lasers,^{8,9} and LCD screens.^{10,11} We have previously published descriptions of simple instruments constructed with plastic interlocking toy bricks, such as Lego, and LEDs for UV–vis absorption and fluorescence measurements intended for student experiments.^{12–14} We add here polarimetry, extending the range of spectroscopic methods possible with the same basic building blocks. The polarimeter has the same layout as a classical polarimeter, making it easy to explain fundamental principles and instrumental design. It is not a black box system; it is inexpensive, easy to assemble, and flexible; can be used for many different compounds at varying concentrations; and has a precision and resolution sufficient for students' use. This makes it a convenient device for a range of quantitative measurements.

The emphasis in this article is on the design and application of the polarimeter. First, the design is explained. Second, basic properties such as optical rotation as a function of concentration, path length, temperature, and wavelength are demonstrated. Third, applications are presented: finding the

enantiomeric excess in arabinose solutions and limonene in a citrus ski wax remover as well as the optical rotation of honeys, syrups, and essential oils.

EQUIPMENT SETUP

Figure 1 illustrates the arrangement of the instrument when the observer faces the detector.

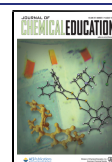
It is also possible to face the light source. This can be achieved by turning the protractor and arrow toward the detector side. However, independent of which direction observations are made in, the light from the source must hit the polarizing film before the plastic sheet that constitutes the dial, as plastic films depolarize plane polarized light.

The flexibility obtained by using Lego bricks makes it simple to increase and reduce the path length and to change wavelengths. Optical rotation is often reported using the D-line of sodium at 589 nm; thus, a yellow LED with a peak λ at 592 nm has been used in most of the experiments here. Optical rotation readings of solutions are made relative to zero readings with solvent (or air). All readings are undertaken at minimum light intensity because readings at maximum light

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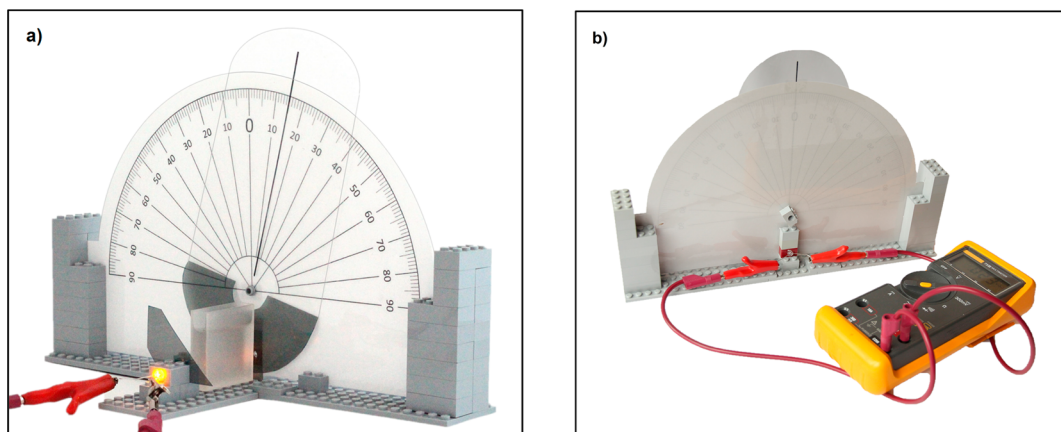


Figure 1. Self-constructed polarimeter. (a) From left to right: LED used as a light source (592 nm), polarizing film fixed between two Lego bricks, sample cuvette, and second polarizing film superimposed onto the end of a plastic dial that can rotate around the protractor. (b) LED used as a detector connected to a multimeter with a high internal resistance ($>10\text{ M}\Omega$).¹⁴

intensity are less accurate.^{5,8} The resolution is about 0.5° ; thus, small rotations have correspondingly larger error margins. See [Supporting Information](#) (SI) for detailed instructions.

The layout closely resembles the classical schematic representation of optical rotation by chiral compounds. It is easy to demonstrate that the amount of radiation (light) passing through the second polarizing film is dependent on the rotatory power of the solution in the cuvette, and the degree of rotation of the second polarizing film.

■ EXPERIMENTAL SECTION

Polarizing film was purchased from American Polarizer (AP42-007T-12X19). Polarizing films intended for physics experiments in schools (from Amazon.com, without specifications) gave almost the same results and may be used. Cuvettes/cells used were of optical glass (path length; 1.0, 2.0, 4.0, and 10.0 cm) and purchased through Amazon.com. Plastic cuvettes cannot be used as plastic depolarizes plane polarized light. For the measurement of rotation as a function of wavelength, LEDs with wavelengths in the range 405–660 nm were used. For all other experiments, a yellow LED with a peak λ of 592 nm and a bandwidth of 20 nm was used. See [SI](#) for more information and instructions for making the protractor, plastic dial, and pieces of polarizing film; for assembling the instrument; and for performing measurements.

Solutions were made of fructose, sucrose, arabinose, limonene, citrus ski wax remover, and some of the honeys and syrups. The essential oils were used neat. See [SI](#) for detailed information.

Rotation measurements were made at minimum light transmission (lowest reading of the voltmeter connected to the detector LED) in a dimly lit room. Control measurements were regularly performed on a commercial scientific instrument (Anton Paar MCP 5100, 589 nm) for comparison. Measurements were made at ambient temperature ($20\text{--}23^\circ\text{C}$), unless otherwise stated. This is in the same range as the literature values.

■ HAZARDS

With respect to chemicals, the following apply: The carbohydrates are nonhazardous. Limonene, ethanol, citrus ski wax remover, and essential oils are both flammable and irritating. The preparation of 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. Experiments with sugars in water represent no hazards. Use a lid or something similar like a microscope slide to cover the cuvette if necessary.

There are no hazards in constructing the device. 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 to build extra walls of Lego-bricks or to cover with small pieces of material to shield the light. This is not 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 is low, and the risk associated with this voltage is minimal.

■ BASIC PROPERTIES: OPTICAL ROTATION VS CONCENTRATION, PATH LENGTH, TEMPERATURE, AND WAVELENGTH

Optical rotation measurements as a function of concentration were performed with (R)- and (S)-limonene ([Figure 2](#)). The linearity between concentration and observed rotation is good for both enantiomers.

Optical rotation as a function of path length and temperature was determined using D-fructose, increased with path length ([Figure 3](#)), and decreased with temperature ([Figure 4](#)).

Optical rotatory dispersion (ORD) is often demonstrated simply as a color change when white light is rotated by a solution of a chiral compound.^{2,3,15} With LEDs of different wavelengths, this can easily be done quantitatively. Specific rotation as a function of wavelength (ORD) was determined for both D-fructose and D-sucrose. Specific rotations were calculated from observed optical rotation according to [eq 1](#) where $[\alpha]$ is specific rotation, α is observed optical rotation, l is the length of the cuvette in dm, and c is the concentration in g/mL.

$$[\alpha] = \frac{\alpha}{l \times c} \quad (1)$$

Specific rotation increased with decreasing wavelength ([Figure 5](#)) displaying a plain ORD curve. The results are in

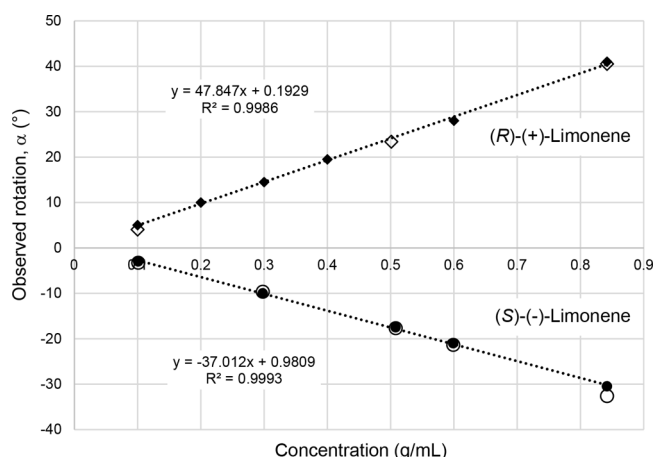


Figure 2. Optical rotation vs concentration for (R)-(+)-limonene (diamond) and (S)-(-)-limonene (circle). Self-constructed polarimeter: filled symbols; wavelength = 592 nm; path length = 4 cm. Anton Paar: open symbols; wavelength = 589 nm.

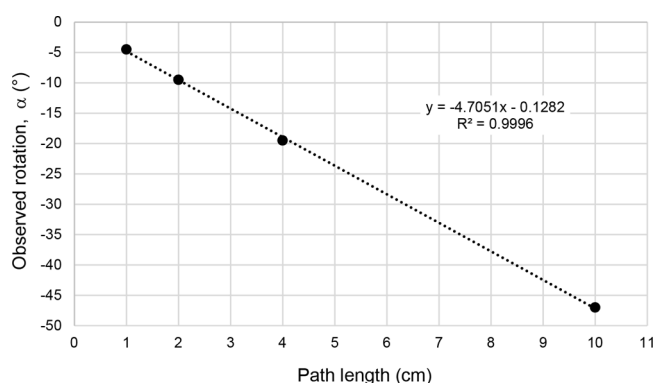


Figure 3. Optical rotation vs path length for D-(-)-fructose (0.5 g/mL). Wavelength = 592 nm.

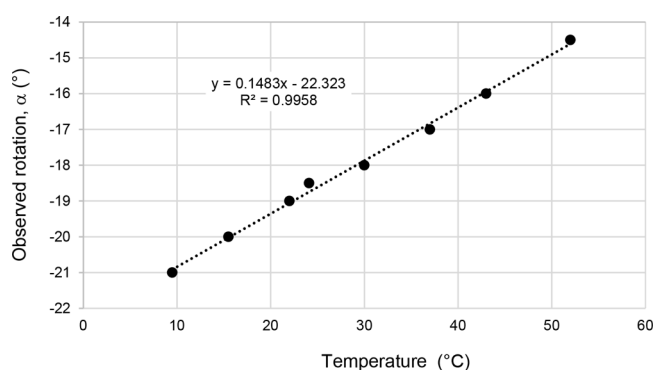


Figure 4. Optical rotation vs temperature of D-(-)-fructose (0.5 g/mL). Wavelength = 592 nm. Path length = 4 cm.

accordance with literature values.^{11,16–19} Extracted values for D-sucrose from Figure 4 in Mahurin et al.¹⁶ are included in Figure 5 for comparison.

SOME APPLICATIONS OF POLARIMETRY

We present here identification of unknown samples of D-fructose, D-sucrose, and D-glucose; quantification of (R)-limonene in citrus ski wax remover; and determination of enantiomeric excess in a mixture of D- and L-arabinose, as well

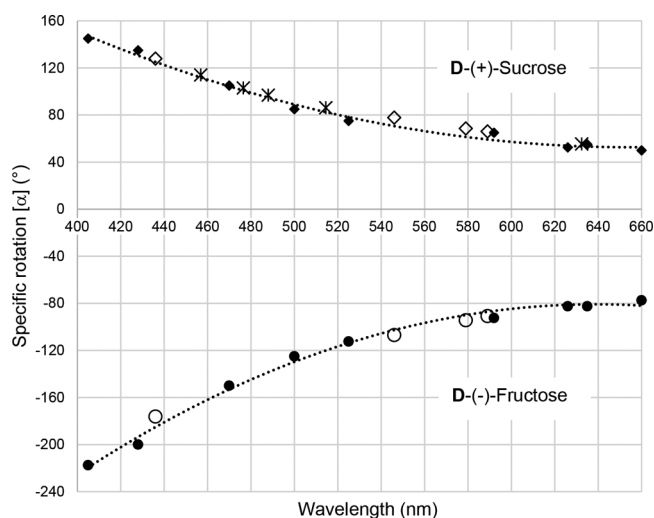


Figure 5. Specific rotation vs wavelength. D-(-)-Fructose and D-(+)-sucrose both at 0.5 g/mL. Self-constructed polarimeter: filled symbols; wavelength = 405, 428, 470, 500, 525, 592, 626, 635, and 660 nm; path length = 4 cm. Anton Paar: open symbols; wavelength = 436, 546, 579, and 589 nm; path length = 10 cm. Extracted values from Mahurin et al.:¹⁶ star symbol.

as discovery of chirality in natural compounds, such as honeys, syrups, and essential oils.

Identification

Identification of unknown solutions of carbohydrates such as glucose, fructose, and sucrose by polarimetry is a common laboratory exercise.²⁰ Unknown solutions of D-fructose, D-sucrose, and D-glucose were measured by students on the self-constructed polarimeter, and by the teacher both on the self-constructed polarimeter and on a commercial polarimeter (Anton Paar). The results are shown in Table 1 together with

Table 1. Optical Rotation Data for Identification of Unknown Sugar Solutions

Solution	Student Result [α] ₅₉₂	Teacher [α] ₅₉₂	Anton Paar [α] ₅₈₉	Literature Value ^a [α] ₅₈₉
(A) D-(-)-Fructose	-94.8° ± 3.3	-93.5°	-93.7°	-92° ± 2
(B) D-(+)-Sucrose	+66.5° ± 2.5	+66.8°	+65.9°	+66.5° ± 1
(C) D-(+)-Glucose	+52.9° ± 2.2	+54.5°	+53.6°	+53° ± 2

^aThe source for these values is Sigma-Aldrich (now Merck). Temperature: 20 °C. Preparation: 10% in H₂O.

literature values. The calculated specific rotations for the student measurements agreed well with both the teacher's results, measurements on a commercial instrument and literature values. Experimental details are given in SI, section 5.3.

Quantification

Quantification has been done by determining the content of (R)-limonene in citrus ski wax remover. Limonene is mostly known as a fragrance and a flavor, but it is also used as a green solvent with a number of different applications including cleaning products, and it is being explored as a starting material and for use in functionalized foods.²¹ The amount of limonene was calculated to be 25% using Figure 2 as the calibrating curve. See SI section 5.4 for experimental details.

Enantiomeric Excess

Determination of enantiomeric excess is an important application in organic chemistry. Most carbohydrates exist in nature almost exclusively as the D-enantiomer making it expensive to purchase the “unnatural” L-enantiomer. Arabinose, on the other hand, exists as both enantiomers in nature. The L-enantiomer is the most common and is found in hemicelluloses, pectin, and gums and is easily obtainable.^{22–26} The D-enantiomer is synthesized from D-glucose^{22,27–29} and can also be purchased at a reasonable price. L-Arabinose is sometimes used as “sugar replacement”, as it apparently inhibits uptake of sucrose.^{23,25,30} Arabinose is harmless and water-soluble and has a high specific optical rotation. It is therefore a suitable compound for investigating quantitative aspects such as enantiomeric excess (ee, most commonly given in %) in a mixture of enantiomers. The results from measuring commercial samples of L- and D-arabinose, a mixture of L- and D-arabinose, together with literature values for the enantiomers are presented in Table 2.

Table 2. Optical Rotation Data of L-(+)- and D-(-)-Arabinose

Solution (20% in H ₂ O)	Optical Rotation α (deg) 592 nm (10 cm)	Specific Optical Rotation $[\alpha]_{592}$ (deg)	Literature Value ^a $[\alpha]_{589}$ (deg)
L-(+)-Arabinose	+21	+105	+103 to +105
D-(-)-Arabinose	-19.5	-97.5	-105 to -103
L-(+)-Arabinose: D-(-)-Arabinose e = 3:1	+10.5	+52.5 ^b	

^aThe source for these values is Sigma-Aldrich (now Merck). Temperature: 20 °C. Preparation: 10% in H₂O for L-(+)-arabinose and 4% in H₂O for D-(-)-arabinose. ^bThe mixture was also measured on an Anton Paar (589 nm) instrument, resulting in a specific optical rotation of +52.1°.

Enantiomeric excess in percent is calculated according to eq 2:

$$\% ee = \frac{[\alpha]_{\text{D mixture}}}{[\alpha]_{\text{D pure enantiomer}}} \times 100\% \quad (2)$$

As seen in Table 2, the specific optical rotation $[\alpha]_{\text{D}}$ of the mixture of L- and D-arabinose is +52.5°, showing an excess of the L-(+)-enantiomer. Using $[\alpha]_{\text{D}} = +105^\circ$ for the L-enantiomer gives ee = 50% which is in accordance with the prepared mixture of 25% L- and 75% D-arabinose. It is simple for the teacher to hand out a range of mixtures and let the students determine the enantiomeric excess (ee) using the self-constructed polarimeter.

Honeys and Syrups

Polarimetry is used frequently in the sugar industry. Having performed measurements with glucose, fructose, and sucrose, it makes sense to continue with an industrial application of polarimetry on sugar-containing compounds such as in analyses of honeys and syrups.^{31–37}

As these viscous liquids can be somewhat nontransparent in the visual (VIS) region of the electromagnetic spectrum but transparent in the near-infrared (NIR) region, NIR polarimetry is preferred for accurate measurements. Nevertheless, it is possible to apply VIS polarimetry semiquantitatively directly.^{31,38} Honeys and syrups rotate plane polarized light both to the left (levorotatory, -) and right (dextrorotatory, +)

mirroring predominantly the amount of D-glucose (+), D-sucrose (+), and D-fructose (-) in them. Honey is a complex mixture. However, fructose dominates in floral honey,^{36,37} and in general, the net optical rotation will be levorotatory. In honeydew, on the other hand, glucose together with other strongly dextrorotatory carbohydrates make the total optical rotation dextrorotatory.^{36,37} Honeydew is not produced from flower nectar like floral honey, but from secretions of living plants or excretions from plant-sucking insects on plants.³⁷ If a honey is dextrorotatory, it could also be adulterated honey.³⁶

In our case (see Table 3), three of the honeys were levorotatory, consistent with floral honeys, while the fourth (Serbian honey) was dextrorotatory and is most likely honeydew.

Table 3. Optical Rotation Data from Some Honeys and Syrups

Type of Honey/Syrup	Calculated Optical Rotation α (deg) for Neat
Kjartan's honey dew/forest honey	-8
Liquid mountain honey European acacia	-6
Kjartan's raspberry honey	-4
Serbian forest honey	+4
Lyle golden syrup	+12.5
Maple syrup Winnitou	+22

Syrups are dextrorotatory containing relatively more sucrose and glucose than fructose; however, there are also different types of syrups. Golden syrups are generally made from refiners' return syrup (leftover liquid with sucrose from production of white sugar from sugar canes) where most of the sucrose is hydrolyzed (inverted) to glucose and fructose.^{39,40} Maple syrup is concentrated maple sap that has not been hydrolyzed and contains mostly sucrose and only small amounts of fructose and glucose.^{41–43} These relative amounts of sucrose, fructose, and glucose are reflected in the optical rotations where Lyle golden syrup has a lower rotation than maple syrup due to the higher content of fructose. See SI section 5.9 for experimental details.

Enantiomers and Essential Oils

Nature is an important pool for chiral compounds. Chiral fragrant compounds are often used in organic chemistry textbooks to explain different activities of enantiomers in biological systems. The significance is of course greater for chiral drugs, but with fragrant compounds the students can themselves experience the difference. Olfactory discrimination of enantiomers is an intriguing field.^{44–47}

Most known are perhaps carvone and limonene. (R)-(-)-Carvone has a sweet mint smell and is naturally found in spearmint oil. (S)-(+)-Carvone has a more spicy smell and is the major compound in oil from caraway seeds.^{44,48,49} Limonene is even more common. (R)-(+)-Limonene is the major component in oil from citrus peel and is in particular associated with oranges. (S)-(-)-Limonene has a more piney and turpentine smell.⁴⁴

Many essential oils contain fragrant chiral compounds, and they rotate plane polarized light both levorotatory and dextrorotatory. Measuring the optical rotation of essential oils shows the diversity of optically active compounds in natural compounds. These oils also lend themselves to investigation, combining a literature study about chiral

Table 4. Optical Rotation of Some Essential Oils at 592 nm in a 4 cm Cuvette

Essential Oil, Species (Supplier)	Optical Rotation α (deg), Neat	Major Chiral Compounds	Literature Value ^a [α] (deg)	Preparation
Eucalyptus oil, <i>Eucalyptus globulus</i> (Karo Pharma)	+2	(+)- α -Pinene ^b	+50.7	Neat
Peppermint oil, <i>Mentha piperita</i> (Karo Pharma)	−9.5 to −10	(−)-Menthol	−49.5	10% in ethanol
		L-(−)-Menthone ^c	−25	Neat
Lavender oil, <i>Lavendula angustifolia</i> (Teleurt)	−3.5 to −4	(R)-(−)-Linalool	−20	Neat
		(R)-(−)-Linalyl acetate	−9.45*	Neat
		(R)-(+)-Camphor	+44.1	25% in ethanol
		(S)-(−)-Camphor	−43	10% in ethanol
		(+)-Borneol ^d	+36	5% in ethanol
Caraway oil, <i>Carum carvi</i> (Carl Roth)	+29	(S)-(+)-Carvone	+55	Neat
		(R)-(+)-Limonene ^e	+115	10% in ethanol
Black spruce oil, <i>Pinus mariana</i> (Teleurt)	−11	(−)-Bornyl acetate	−46	1% in ethanol
		(−)-Camphene	−106.1	4% in ether
		(−)- α -Pinene	−50.7	Neat
		(+)- δ -3-Carene	+17	Neat
		(S)-(−)-Limonene ^f	−94.4	10% in ethanol
Pine needle oil, <i>Pinus sylvestris</i> (Teleurt)	−8	(+)- α -Pinene	+50.7	Neat
		(−)- α -Pinene	−50.7	Neat
		(−)- β -Pinene	−22	Neat
		(−)-Camphene	−106.1*	4% in ether
		(+)- δ -3-Carene	+17	Neat
		(S)-(−)-Limonene	−94.4	10% in ethanol
		(R)-(+)-Limonene ^g	+115.5	10% in ethanol
Orange Douce oil, <i>Citrus sinensis</i> (Florame)	+40	(R)-(+)-Limonene ^h	+115.5	10% in ethanol
Lemon oil, <i>Citrus limon</i> (Florame)	+25	(R)-(+)-Limonene	+115.5	10% in ethanol
		(−)- β -Pinene ⁱ	−22	neat
Mandarin oil, <i>Citrus reticulata</i> (Pima)	+31	(R)-(+)-Limonene ⁱ	+115.5	10% in ethanol

^aThe source for these values is Sigma-Aldrich (now Merck) except for the two marked with an asterisk for which the source is PubChem. Temperature: 20–25 °C. Preparation: as stated in the last column. ^bSee refs 54–56. ^cSee refs 57–59. ^dSee refs 60–65. ^eSee refs 49 and 66–68. ^fSee refs 69–72. ^gSee ref 73–75. ^hSee ref 76. ⁱSee refs 76 and 77.

compounds in oils, their uses, and practical experiments for their extraction.^{50–53} To facilitate exploration, the measured optical rotations of some essential oils together with their major chiral compounds as found in literature reports are given in Table 4. The net rotation is, of course, dependent on all chiral compounds present, and their relative amounts. There is no simple direct correlation between the net rotation of the essential oils and the compounds included in Table 4. The exact amounts of different compounds can vary depending on season, variety, and origin and so may also the optical rotation.

SUMMARY

A simple, inexpensive, and flexible polarimeter was made from two LEDs, a polarizing film, a few wires, a single resistor, a battery, a few Lego bricks, and one multimeter. This polarimeter is used to demonstrate basic properties of polarimetry such as optical rotation as a function of concentration, path length, temperature, and wavelength, as well as to investigate some applications such as determining the enantiomeric excess in a solution of arabinose, the amount of limonene in citrus ski wax remover, and the optical rotations of syrups, honeys, and essential oils.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.9b00763>.

Instructions and patterns for building the polarimeter, and experimental details (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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