NANOYOU Teachers Training Kit in Nanotechnologies

Experiment B – Liquid Crystals

Experiment Module

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MATERIAL INCLUDED IN THIS EXPERIMENT PACKAGE:

For teachers:

TEACHER RESOURCES FOR EXPERIMENT B

For students:

EXPERIMENT B-STUDENT BACKGROUND READING

EXPERIMENT B-STUDENT SYNTHESIS PROCEDURE

EXPERIMENT B-STUDENT LABORATORY WORKSHEET

LEVEL OF EXPERIMENT: Medium

The research leading to these results has received funding from the European Community's Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 233433

TEACHER RESOURCES EXPERIMENT B: LIQUID CRYSTALS

AIM: Liquid crystals (LCs) are an example of **self-assembled molecules** that are sensitive to external factors, such as temperature, and that change their assembly as a consequence of these variations. The effect in some types of LCs is a change of colour. This experiment will show students two fundamental concepts: 1. The way a material behaves at the macroscale depends on its structure at the nanoscale and 2. At the nanoscale LCs are self-assembled molecules that organise themselves into nanostructures which have specific optical properties.

FIELD OF NANOTECHNOLOGY APPLICATION: Fundamental concepts in nanoscience

EXTRA TEACHERS' READING: Chapter 5 "Overview of nanomaterials" in Module 1 of NANOYOU Teachers Training Kit in Nanotechnologies

REQUIRED STUDENT PRE-KNOWLEDGE:
- Electromagnetic radiation, colour and absorbance
- Concept of self-assembly
- Concept of chirality

STUDENT READING:
- NANOYOU Students' background document for Experiment B

EXPECTED OUTCOME:
- Understanding of concept of self-assembly
- Understanding that the way a material behaves at the macroscale is affected by its structure at the nanoscale
- Learn about liquid crystals and how they work
- Test a real thermotropic liquid crystal and see how its colour changes with temperature
- Create a liquid crystal thermometer

STUDENT ASSESSMENT:
-NANOYOU Experiment B-Student laboratory worksheet

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BACKGROUND INFORMATION

Self-assembly

The concept of self-assembly derives from observing that, in natural biological processes, molecules self-assemble to create complex structures with nanoscale precision. Examples are the formation of the DNA double helix or the formation of the membrane cell from phospholipids. In self-assembly, sub-units spontaneously organise and aggregate into stable, well defined structures through non-covalent interaction. This process is guided by information that is coded into the characteristics of the sub-units and the final structure is reached by equilibrating to the form of the lowest free energy. An external factor, such as a change in temperature or a change in pH, can disrupt this organisation. For instance, a protein self-assembles in a specific structure, but if exposed to conditions such as high heat or high acidity, it can denature, which means that its structure is damaged, and the protein unfolds. This means that the protein loses its function as its structure is damaged. So in Nature self-organised structures have specific functions.

Molecules in Nature change conformation and move from one self-organised structure into another as they bind to certain ions or atoms. A lot of examples can be given, like haemoglobulin (which captures and releases an iron ion), or the potassium-sodium pump, chlorophyll, etc.

In this experiment students will see an example of a self-assembled macromolecule, a liquid crystal. Liquid crystals have properties that are directly connected to the way their long molecules self-assemble into nanostructures. Although we cannot see these with our naked eye, we can see macro-scale changes in the optical properties (colour) of the liquid crystal as its temperature is changed. When the temperature of a liquid crystal changes, its molecules self-assemble in a different nanostructure, and this influences the way light is reflected by the liquid crystal, so that a different colour is observed. **Therefore this colour change is a direct consequence of a change in the self-organisation of the liquid crystal molecules.**

What is a liquid crystal?

A liquid crystal is a fourth state of matter: it has properties between those of a conventional liquid and those of a solid crystal. Liquid crystals are partly ordered materials, somewhere between their solid and liquid phases. This means that LCs combine the fluidity of ordinary liquids with the interesting electrical and optical properties of crystalline solids.

Liquid crystals are temperature sensitive since they turn into solid if it is too cold and into liquid if it is too hot. This phenomenon can, for instance, be observed on laptop screens when it is very hot or very cold.

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The molecules in a liquid crystal are often shaped like rods or plates or some other forms that encourage them to **align collectively along a certain direction** (**Figure 1**).

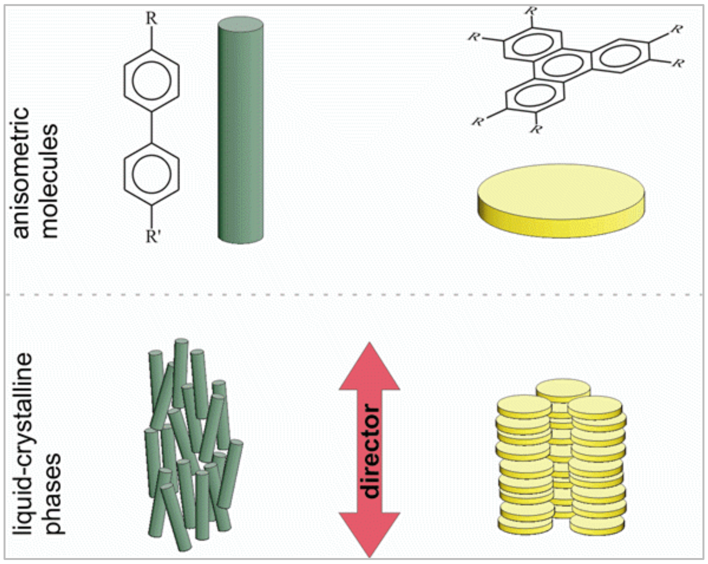

Figure 1. Example of the self-organisation of anisometric (i.e., with asymmetrical parts) molecules in liquid-crystalline phases. On the left: rod-like molecules form a nematic liquid, in which the longitudinal axes of the molecules are aligned parallel to a common preferred direction ("director"). On the right: disc-like (discotic) molecules arrange to molecule-stacks (columns), in which the longitudinal axes are also aligned parallel to the director. As a result of their orientational order, liquid crystals exhibit anisotropic physical properties, just like crystals. (Image Credit: http://www.ipc.uni-stuttgart.de/~giesselm/AG_Giesselmann/Forschung/Fluessigkristalle/Fluessigkristalle.html)

A liquid crystal is formed by the self-assembly of molecules in ordered structures, called *phases.*

An external perturbation, such as a change in temperature or magnetic field, even very small, can induce the LCs to assume a different phase. The molecules in liquid crystal displays for instance are reoriented by rel*atively weak electrical fields.* Different phases can be distinguished by their different optical properties (**Figure 2**).

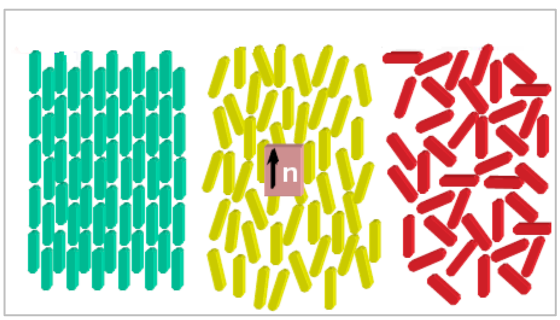

Figure 2. Schematic representation of molecules in a solid (left, molecules are well organised), in a liquid crystal (centre) molecules have a long range distance order) and in a liquid (right) molecules are not ordered. (Image credit: Copyright IPSE Educational Resources, University of Wisconsin Madison)

Liquid crystals (LCs) are divided into three groups:

- **Thermotropic LCs:** they consist of organic molecules, typically having coupled double bonds, and exhibit a phase transition as temperature is changed (**Figure 3, left**)
- **Lyotropic LCs**: they consist of organic molecules, typically hydrophilic (water-loving) and exhibit a phase transition as a function of both temperature and concentration of the LC molecules in a solvent (typically water) (**Figure 3, right**)

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- **Metallotropic LCs**: they are composed of both organic and inorganic molecules, and their LC transition depends not only on temperature and concentration, but also on the organic-inorganic composition ratio.

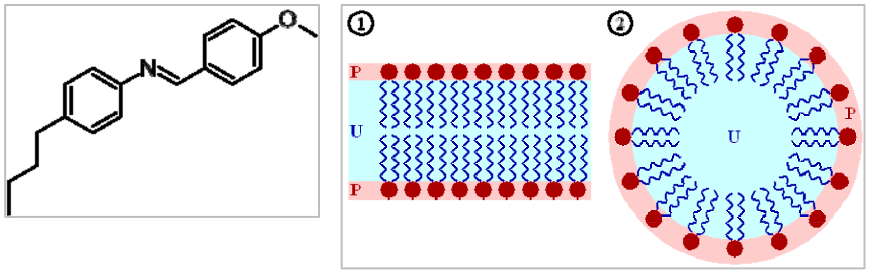

Figure 3. (**Left**) chemical structure of N-(4-Methoxybenzylidene)-4-butylaniline (MBBA); (Right): Structure of lyotropic liquid crystal: 1 is a bilayer and 2 is a micelle. The red heads of surfactant molecules are in contact with water, whereas the tails are immersed in oil (blue). (Image credit: Wiki commons, Creative Commons Attribution ShareAlike 3.0).

Lyotropic liquid-crystalline phases are **abundant in living systems**, such as biological membranes, cell membranes, many proteins (like the protein solution extruded by a spider to generate silk), as well as tobacco mosaic virus. Soap is another well known material which is in fact a lyotropic liquid crystal. The thickness of the soap bubbles determines the colours of light they reflect.

In this experiment module, the students will study the properties of a **thermotropic liquid crystal** meaning that its properties change with changes in temperature.

Liquid crystals that change colour when temperature is changed

Liquid crystal phases that respond to certain temperature ranges are called **thermotropic phases**. Many thermotropic LCs exhibit a variety of phases as temperature is changed, as illustrated in **Figure 4**. The ordering inside a thermotropic liquid crystal exists in a specific temperature range. At high temperatures, the thermal motion will destroy the delicate cooperative ordering of the liquid crystal phase, pushing the material into a conventional isotropic liquid phase. At too low a temperature, most liquid crystal materials will form a conventional (though anisotropic) crystal. These intermediate phases have some level of order, which is progressively lost as the temperature rises. In the smectic phases, which are found at lower temperatures than the nematic, molecules form well-defined layers that can slide over one another. The smectics are thus positionally ordered in one direction. In the nematic phase, molecules have no positional order but they have long-range orientational order. This means that the molecules move quite randomly but they all point in the same direction (within each domain).

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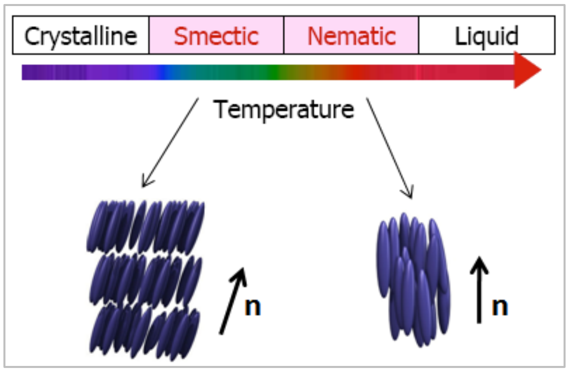

Figure 4. Schematic representation of the structure transition of a thermotropic LC from the smetic to the nematic phase as the temperature is increased. (Image credit: own work adapted from Wiki commons, Creative Commons Attribution ShareAlike 3.0 and from IPSE Educational resources "liquid crystals", University of Wisconsin-Madison.)

What is important to note is that some specific structural and optical properties are associated with each LC phase: **as the temperature is changed, the colour of the LCs changes.** Thus changing the temperature of the material allows moving the material from one phase to the other, so that the material exhibits these different colours. This type of liquid crystal is used in thermometers and in miniaturised temperature sensors (for instance to locate short-circuits on circuit boards).

A particular type of liquid crystal phase is the **chiral nematic phase**. The chiral nematic phase exhibits chirality (handedness). This phase is often called the **cholesteric phase** because it was first observed for cholesterol derivatives. In this experiment students will analyse this type of liquid crystal (**Figure 5**).

Figure 5. Schematic representation of ordering in chiral liquid crystal phases: a chiral nematic phase (also called the cholesteric phase) in an LC (Image credit: Wiki Commons, Creative Commons Attribution ShareAlike 3.0)

Only **chiral molecules** (i.e., those that lack inversion symmetry) can give rise to such a phase. In this phase, the molecules are **stacked in rotating layers, like a spiral staircase (helix)**. In each "step" of the staircase the molecules are arranged in a specific order, but there is a finite angle between each "step" and the next (**Figure 6**).

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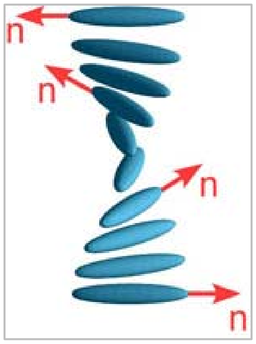


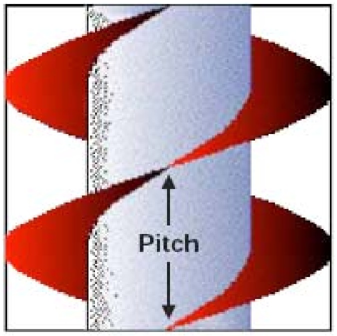
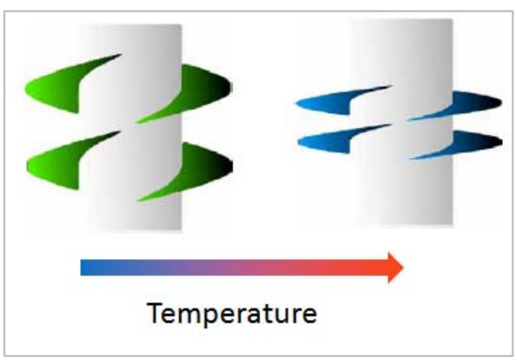


Figure 6. (Left): Schematic representations of stacked rotating layers in a chiral LC forming a "spiral staircase" having a pitch *p*. (Right) schematic representation of the pitch in a chiral LC (Images credit: Wiki commons, Creative Commons Attribution ShareAlike 3.0).

The **chiral pitch, *p*,** refers to the distance over which the LC molecules undergo a full 360° twist. As the temperature of the liquid crystal changes, the pitch changes, which leads to tighter or looser helixes.

What gives the colour to the liquid crystal?

When light strikes a liquid crystal, some of the light is reflected. **What we see is the reflected light**. The colour (i.e. the wavelength) of the reflected light depends on how tightly twisted the helix is. If the pitch in the liquid crystal is of the same order as the **wavelength of visible light (400-700 nm)**, then interesting optical interference effects can be observed. The colour of the light reflected depends on the pitch in the liquid crystal, that is, on **how tightly twisted the helix is**. When the helix is tightly twisted, the pitch is smaller, so it reflects smaller wavelengths (blue end of the spectrum); when the liquid crystal is less twisted, it has a larger pitch, so it reflects larger wavelengths (red end of the spectrum).
An increase in temperature leads to a decrease in the pitch. By increasing the temperature of the liquid crystal one should expect a colour change from the red end of the spectrum to the blue end of the spectrum, so from orange, to yellow, green, blue and violet **(Figure 7)**.



The chiral twisting that occurs in chiral LC phases also makes the system respond differently in right- and left-handed circularly polarised light. *These materials can thus be used as polarisation filter*.

Figure 7. Representation of a pitch change in a chiral LC as the temperature is changed. Image credit: Image adapted from IPSE Educational Resources (Liquid crystals), University of Wisconsin Madison).

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WHAT CAN THIS EXPERIMENT TEACH ABOUT NANOTECHNOLOGY?

The properties of materials at the macroscale are affected by the structure of the material at the nanoscale. Changes in a material's molecular structures are often too small to see directly, but sometimes we can see changes in the material's properties. Liquid crystals are an excellent example, in particular the type used in this experiment, since its optical properties (colour) change visibly as the temperature of the liquid crystal is changed. In nanotechnology, scientists take advantage of the peculiar properties of materials at the nanoscale to engineer new materials and devices.

Liquid crystals are **an example of self-assembled molecules which change their spatial organisation in dependence of external factors** such as temperature. Self-assembly is another fundamental concept in nanoscience.

THIS EXPERIMENT IN CLASS:

1. Start with a discussion on self-assembly. What other molecules self-assemble in organised structures? (e.g., proteins, DNA). *Discuss how the structure is fundamental for the function of the macromolecule.*

2. Talk about the fact that the way a material behaves at the macroscale is affected by its nanostructure. Although we cannot see this with our eyes, we can observe changes in the material properties, such as its colour. Liquid crystals are such an example: they are self-assembled molecules which have specific properties, like colour, depending on the structure they have.

4. Liquid crystals that change colour with temperature. Discuss how they work. Give examples (small flat thermometers, sensors, etc.)

5. Run the experiment in the lab (details below) dividing students into groups of two or more as required.

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OVERVIEW OF SAMPLES TO BE PREPARED

The table below gives quantities of material needed to make four samples of liquid crystal each sensitive to a different temperature interval. Depending on time and class level, students synthesise four different liquid crystal mixtures, or the instructor prepares them in advance for the class.

NB. Synthesis is very quick and takes little time, but it requires weighing 3 different solids four times, which might be time consuming. You can group the class in two, and let each half make only two mixtures, and then ask the students to swap the vials. **Each vial will have enough material for up to 4-6 students (2-3 pairs).**

Table 1.

Liquid crystal	Cholesteryl oleyl carbonate	Cholesteryl pelargonate	Cholesteryl benzoate	Temperature (°C)
Type 1	0.65	0.25	0.10	17-23
Type 2	0.45	0.45	0.10	26.5-30.5
Type 3	0.40	0.50	0.10	32-35
Type 4	0.30	0.60	0.10	37-40

MATERIAL

Below is an indication of material needed for each pair of students

- Cholesteryl oleyl carbonate (Sigma-Aldrich 151157, 25 gr cost about 60 Euros), see table for quantities
- Cholesteryl pelargonate (Sigma-Aldrich C78801, 100 gr cost about 115 Euros), see table for quantities
- Cholesteryl benzoate (Sigma-Aldrich C75802, 25gr cost about 40 Euros), see table for quantities
- 4 glass vials (able to hold 10 mL)
- 1 plastic funnel
- 1 hot plate (no stirring needed) or a heat gun or hairdryer
- Paper to clean
- 1 balance (if possible with 0.01 g resolution)
- Plastic vessels (for measuring solids on the balance)
- 1 glass Pasteur
- Eye protection
- Latex gloves
- 1 spatula

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SAFETY NOTE: Follow school lab safety guidelines. Before using all materials read MSDS sheets carefully. Use these materials with normal chemical precautions according to MSDS. Wear eye protection and gloves. Solids should not be inhaled and contact with skin, eyes or clothing should be avoided. Wash thoroughly after handling. After preparing the liquid crystal, keep in a closed glass vial and do not open and inhale. All experiments will be carried out at your own risk. Aarhus University (iNANO) and the entire NANOYOU consortium assume no liability for damage or consequential losses sustained as a result of the carrying out of the experiments described.

PROCEDURE

1. Synthesis of four different liquid crystal mixtures

- Measure the amounts of solids that are needed using a scale and three different plastic vessels. NB. clean the spatula well between measurements using some paper!

- Mix the three solids in the large glass vial using a plastic funnel. **TIP**: you can secure the funnel on the mouth of the glass vial with clear wrapping paper to make sure the funnel stays in place when the students add the solids.

Note that the solids (particularly cholesteryl oleyl carbonate) are quite sticky so be sure to gently push the solids down the funnel and get as much solid as possible removed from the funnel walls. If lots of solid remains in the spatula or funnel walls, keep both of these in place as you heat up the glass vial, so the solid can melt and you don't lose it.

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- Heat up the glass vial using a hot plate or a heat gun. The hot plate should be set at 185 C. If you don't have either of these, use a hairdryer putting it at the higher temperature and higher flow. It might take a while to induce melting. Be patient! **At the end the liquid crystal should look transparent and with the consistency of honey.**

WARNING: Caution should be taken when using a hot plate or heat gun. These should be operated only in the presence of a teacher. The mouth of a heat gun can become very hot, so do not touch it, do not touch the vial as you heat it, and do not touch it immediately after you have turned the heat gun off. Wait a few minutes before doing so, and always wear gloves.

- While the sample is still liquid gently move the vial around at an angle with your hands (see Figure) so that the liquid crystal spreads around the vial walls.

- **Clearly mark the vial** with a number corresponding to the type of liquid crystal you have made (1 for Type 1 and so on).

- Students should observe the **colour change as the liquid cools down**.

Prepare (or let the students do so) the four different vials and then let the students test them!

Students should follow instructions in the "Experiment B- Students worksheet".

2. Preparation of four liquid crystal sheets and testing in water bath:

MATERIAL:
Below is an indication of material needed for each pair of students

- 4 vials of liquid crystal mixture, each containing a different one
- A water bath (hotplate, Pyrex glass water container half filled with water, thermometer)
- 1 sheet of clear contact paper
- 1 clothes peg
- 1 spatula
- 1 pair of scissors
- 1 A4 sheet of black cardboard
- 1 A4 size sheet of foam

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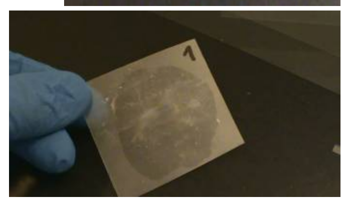
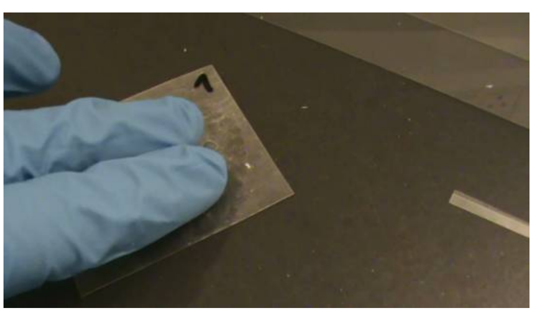
- 1 paper cutter
- Gloves
- Protection glasses
- Tissue paper
- A room thermometer

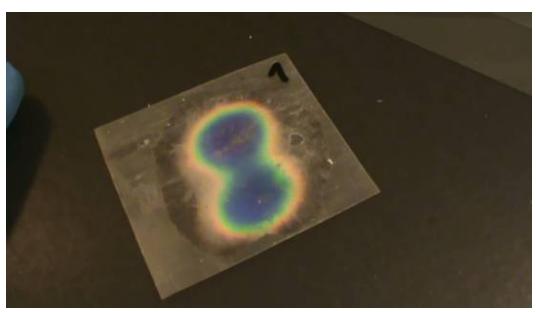


- **Prepare four different crystal liquid sheets.** Cut two pieces of transparent contact paper (about 10x10 cm), peel off the back paper and place on the laboratory bench. With a spatula place some liquid crystal type 1 on the centre of the sheet. If the liquid crystal is very cold and turned into a solid, heat the vial first with a hairdryer (it should be the consistency of honey). You will need 2-3 spatulas of material. Place the second piece of contact paper on top of the first one, so that the two sticky parts attach to each other. As you do so, gently press the middle area where the liquid crystal is and distribute evenly. You need to create a thin layer of liquid crystal about 4x4 cm. Do not press too hard otherwise the material will come out from the edges. Cut the sheet at the end around the edges. Write with a permanent marker on the corner of the sheet a number corresponding to the type of liquid crystal (1 for "Type 1" and so on).

Students should answer Q1 and Q2 in the "Experiment B-student worksheet"

- **Test the liquid crystal sheets with your fingers.** Place the four liquid crystal sheets you have just made on a **white A4 paper**. Wait few seconds. What do you see? Now press with your one finger (wearing gloves) against each of the liquid crystal sheets. Repeat the experiment putting the sheets on **black A4 size cardboard**. In order to compare the different sheets you should keep the finger on each sheet for the same time. Students should record their observations.





Students should answer Q3 and Q4 in the "Experiment B-student worksheet"

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- **Test each liquid crystal sheet in a water bath**. The temperature of the water bath should be initially 15°C, and then gradually increased to reach the working temperature of each liquid crystal mixture.

Liquid crystal	Water temperature range (°C)
Type 1	15-23
Type 2	23-30
Type 3	30-35
Type 4	35-40

Students should **observe the colour change of each liquid crystal as the water temperature increases** (it is better to test each sample one at the time, so students can monitor closely what happens in each sample).

To be able to do so, place a black A4 card safely at the back of the water bath. NB: it should not touch the hotplate!

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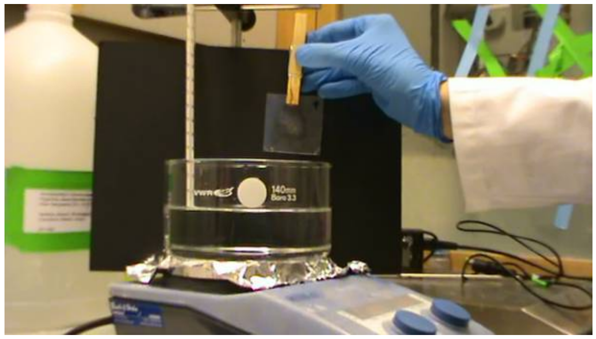

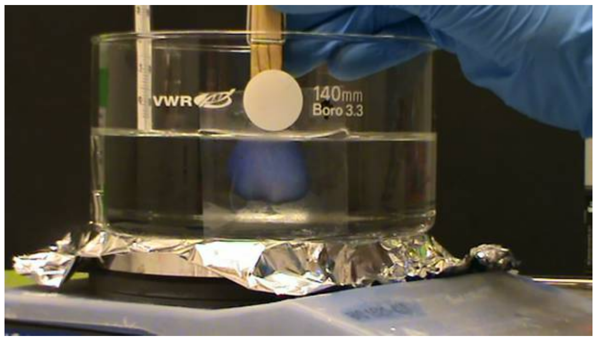

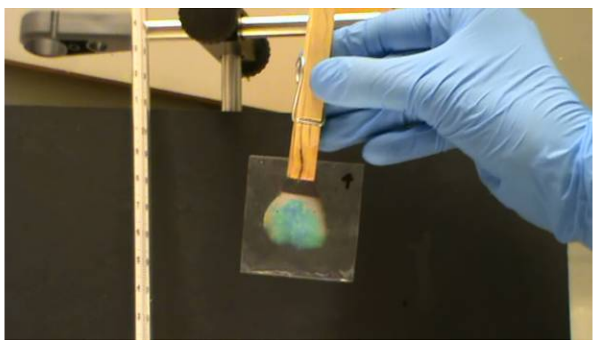

Students should answer Q5 to Q12 in the "Experiment B-student worksheet" as they test the different sheets of liquid crystals.

- To confirm the sensitivity of the liquid crystals to temperature, students should test Sheet 1 (Type 1) at higher temperatures and/or sheet 2 to 4 at lower temperatures (compared to their working temperature).

- When finished, the water bath should be turned off.

- Use **Video 1** if experiment in class cannot be performed, or as an integrative class tool.

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3. Fabrication of a liquid crystal room thermometer

Now you can prepare a **liquid crystal room thermometer**. You can use the liquid crystal sheets made in the previous part of the experiment. You can make new ones if necessary. **Safety note:** wear gloves as you or the students make the thermometer. Be careful not to squeeze the liquid crystal sheets and push the liquid crystal outside the sheet. If this happens, clean immediately with paper.

NB. The material listed above will make 1 thermometer. The teacher can choose to ask each pair of students to make a thermometer, or make just one per class.

- Write on the white foam the word NANO. You will need to "fill" each letter with one liquid crystal sheet, so make sure the single letters are large enough (see picture).

- With a paper cutter, cut the four letters from the foam board.

- Attach a different liquid crystal sheet to the back of each letter, following this order:

N – Type 1
A – Type 2
N – Type 3
O – Type 4

- Secure each letter one at a time using long strips of clear contact paper. Otherwise you can use transparent tape. Make sure that the liquid crystal sheets do no overlap inside the letters. **The idea is that each letter should contain only one liquid crystal sheet**.

- Once you have attached all the liquid crystals sheets, attach the white foam to the black cardboard (placing the side where the sheets are against the cardboard).

- Now you have a room thermometer!

- If no colours are displayed, then the room is at less than 17°C. If you are in a room with a radiator, you can place the thermometer over it… and see what happens!

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- Another test, if your room is too cool to get a response from the thermometer, is to place it over a working laptop computer….it will show what we all know, that laptops heat up!

- You can use your thermometer throughout the year; when is hot take it outside in the sun, or you can place it on the class window.

ANSWERS TO QUESTIONS

Q1 & Q2. Depending on the temperature of the room, answers will be different. If the room is in the 18-20°C range, the sample Type 1 should be able to detect the room temperature (and thus be coloured even without touching it). If the room is below 17°C, none can be used (they will all appear clear at room temperature).

Q3. The colour seen is a consequence of reflected light; therefore a black background is needed to see it. This is why in order to see the colours the students need to place the sample on a piece of black paper.

Q4. In order to display some colour each liquid crystal sheet tested must be touched by an object that has a temperature in its working temperature range. Our hands are no more than 36 C warm, so Type 3 and Type 4 crystal liquid will not show any colour. If you rub the sheet (rather than just pressing it with the finger) you might generate enough heat to warm it up to the working temperature, and hence see a colour. Most likely this will still not be enough for Type 4.

Q5. Sheet 1 (Type 1) liquid crystal will start to display some colour around 17°C. Since the reading of the thermometer is subject to error, and the temperature of the sheet is will most likely not be identical to that of the water bath, the colour might appear few °C before or after 17°C. If this is reported by the students, it should be critically discussed.

Q6. All liquid crystal sheets will display a colour gradient throughout their working temperature window which is directly related to the wavelength scale of visible radiation. The colour gradient will be from the red end of the spectrum to the blue end of the spectrum (as temperature increases). This is because the colour of the light reflected depends on the pitch in the liquid crystal, that is, on how tightly twisted the helix is. When the helix is tightly twisted, the pitch is smaller, so it reflects smaller wavelengths (blue end of the spectrum); when the liquid crystal is less twisted, it has a larger pitch, so it reflects larger wavelengths (red end of the spectrum). An increase in temperature leads to a decrease of the pitch, so on increasing the temperature of the liquid crystal one should expect a colour change from the red end of the spectrum to the blue end of the spectrum, from orange, to yellow, green, blue and violet.

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Q7. When the crystal liquid sheet Type 1 is removed from the water bath it gradually changes colour. If the room where the experiment is being performed has a temperature of less than 17°C, it will gradually become transparent.

Q8. The temperature of the water bath is around 17°C.

Q9. No, because 30°C is outside the Type 1 working temperature (which is 17-23 C).

Q10. Yes, it is the same for the reasons explained in the answer to Q6.

Q11. Sheet 3 and Sheet 4 are sensitive to higher temperatures than Sheet 1. When they are taken out of the water bath they lose their colour very quickly because the room temperature is totally out of their working temperature range. On the other hand, Sheet 1 has a working temperature range which includes room temperature (normally RT is around 18°C), so Sheet 1 will be much slower in losing its colour and will possibly retain some colour (if RT is between 17 and 23°C).

Q12. Type 4, because it has a working temperature range equal to the fever range (37-40°C).

Q13. If no colour appears on the first “N” it means that the room is at a temperature lower than 17°C.

CREDITS:

This experiment was adapted from the activity “Preparation of Cholesteryl Ester Liquid Crystals” available at http://mrsec.wisc.edu/Edetc/nanolab/LC_prep/index.htm” and from the “Exploring materials: Crystal Liquids” activity developed by the NISE network (Creative Commons Attribution ShareAlike 3.0). The activity was developed for the NISE Network with funding from the National Science Foundation under Cooperative Agreement #ESI-0532536. Any opinions, findings, conclusions or recommendations expressed in this report are those of the authors and do not necessarily reflect the views of the Foundation.

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