# Preparation of Cholesteryl Ester Liquid Crystals

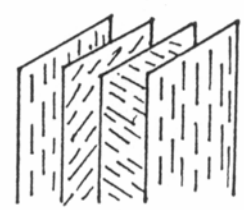
The procedure for temperature sensitive liquid crystals is based on G. H. Brown and J. J. Wolken, *Liquid Crystals and Biological Systems*, Academic Press, NY, 1979, pp. 165-167 and W. Elser and R. D. Ennulat, *Adv. Liq. Cryst.* **2**, 73 (1976). This experiment is from the Lab Manual for Nanoscale Science and Technology [http://mrsec.wisc.edu/Edetc/nanolab/LC_prep/index.html](http://mrsec.wisc.edu/Edetc/nanolab/LC_prep/index.html)
The procedure for pressure sensitive liquid crystals is based on Griffiths, Jonathan S., *Experimental Chemistry*, Stockton State College, Pomona, NJ, 1988
Additional information and procedures added by David A. Katz, Department of Chemistry, Pima Community College

Liquid crystals are organic compounds that are in a state between liquid and solid forms. They are viscous, jelly-like materials that resemble liquids in certain respects (viscosity) and crystals in other properties (light scattering and reflection). Liquid crystals must be geometrically highly anisotropic (having different optical properties in different directions)-usually long and narrow - and revert to an isotropic liquid (same optical properties in all directions) through thermal action (heat) or by the influence of a solvent. Liquid crystals are classified as:

smectic: molecules arranged in horizontal layers or strata and are standing on end either vertically or at a tilt.

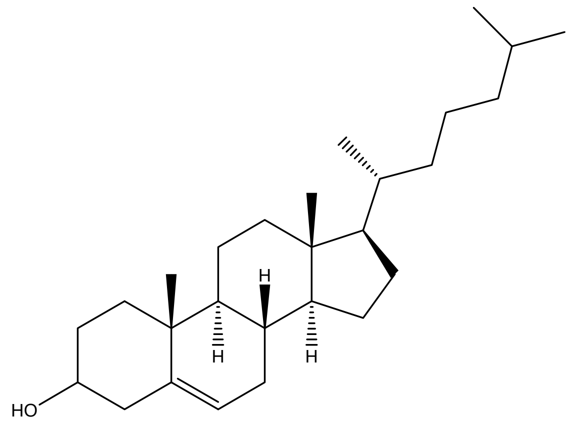
nematic: molecules possess a high degree of long-range order with their long axes approximately parallel, but without the distinct layers of the smectic crystals.

lyotropic: molecules consist of a nonpolar hydrocarbon chain with a polar head group. In a solvent, such as water, the water molecules are sandwiched between the polar heads of adjacent layers while the hydrocarbon tails lie in a nonpolar environment.



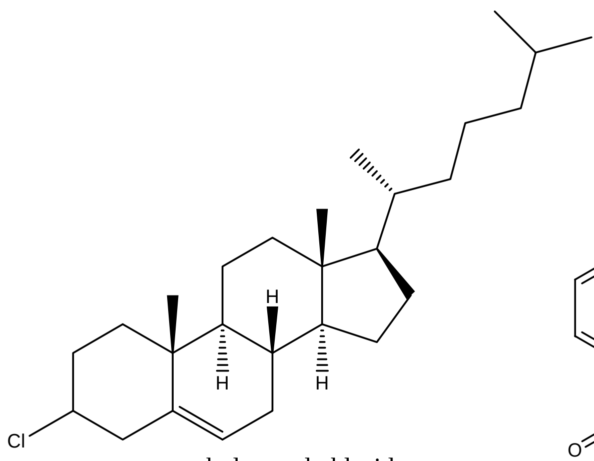
If smectic and nematic liquid crystals are subjected to changes in temperature, they change their form and their light transmission properties splitting a beam of ordinary light into two polarized components to produce the phenomenon of double refraction. This results in the appearance of the characteristic iridescent colors of these types of liquid crystals. This type of liquid crystal finds use in thermometers, egg timers, and other heat sensing devices. Changes in structure can also be accomplished using a magnetic field which make them useful in calculator or other LCD displays. Temperature sensitive liquid crystals were used in Mood Rings.

When lyotropic liquid crystals are subjected to disturbances, such as stirring or squeezing, the layers of crystals are disturbed altering their light transmission characteristics to produce color changes similar to the smectic and nematic liquid crystals described above. These are the type of liquid crystals used in the Press Me stickers.

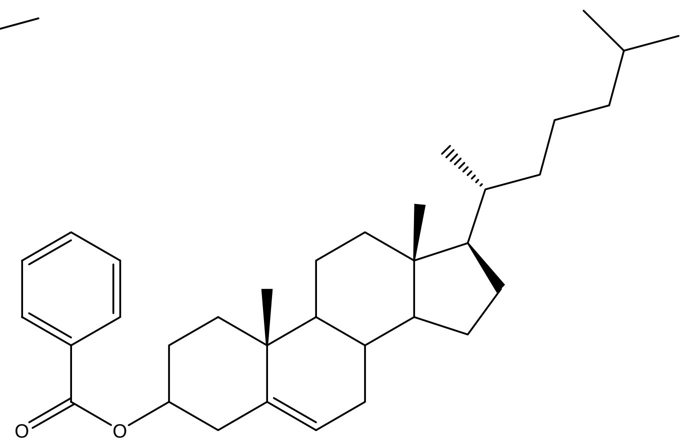


cholesterol

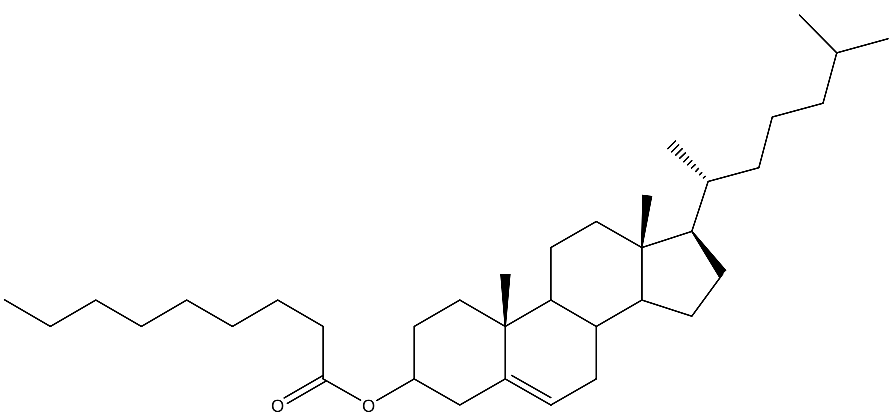
Common liquid crystals are composed of derivatives of cholesterol, $C_{27}H_{46}O$. Some common liquid crystal materials are cholesteryl chloride, $C_{27}H_{45}Cl$, cholesteryl benzoate, $C_{34}H_{50}O_2$, cholesteryl pelargonate, $C_{36}H_{62}O_2$, (also called cholesteryl nonanoate), and cholesteryl oleyl carbonate, $C_{46}H_{80}O_3$.



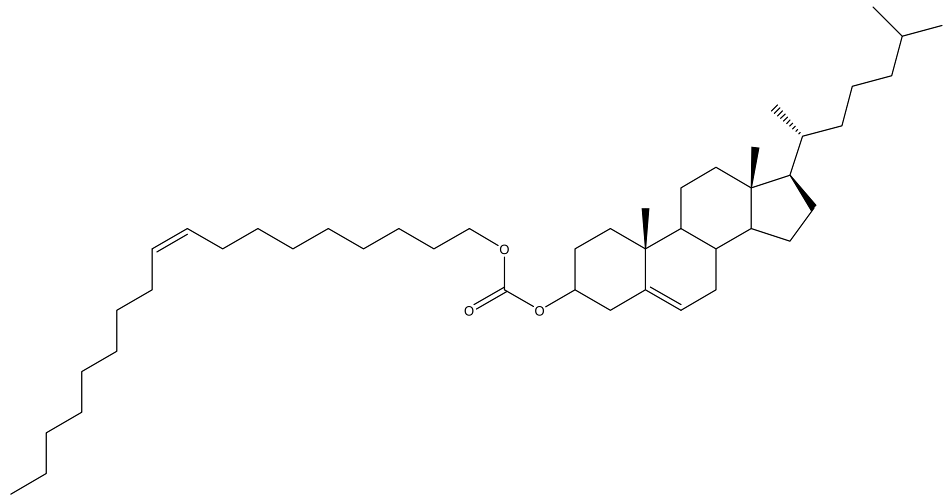
cholesteryl chloride



cholesteryl benzoate



cholesteryl pelargonate



cholesteryl oleyl carbonate

These cholesteric-nematic liquid crystals reversibly change color as the temperature changes. One of the principal advantages of liquid crystals is their ability to map out thermal regions of different temperature. Liquid crystal films exposed to the atmosphere will decompose slowly; their lifetime can be extended by encapsulation.

## Materials Needed

cholesteryl chloride
cholesteryl benzoate
cholesteryl pelargonate (also called cholesteryl nonanoate)
cholesteryl oleyl carbonate
applicator sticks
black plastic (use a heavy plastic trash bag)
Petri dishes
Plastic shipping tape, 2 inches wide
scissors
Glass vials
Funnels, to fit glass vials
Heat gun
Thermometer, 120°C

## Safety Precautions

Wear approved eye protection at all times in the laboratory.

Cholesteryl liquid crystal materials may cause irritations to skin or eyes.  In the event of contact, rinse well with fresh water..

## Disposal

Dispose of all materials in the proper waste containers.

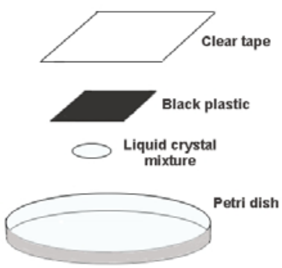
## Procedure

### A. Pressure sensitive liquid crystals

This procedure makes a **pressure sensitive** liquid crystal mixture.

Place 0.38 g cholesteryl oleyl carbonate, 0.38 g cholesteryl pelargonate, and 0.25 g cholesteryl chloride in a vial.

Melt the solid in a sample vial using a heat gun.




Use a wood applicator stick to place a small amount of the liquid crystal mixture to the inside of a Petri dish. Place a piece of black plastic, approximately 2.5 cm square, on top of it and seal it in place with a 5 cm square of clear plastic shipping tape.

Press on the liquid crystal mixture from the tape side while viewing the top of the Petri dish. What happens?

You can repeat this procedure making another mixture of pressure sensitive liquid crystals by varying the amounts of cholesteryl oleyl carbonate, cholesteryl pelargonate, and cholesteryl chloride. The total mass of the mixture of the three compounds should be approximately 1.0 g.

Mixture composition tried:

cholesteryl oleyl carbonate ___________ g

cholesteryl pelargonate ___________ g

cholesteryl chloride ___________ g

What are the results of your mixture?

## B. Temperature sensitive liquid crystals

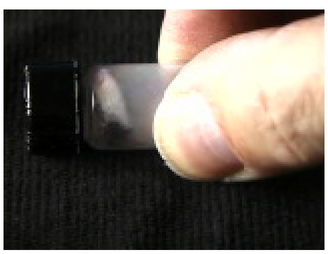
This procedure makes a **temperature sensitive** liquid crystal mixture.

Speak with other groups in your lab class. Each group should select a different mixture of cholesteryl oleyl carbonate, cholesteryl pelargonate, and cholesteryl chloride from Table 1.

Weigh the liquid crystal materials and place them in a glass vial.

Melt the solid in the vial using a heat gun.

The product changes color as it cools. Different compositions give different color patterns over different temperature changes.

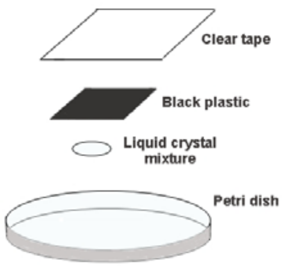


Touching the vial changes the temperature and results in color changes.

Can you measure the temperature change of the liquid crystal mixture?

Heat the sample with the heat gun to melt the liquid crystal mixture.

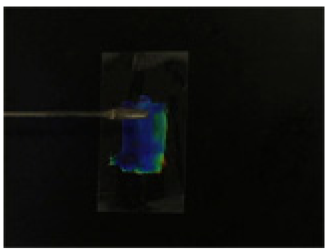
Use a wood applicator stick to place a small amount of the liquid crystal mixture to the inside of a Petri dish.  Place a piece of black plastic, approximately 2.5 cm square, on top of it and seal it in place with a 5 cm square of clear plastic shipping tape.




Gently warm or cool the liquid crystal mixture from the tape side while viewing the top of the Petri dish.  Note the temperature when changes occur.  What happens?

Share your liquid crystal mixture with other groups in your lab class.

**Alternative method:**



Use a wood applicator stick to transfer and spread the liquid on the sticky side of a piece of packing tape. Cover with a second piece of tape, sticky sides together. Trim as necessary.
The black plastic is optional.

**Table 1.** Chloesteryl liquid crystal mixtures and their transition temperatures. Different compositions give different color patterns over different temperature ranges.

| Cholesteryl oleyl carbonate | Cholesteryl pelargonate | Cholesteryl benzoate | Transition range, degrees C |
|---|---|---|---|
| 0.65 g | 0.25 g | 0.10 g | 17-23 |
| 0.70 g | 0.10 g | 0.20 g | 20-25 |
| 0.45 g | 0.45 g | 0.10 g | 26.5-30.5 |
| 0.43 g | 0.47 g | 0.10 g | 29-32 |
| 0.44 g | 0.46 g | 0.10 g | 30-33 |
| 0.42 g | 0.48 g | 0.10 g | 31-34 |
| 0.40 g | 0.50 g | 0.10 g | 32-35 |
| 0.38 g | 0.52 g | 0.10 g | 33-36 |
| 0.36 g | 0.54 g | 0.10 g | 34-37 |
| 0.34 g | 0.56 g | 0.10 g | 35-38 |
| 0.32 g | 0.58 g | 0.10 g | 36-39 |
| 0.30 g | 0.60 g | 0.10 g | 37-40 |

## Questions

### A. Pressure sensitive liquid crystals

1. Describe the behavior of the pressure sensitive liquid crystal mixture.

2. If you or another group tried a different mixture of pressure sensitive liquid crystal material:

   a) What was the composition of the mixture you used?

      cholesteryl oleyl carbonate ___________ g

      cholesteryl pelargonate ___________ g

      cholesteryl chloride ___________ g

   b) How did this mixture of liquid crystal materials behave?

3. If you or another group tried a different mixture of pressure sensitive liquid crystal material:

a) What was the composition of the mixture?

cholesteryl oleyl carbonate ___________ g

cholesteryl pelargonate ___________ g

cholesteryl chloride ___________ g

b) How did this mixture of liquid crystal materials behave?

4. If you or another group tried a different mixture of pressure sensitive liquid crystal material:

a) What was the composition of the mixture?

cholesteryl oleyl carbonate ___________ g

cholesteryl pelargonate ___________ g

cholesteryl chloride ___________ g

b) How did this mixture of liquid crystal materials behave?

5. Can you attribute any differences in behavior to a specific component of the liquid crystal mixture?

## B.  Temperature sensitive liquid crystals

1. a) What was the composition of the temperature sensitive liquid crystal mixture you used?

   cholesteryl oleyl carbonate ___________ g

   cholesteryl pelargonate ___________ g

   cholesteryl benzoate ___________ g

   b) What was the measured temperature change for this liquid crystal mixture?

   c) How did this mixture of liquid crystal materials behave?

2. If you or another group tried a different mixture of temperature sensitive liquid crystal material:

   a) What was the composition of the liquid crystal mixture?

   cholesteryl oleyl carbonate ___________ g

   cholesteryl pelargonate ___________ g

   cholesteryl benzoate ___________ g

   b) What was the measured temperature change for this liquid crystal mixture?

   c) How did this mixture of liquid crystal materials behave?

3. If you or another group tried a different mixture of temperature sensitive liquid crystal material:

a) What was the composition of the liquid crystal mixture?

cholesteryl oleyl carbonate ___________ g

cholesteryl pelargonate ___________ g

cholesteryl benzoate ___________ g

b) What was the measured temperature change for this liquid crystal mixture?

c) How did this mixture of liquid crystal materials behave?

4. If you or another group tried a different mixture of temperature sensitive liquid crystal material:

a) What was the composition of the liquid crystal mixture?

cholesteryl oleyl carbonate ___________ g

cholesteryl pelargonate ___________ g

cholesteryl benzoate ___________ g

b) What was the measured temperature change for this liquid crystal mixture?

c) How did this mixture of liquid crystal materials behave?

5. Can you attribute any differences in behavior to a specific component of the liquid crystal mixture?