NANOTECHNOLOGY EXPERIMENTS FOR GENERAL CHEMISTRY LABORATORY CLASSES

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- **Nanotechnology**
  - **Major area of research and development**
  - **Only now being introduced into textbooks for general chemistry**
  - **Almost no inclusion in the student laboratory.**
- **Lab procedures and kits developed at the Materials Research Science and Engineering Center (MRSEC) at the University of Wisconsin-Madison** http://mrsec.wisc.edu/Edetc/index.html

  (Go to video lab manual)

Courses

CHM 121IN, Chemistry and Society

CHM 125IN, Consumer Chemistry

- **Non-major courses**
- **Taught as hands-on active learning courses**
- **Experiments introduced in 2003**

CHM 151-152IN, General Chemistry

ENG 110IN, Solid State Chemistry

- **LED and solar cell experiments included in laboratory**

Nanoparticles
Murano Glass

- **The Venetian island of Murano is located 1.5 km from the city of Venice.**
- **Murano originated its glassmaking in 8th-century Rome with significant Asian and Muslim influences.**
- **Murano glass is made up of 70% silica sand, with 30% of other substances called “fluxes” and "stabilizers" (soda and lime).**
- **The colors are obtained by adding small amounts of minerals, oxides, and chemical derivatives to the base composition of the glass powder.**

cobalt **copper** **chromium** **iron** **manganese** **gold**

A Ruby-Red Colloidal Gold demonstration kit is available from Flinn Scientific

Ruby Red Gold Nanoparticles

Measure 20 mL of 1mM hydrogen tetrachloroaurate solution ($HAuCl_4$) into a flask.

Heat to a gentle boil on stirring hotplate

Add 2 mL 1% trisodium citrate solution, $Na_3C_6H_5O_7 \bullet 2H_2O$

Observe color changes approximately 2 to 10 minutes

Shine laser through solution to show colloid

Addition of 5 to 10 drops of 1M NaCl solution will cause the gold nanoparticles to agglomerate forming a blue colored mixture. Those particles will settle out of the mixture.

References:

- A. D. McFarland, C. L. Haynes, C. A. Mirkin, R. P. Van Duyne and H. A. Godwin, "Color My Nanoworld," *J. Chem. Educ.* (2004) **81**, 544A.
- Hydrogen tetrachloroaurate trihydrate, approx. US$122/g Sigma Aldrich

Kit: Product no. AP7117, available from Flinn Scientific Inc., (A cost effective alternative)

Silver Nanoparticles

Two preparations:

0.001 M silver nitrate, $AgNO_3$, with 0.002 M sodium borohydride, $NaBH_4$
http://education.mrsec.wisc.edu/nanolab/silver/index.html

1 mM silver nitrate, $AgNO_3$, with 10 mM trisodium citrate, $Na_3C_6H_5O_7 \bullet 2H_2O$
http://www.lawrencehallofscience.org/sites/default/files/pdfs/college_resources/modules/SilverNanoparticles.pdf

Color varies with particle size 5 – 80 nm and concentration.

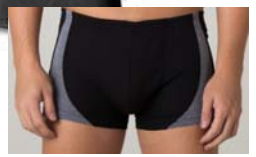
https://www.linkedin.com/pulse/20141027215533-104501454-what-kind-of-colloidal-silver-should-one-look-for

Golden silver nanoparticles kit: Product no. AP7483, available from Flinn Scientific Inc.

Silver Nanoparticles

Antimicrobial properties

- **Food storage containers and packaging**
  - **Banned in some areas due to migration of nanoparticles into food.**
  - **Silver nanoparticles can bind with DNA and interferes with DNA replication.**
- **Clothing**
  - **Silver nanoparticles may be released into human sweat.**
  - **Particles do not appear to be released into washing machines.**
- **Medical applications**
  - **Used for treatment of wounds and burns.**
  - **Mixed information on interactions and effects.**



"Golden silver nanoparticles" kit: Product no. AP7483, available from Flinn Scientific Inc.

Aerogels

By definition, an aerogel is an open-celled, mesoporous, solid foam that is composed of a network of interconnected nanostructures and that exhibits a porosity (non-solid volume) of no less than 50%.
Pore sizes range from 2 to 50 nm in diameter.

Bulk densities range from 0.0011 to ~0.5 g cm^{-3}.

An aerogel is over 98% air.

Aerogels can be made from silica, most of the transition metal oxides (e.g., iron oxide), most of the lanthanide and actinide metal oxides (e.g., praseodymium oxide), several main group metal oxides (e.g., tin oxide), organic polymers (such as resorcinol-formaldehyde, phenol-formaldehyde, polyacrylates, polystyrenes, polyurethanes, and epoxies), biological polymers (such as gelatin, pectin, and agar agar), semiconductor nanostructures (such as cadmium selenide quantum dots), carbon, carbon nanotubes, and metals (such as copper and gold)

Aerogel was first created by Samuel Stephens Kistler in 1931, as a result of a bet with Charles Learned over who could replace the liquid in "jellies" with gas without causing shrinkage.

Aerogels are produced by extracting the liquid component of a gel through supercritical drying.

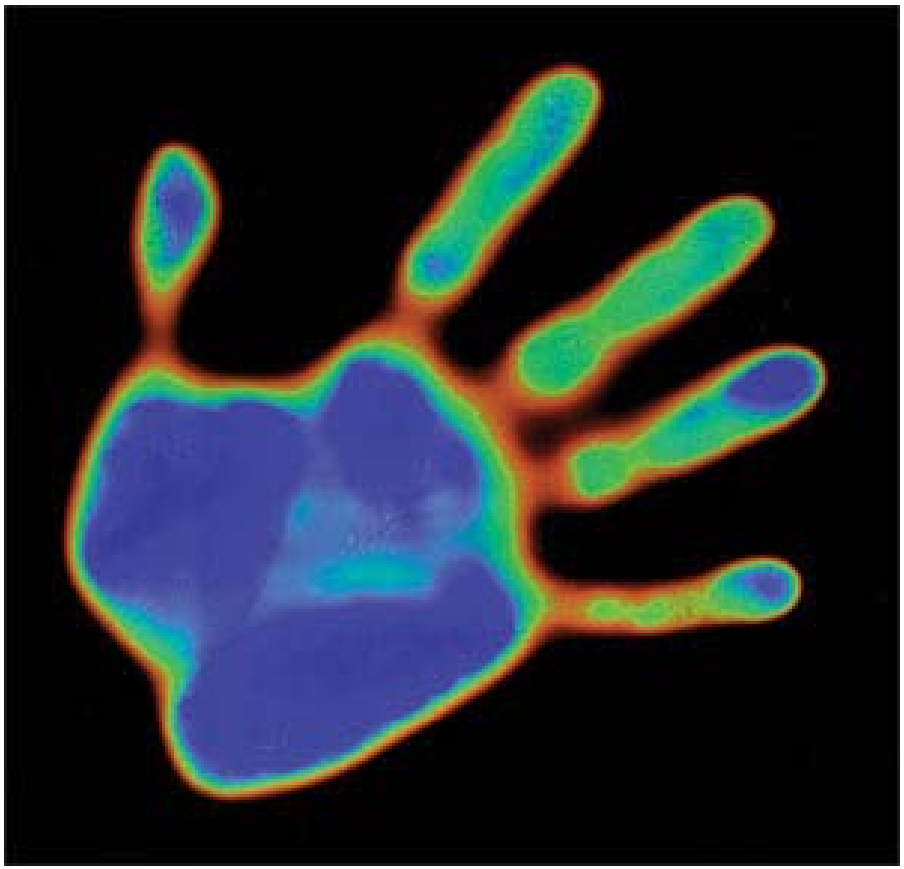
Aerogels are good thermal insulators.

Mood Rings

Dark blue: Happy, romantic or passionate
Blue: Calm or relaxed
Blue-green: Somewhat relaxed
Green: Normal or average
Amber: A little nervous or anxious
Gray: Very nervous or anxious
Black: Stressed, tense or feeling harried

Liquid Crystals

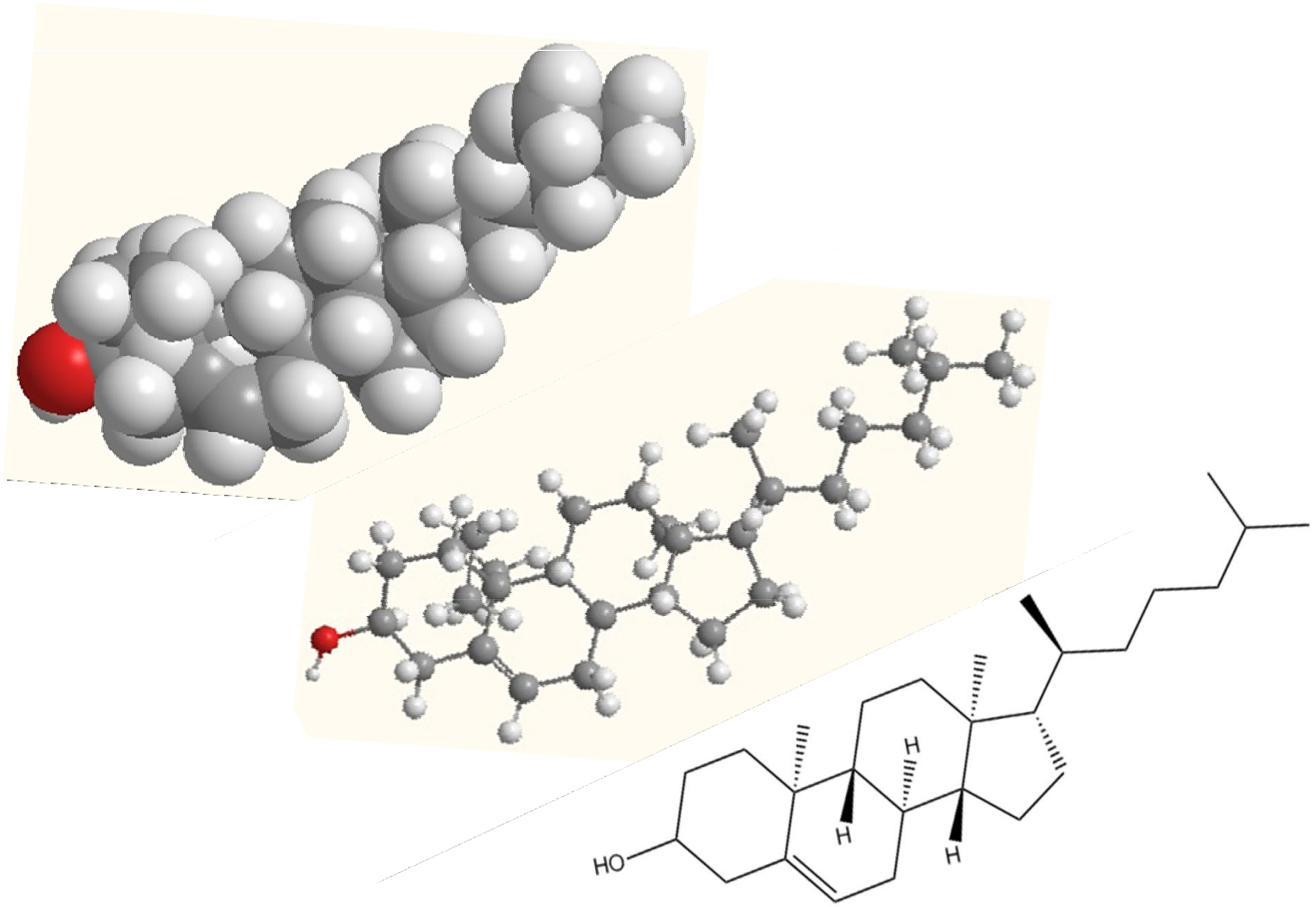
Both pressure sensitive and temperature sensitive (thermochromic) mixtures are prepared



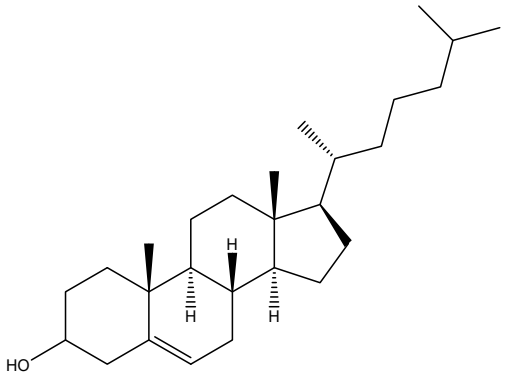
Liquid Crystals

- **Organic compounds in a state between liquid and solid**
- **Viscous, jelly-like materials that resemble liquids in viscosity and crystals in light scattering and reflection**
- **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.**

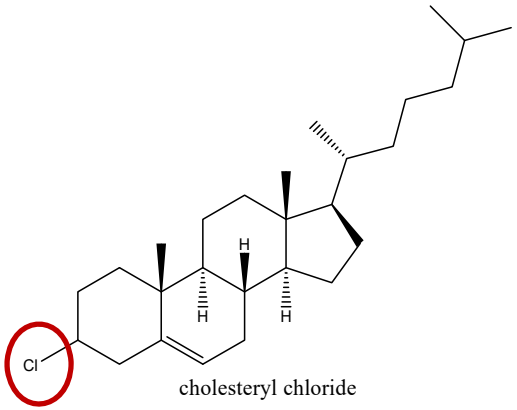
Cholesterol – three models



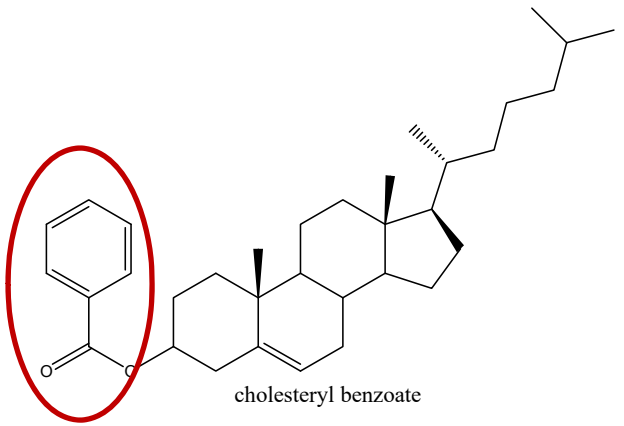
Cholesteryl Ester Liquid Crystals



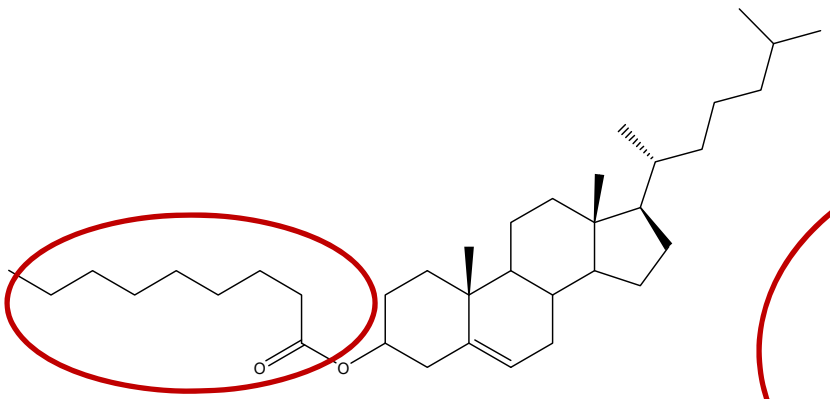
cholesterol



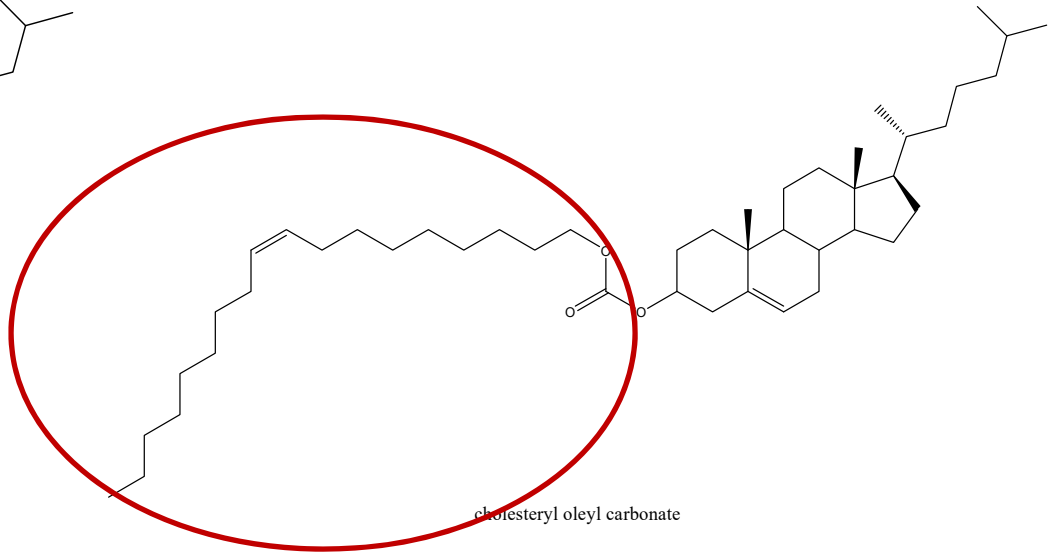
cholesteryl chloride



cholesteryl benzoate



cholesteryl pelargonate



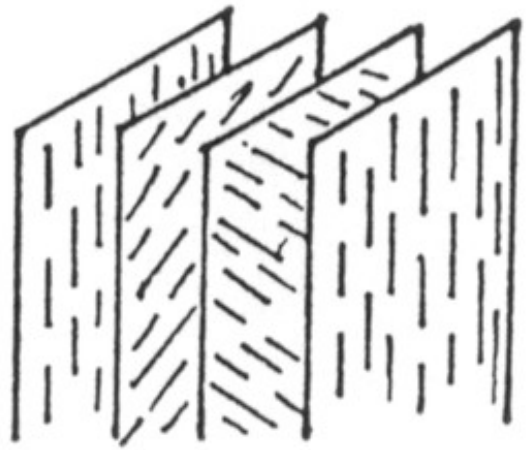
cholesteryl oleyl carbonate

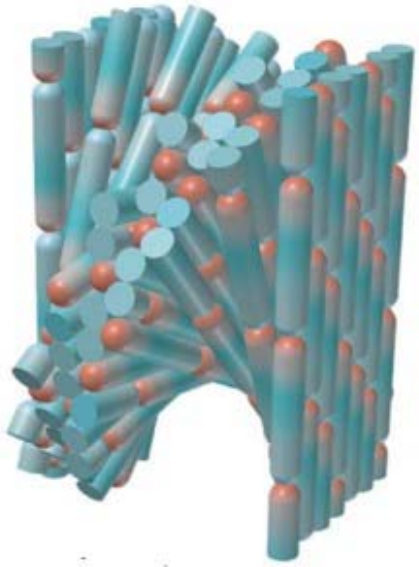
Types of Cholesteryl Liquid Crystals

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.



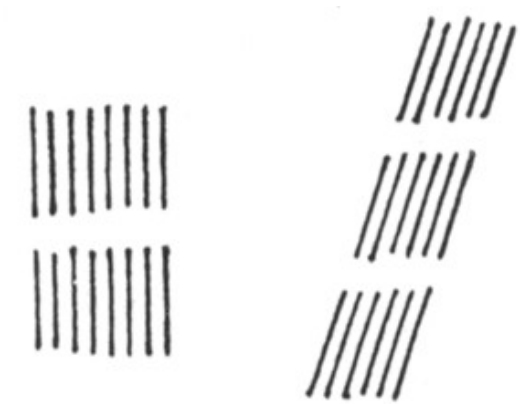


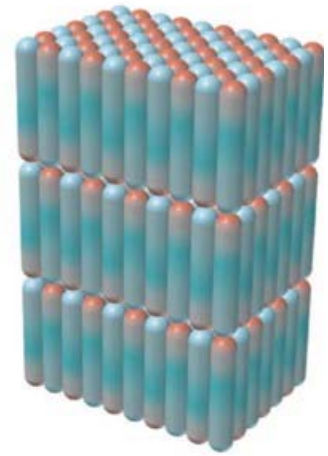
These tend to be pressure and temperature sensitive

Types of Cholesteryl Liquid Crystals

Smectic

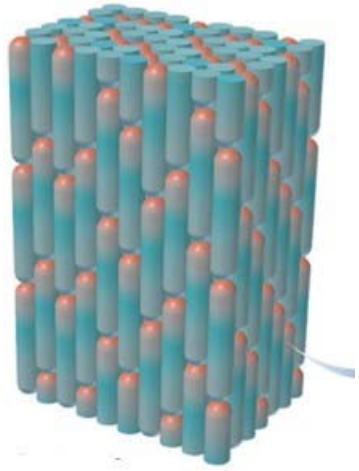
Molecules arranged in horizontal layers or strata and are standing on end either vertically or at a tilt.





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.



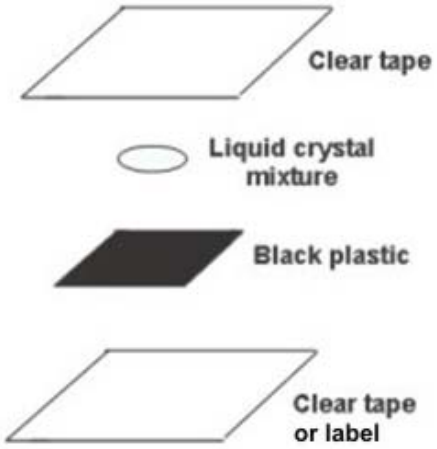
These are temperature sensitive

Cholesteryl Ester Liquid Crystals

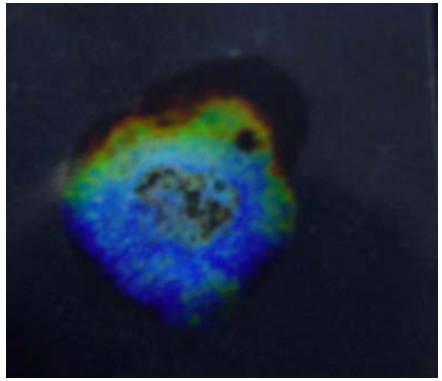
Mix liquid crystal materials

Melt the material

Allow to cool



Make a liquid crystal sticker.



Temperature Transition of a Mood Ring

Dark blue: Happy, romantic or passionate

Blue: Calm or relaxed

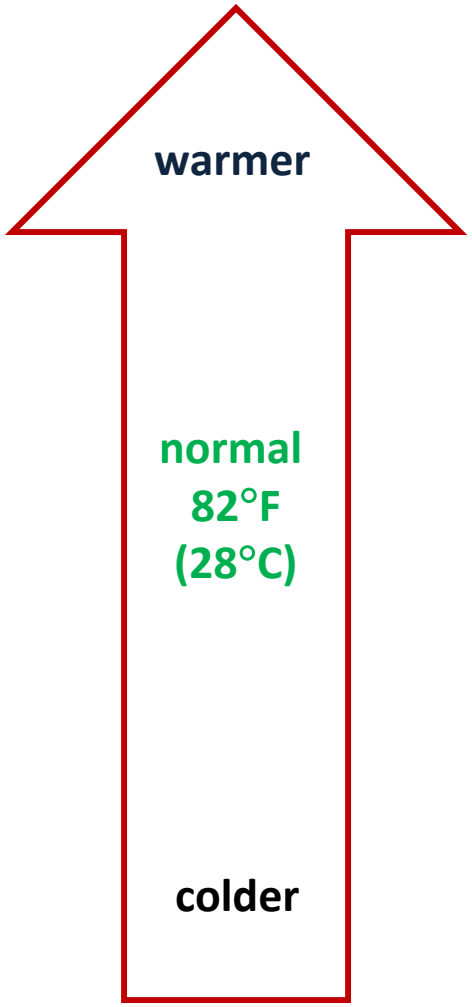
Blue-green: Somewhat relaxed

Green: Normal or average

Amber: A little nervous or anxious

Gray: Very nervous or anxious

Black: Stressed, tense or feeling harried

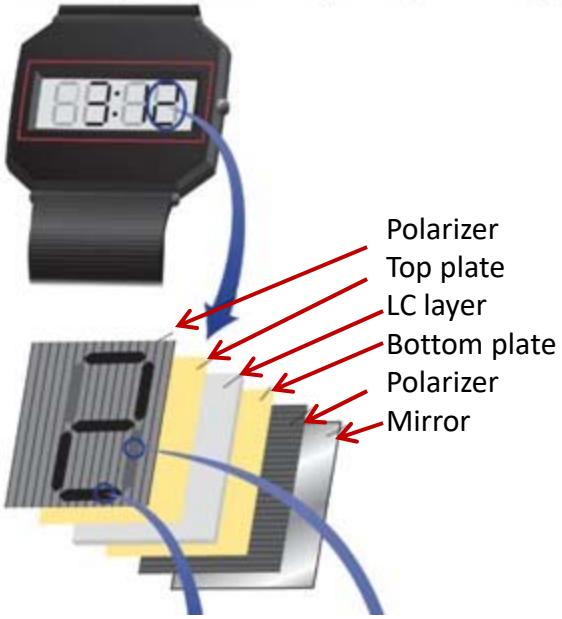


Calibrate a Mood Ring

Suspend a mood ring in a water bath with a magnetic stirrer and temperature probe.

Slowly heat the bath to determine transition colors.

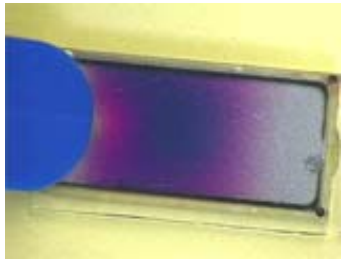
Investigation of an LCD watch display



Take watch apart

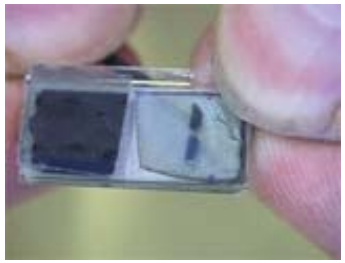
View with polarizer

Remove electronics and display



Display is touch sensitive

Determine transition temperature

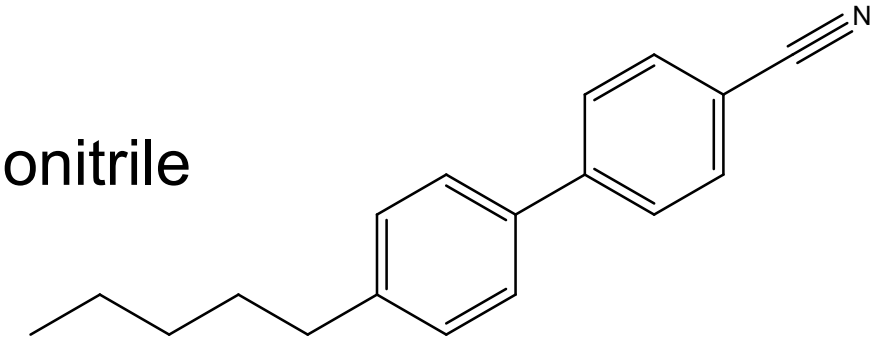


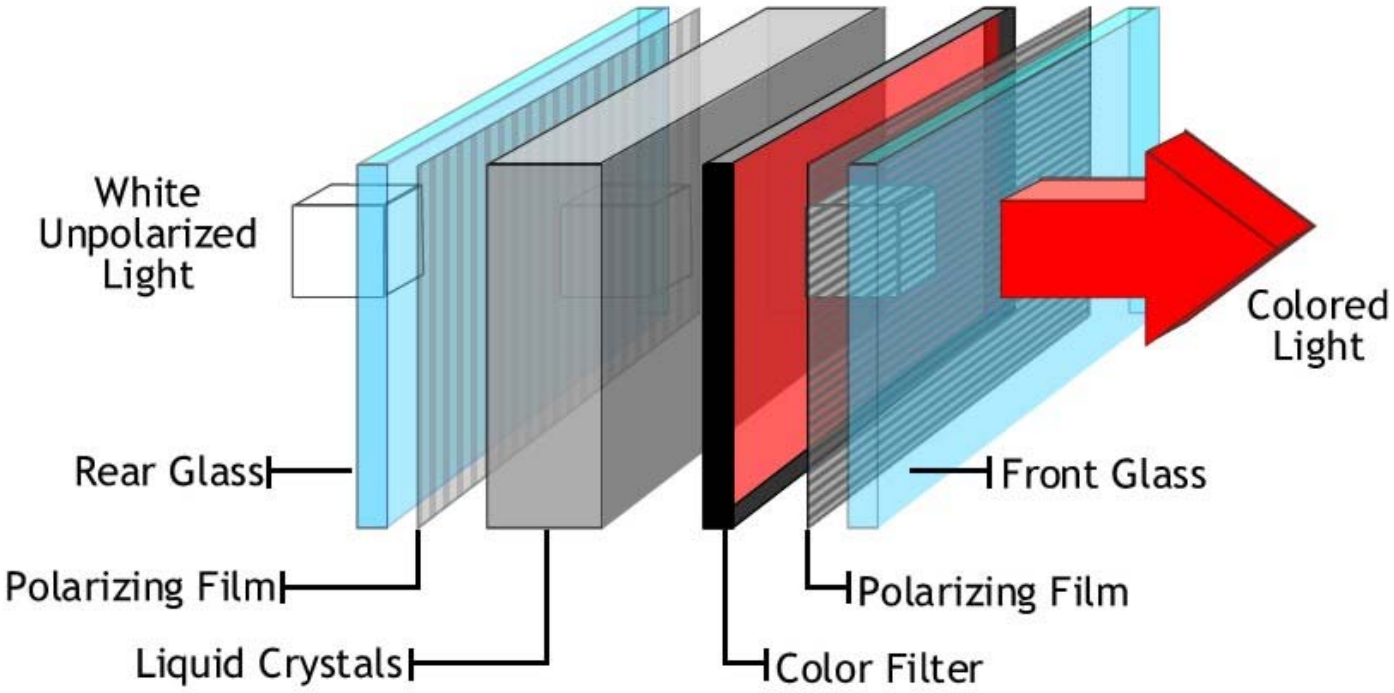
Cut and rotate part of top polarizer

Purchase LCD watch at a dollar store.

Liquid Crystal “Pixel”

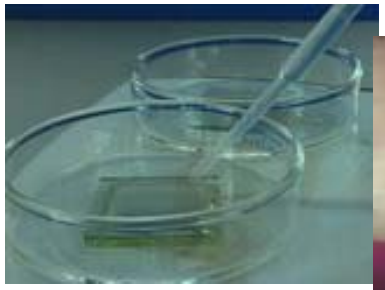
Uses 4’-pentyl-4-biphenyl-carbonitrile





Liquid Crystal “Pixel”

Prepare polyvinylalcohol solution

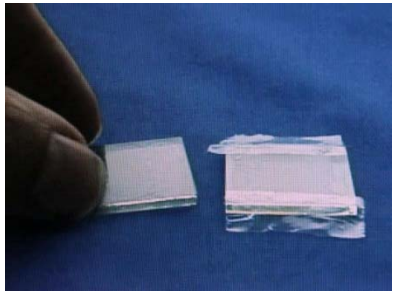


Coat conductive glass

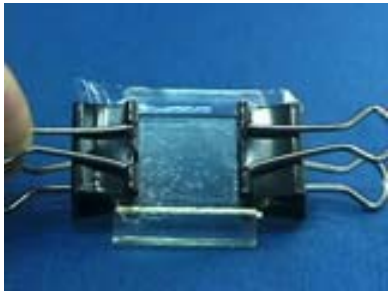
Wipe surface

Clean edge

Prepared glass plate

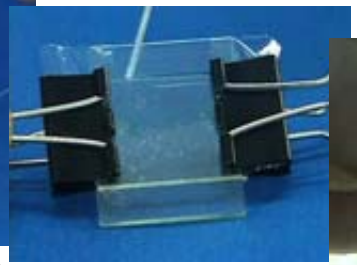


Plastic film spacers

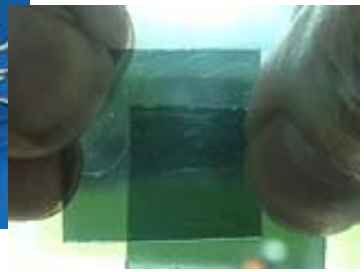


Clamp together

4’-pentyl-4-biphenyl-carbonitrile



Add to “pixel”



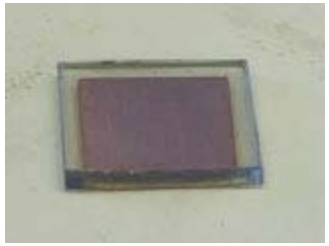
Add polarizing filter

Attach 9-V battery.
Finished “pixel”

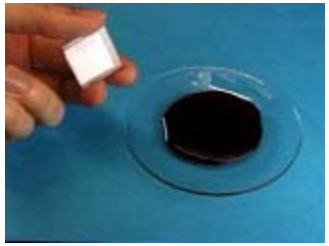
Titanium Dioxide Raspberry Solar Cell

Grind nanocrystalline TiO_2 with dilute acetic acid

Coat surface conducting glass



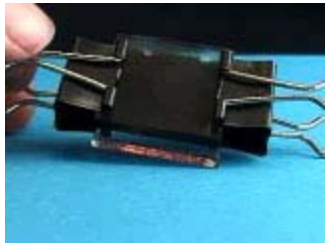
Bake coating on hot plate



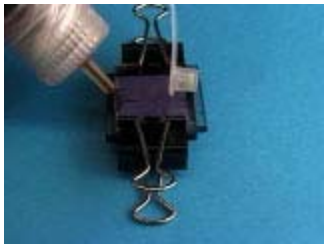
Dip into berry juice

Rinse

Coat 2nd piece of glass with carbon



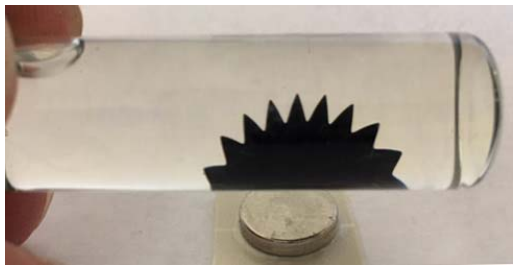
Clamp together



Dope with KI_3 solution

Measure voltage

Aqueous Ferrofluid



- Colloidal suspensions of magnetic nanoparticles.
- Responds to an external magnetic field
- Fe_2O_3 magnetite nanoparticles can be produced by mixing Fe(II) and Fe(III) salts together in a basic solution.
- Surfactants are used to prevent the nanoparticles from approaching one another too closely.
- Ferrofluids exhibit “spikes” when placed in the proximity of a strong magnet.

Aqueous Ferrofluid

Mix $FeCl_2$ and $FeCl_3$
(note colors of solutions)

React with aqueous NH_3

Allow to settle

Decant liquid and transfer solid to a weighing boat

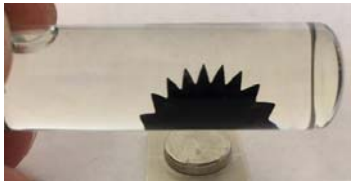
Rinse with water and tetramethylammonium hydroxide

Place a magnet under the Ferrofluid to observe magnetic spikes

Store in 35% 2-propanol

Make a Ferrofluid Display

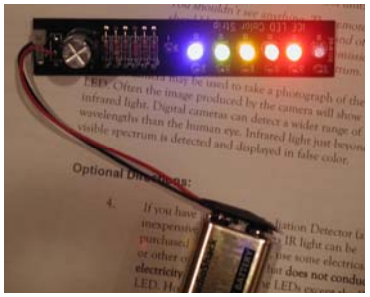
- **Ferrofluid: FeroTec EFH-1 (www.ferrotec.com)**
- **Use a clean glass vial or small narrow bottle**
- **Add 35% isopropyl alcohol (2-propanol) to vial**
- **Add Ferrofluid using a dropper**
- **Top off with additional isopropyl alcohol (leave a small bubble)**
- **Close vial tightly**
- **Seal with shrink tubing**

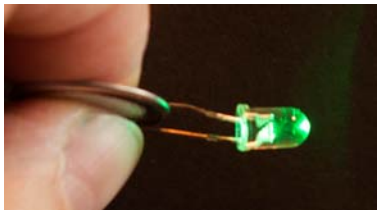


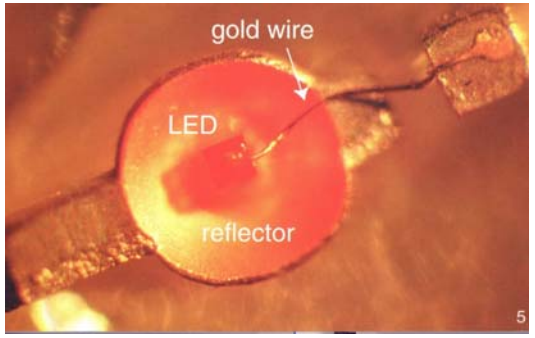
LED's

Experiments:

- **Observe diode behavior**
- **Determine relative wavelength of light**
  - **View through diffraction grating**
- **Determine relative energies of different colored LED's**
- **Determine effect of temperature**
  - **Immerse in liquid nitrogen**
- **Measure voltages**
- **Control light path with an optical fiber**
- **Apply LED light to a luminescent material**







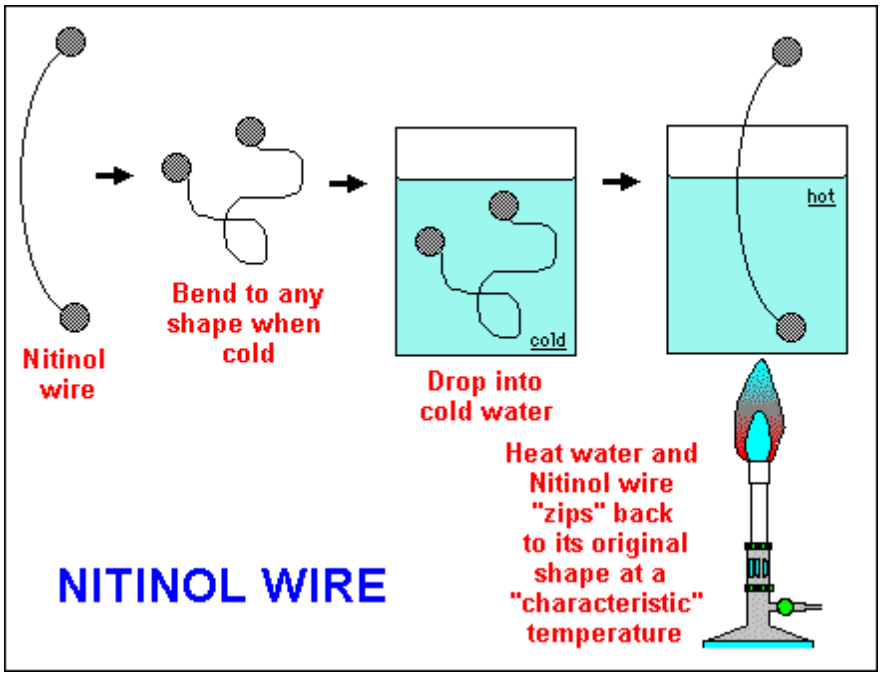
LED Color Strip Kit available from ICE, University of Wisconsin

Nitinol

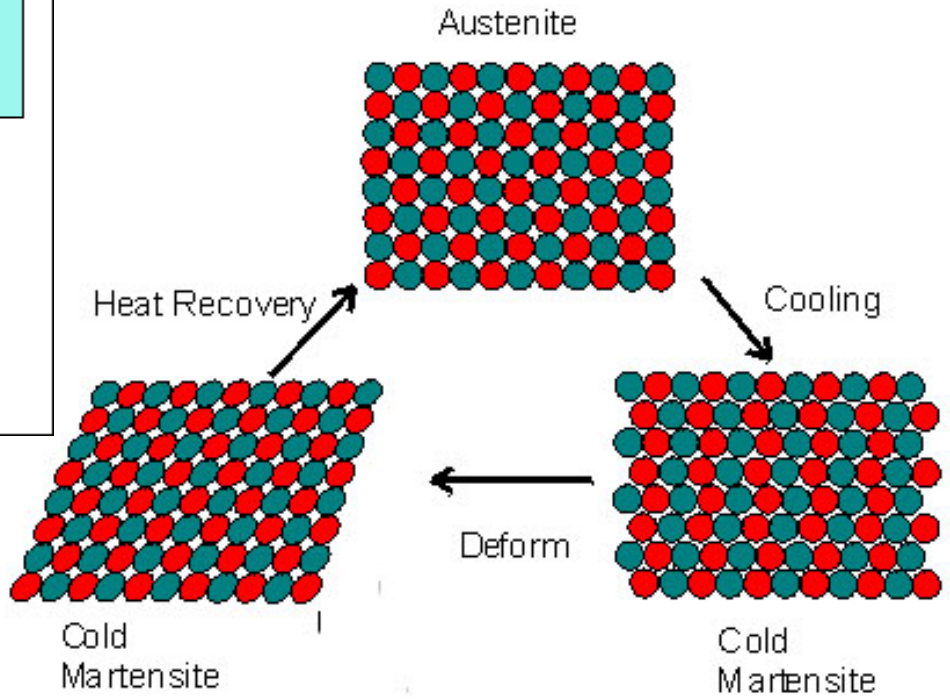
Nickel-Titanium-Naval Ordance Laboratory

Discovered in 1958 by metallurgist William J. Buehler.

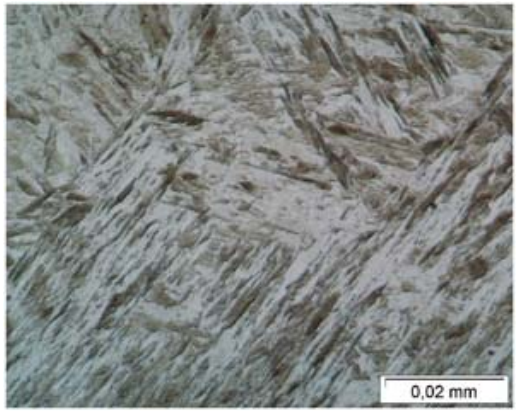
He was seeking an alloy for missile nose cones that could better withstand reentry.



- Explore properties.
- Train wire into spiral (or other) shape.



The **Martensite phase** is named after the German metallurgist Adolf Martens (1850–1914), who, in the 1890s, found that the hardest steels had a regular crystalline structure of plate shaped crystal grains. The martensite is formed by rapid cooling (quenching) of austenite which traps carbon atoms (in carbon steel) that do not have time to diffuse out of the crystal structure.



The **Austenite phase** is named after Sir William Chandler Roberts-Austen (1843-1902). In this high temperature phase, iron changes it crystal structure resulting in a softer and ductile form of iron.

Nitinol

The Thermobile, a Nitinol motor

works by immersing the bottom edge of the brass pulley into hot water between 50°C and 75°C. Within a few seconds, the Thermobile will begin to spin continuing as long as the bottom is at a temperature above 50°C.

A Nitinol butterfly

uses Nitinol wire, consisting of approximately 50% each of nickel and titanium

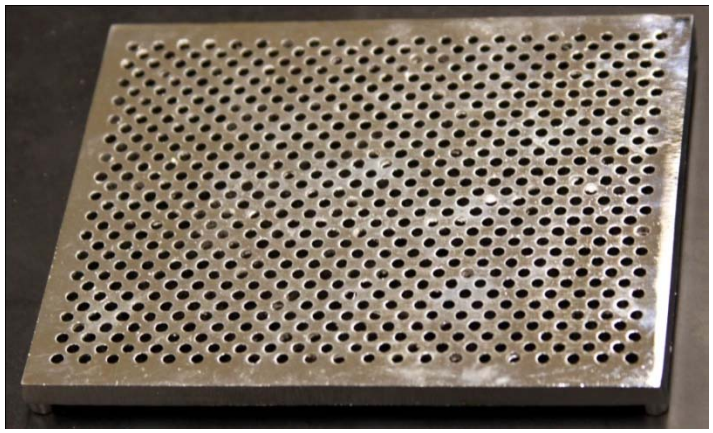
(manufactured under the name BioMetal®)

This alloy has been stretched and, when heated, will shorten to its original shape.

Memory Metal

A sample of Nitinol wire in the shape of ICE
(Available from ICE, University of Wisconsin)

Training Nitinol Wire

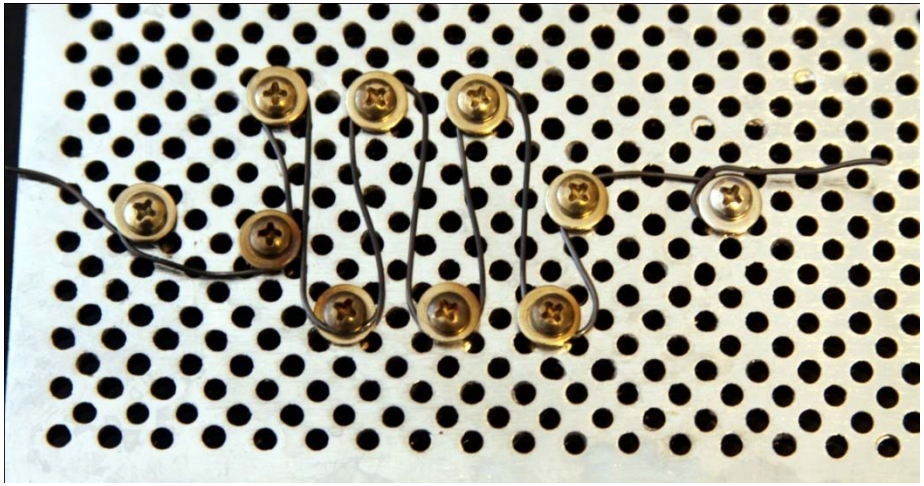


Obtain a wire bending plate

This is a Beadalon Thing-a-ma-Jig composed of heavy aluminum.

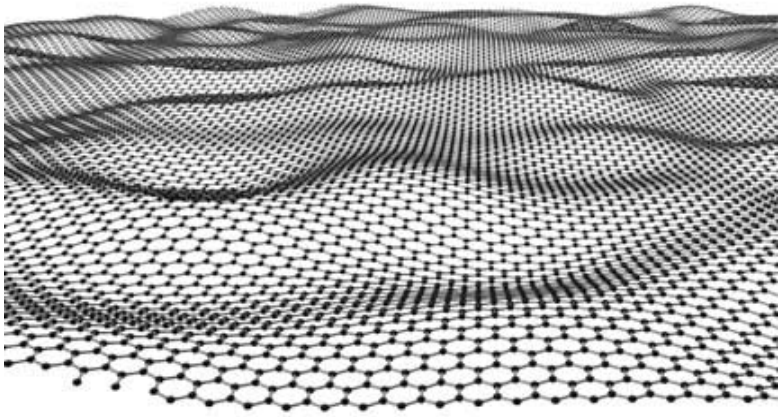
(Purchased from Amazon.com)

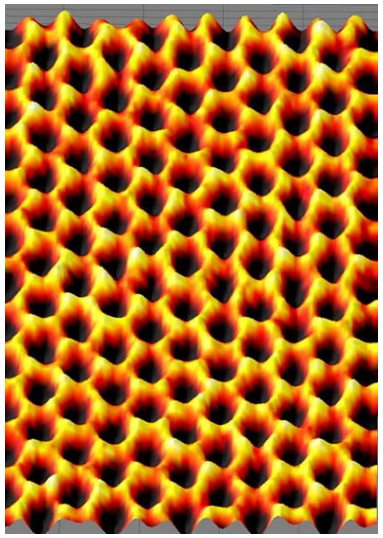
Fasten Nitinol wire in place with stainless steel screws and washers

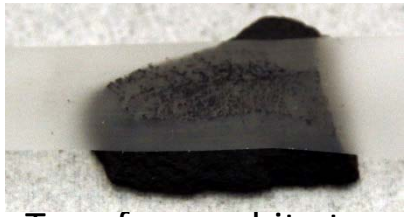


Heat with a mini blow torch or use a small furnace at 500°C

Graphene



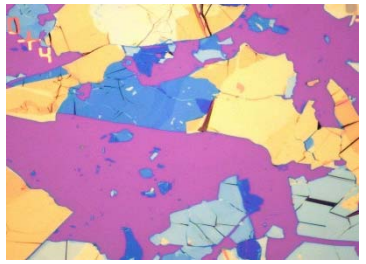




Transfer graphite to Scotch tape

Peel layers apart

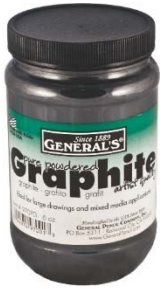
Transfer to a silicon wafer (I found some on eBay)



View under a microscope (lots of debris)

Graphene in a Blender

Method developed by Jonathan Coleman and colleagues at Trinity College Dublin. Graphene flakes 300-800 nm in size were made.



Best done in a high-power (400-watt) kitchen blender.

Use about 500 mL water, 10-25 milliliters of detergent and 20-50 grams of graphite powder.

Run the blender for 10-30 minutes.

Yield may be increased by decanting water, adding fresh water and detergent, and blending again.

Graphene can be collected by filtering.

Graphene/graphite will re-aggregate if left to settle in the blender jar.

Note: Blender jar will probably not be useable for food applications after this experiment.

Sources

Cholesteryl liquid crystals: Sigma-Aldrich Chemical Co.

Flinn Scientific has a demo kit using 2 cholestryl liquid crystals

Nanocrystalline titanium dioxide solar cell kit: ICE – Univ. of Wisconsin (get one kit only)

Aqueous Ferrofluid - chemicals and kits: Flinn Scientific and Sigma-Aldrich

LED Color Strip Kit: ICE-Univ. of Wisconsin (get one kit only – additional materials from an electronics store)

Nitinol wire: Images Scientific Instruments and ICE

Nitinol Butterfly: Images Scientific Instruments

Beadalon wire bending plate: Craft store or Amazon.com

Mini-torch: Harbor Freight Tools

Silicon wafers: eBay (It may take time to find them at a reasonable price.)

Web sites:

http://www.chymist.com

Click on Laboratory Experiments on left-hand menu.

Materials Research Science and Engineering Center (MRSEC) at the University of Wisconsin-Madison

http://mrsec.wisc.edu/Edetc/index.html

Go to video lab manual

Note: See The Nano Song

http://science360.gov/obj/tkn-video/a16c0a32-da1c-4c50-b455-6f166c5f2f96