**Essential Oil Extraction using Liquid CO2**

**HIGH SCHOOL**

**Green Chemistry & Sustainable Science**

*This lesson plan was based on a laboratory experiment McKenzie, Lallie C.; Thompson, John E.; Sullivan, Randy; Hutchison, James E.* ***Green chemical processing in the teaching laboratory: a convenient liquid CO2 extraction of natural products.*** *Green Chemistry (2004), 6(8), 355-358.*

**Teacher Background Information:**

Many fruits and vegetables contain essential oils, which are water repellent or **hydrophobic liquids** that give the fruit or vegetable its distinctive fragrance. These essential oils are often extracted for use in perfume, cosmetics, food, medicine and house cleaning products. Many of these essential oils are extracted through liquid chemical extraction using dangerous chemical solvents, such as methylene chloride. Conventional methods used to extract essential oils include steam distillation or liquid chemical extraction. Steam distillation requires high energy input as energy is required to boil water to produce steam. The energy used combined with the dangers of heating large amounts of matter on an industrial level means that this process does not adhere to the principles of green chemistry. This is an important component of teaching students about green chemistry as Green Chemistry is not just a concept used in the lab but a concept meant to be used on an industrial scale to make products which are useful to the world. Steam distillation may seem like a benign process until it is evaluated against the 12 principles on an industrial scale.

Scientists have discovered the use of carbon dioxide (CO2) at elevated pressure is an alternative method of extracting essential oils and that is the process which you will discover with your students through this activity. In this lesson liquid CO2 will be used to extract an essential oil, simulating industrial processes that use supercritical CO2 as an extraction solvent. Supercritical CO2 is the point at which carbon dioxide reaches a “triple point” and the CO2 maintains properties of both a liquid and a gas. This unique phase allows for highly efficient separations in an industrial setting. Since reaching the supercritical point of CO2 is not feasible within a classroom setting, liquid CO2 will be generated to act as the extraction solvent in this lesson. This will allow for the observation of a phase change, as well as demonstrating the use of CO2 as an extraction solvent.

It is important to note that the use of CO2 for extraction does not affect the net amount of CO2 in the environment, thus using liquid CO2 for essential oil extraction is not considered to affect climate change in any way. Instead, the use of liquid CO2 is considered a greener way of essential oil extraction since it reduces the amount of energy input and eliminates the need for dangerous solvents. Because CO2 does not have high reactivity with essential oils, which can lead to the breakdown of the essential oil, its use in essential oil extraction is gaining popularity. Currently, supercritical CO2 is used to remove caffeine from coffee beans to produce decaffeinated coffee and as a replacement for perchloroethlyene in dry cleaning applications.

In this experiment, students will extract the essential oil d-limonene from the rind (skin) of lemon peels using both a steam distillation or simple distillation method and the method of using liquid CO2. They will analyze the difference between the two methods and make connections between the laboratory activities they do in the classroom and the industrial chemical processes that are used to make products. D-limonene gives lemons, oranges and limes their citrus-like scent.

**Please refer to additional teacher background info and safety considerations at the end of this lesson.**

**\* The steam distillation can be a demo lab and the liquid CO2 a hands on students lab if you feel that you only have one class period to cover this material.**

**Educational Goal:** To understand chemical, steam and CO2 extraction methods and their relationship to green and industrial chemistry practices.

**Student Objectives:** Students will …

* Extract essential oils from lemons using steam distillation
* Extract essential oils from lemons using liquid CO2
* Compute the use of energy in both extractions
* Compare the use of energy in both extractions
* Compare the use of hazardous chemicals in both extractions
* Learn about phase changes of CO2
* Learn about extraction methods based on polarity

**Materials: (per lab group -3 students)**

**Steam Distillation**

* Watt’s up meter (optional)
* 1 Lemon
* 1 pair of scissors
* Scale
* Weigh boat or weigh paper
* Spatula
* De-ionized water (DI H2O)
* Distillation apparatus
  + 2 ring stands with clamps
  + Heat source (i.e. hot plate, bunsen burner)
  + Distilling flask (can be an Erlenmeyer flask)
  + Condenser
  + Joint adapters
  + Thermometer
  + Collection flask, pre-marked by a permanent marker at 20 mL (will need cork ring for support if using a round bottom collection flask)
  + Cold water source
  + Tubing

**CO2 Extraction**

* 3 x pairs of gloves
* 2 x lemons
* 1 x lemon peel grater –medium grade. Zesters do not work well.
* 2 x weigh boats
* 1 x spatula
* 5 x 15 mL polypropylene centrifuge tubes with caps (Corning Catalog #430052)
* 1 x 100 mL or larger plastic graduated cylinder
* 1 lb dry ice
  + *Dry ice should be crushed as fine as possible. Chunkier ice takes a longer time to react and with limited success.*
* 1 x balance
* 10 x parafilm pieces cut into 1” x 2” squares
* Kimwipes or non-absorbent toilet paper
* 1 x pair of tweezers

**Time Required:** 2 x 60 minute class periods

**NGSS Standards:**

* **HS-ETS1-2.** Design a solution to a complex real-world problem by breaking it down into smaller, more manageable problems that can be solved through engineering
* **HS-ETS1-3.** Evaluate a solution to a complex real-world problem based on prioritized criteria and tradeoffs that account for a range of constraints including cost, safety, reliability, and aesthetics as well as potential social, cultural and environmental impacts.
* **HS-PS1-10.** Use evidence to support claims regarding the formation, properties and behaviors of solutions at bulk scales.

**Green Chemistry Principles Addressed:** 5, 6, 11, 12

**Teacher Prep:**

* Collect materials and lay out for student access
* Crush the dry ice into a fine powder

**Procedure:**

Day 1

* Show the PowerPoint Slides 1-5 for this lesson to give the background information to the students.
* Explain to the students that they will now extract lemon oil, d-limonene from a lemon using steam distillation.
* Hand out the student lab sheet and have the students review the information.
* Check for understanding and answer any questions.
* Instruct the students to begin the lab activity for steam distillation.
* When all students have finished, debrief the students data sheet questions, paying special attention to the analysis of the process against the 12 principles of green chemistry.

Day 2

* Review essential oil extraction from the last class period.
* Show PowerPoint slides 5 - 9
* Instruct the students to begin the second part of the lab.
* During the lab, ensure that finely crushed dry ice is placed into the tube.
  + Quickly transfer freshly, finely crushed dry ice into the tube, and cap the tube tightly making sure not to over-strip the cap
  + Quickly seal the cap with 2 parafilm squares. The longer the parafilm is exposed to the cold tube without being tightly wound around it, the more likely it will become brittle and break thus unable to provide a tight seal.
  + Have students stand at least a foot away from the experiment after they have dropped their test tubes into the water and have them watch from the side as tops can sometimes pop off due to pressure.

\*Make sure that the students are firmly sealing the test tubes. This seal is critical to the process and production of the extraction, as it ensures sufficient pressure inside the tube

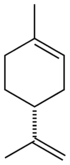
* After the lab is complete have the students fill out the student lab sheet.
* Show Powerpoint slide #10
* Hand out the 12 principles comparison student table. Have students work in groups to complete the table and then discuss their answers as a class.

**Steam Distillation Lemon Oil Extraction**

**Student Lab Procedure**

Many fruits and vegetables contain essential oils, which are water repellent or **hydrophobic liquids** that give the fruit or vegetable its distinctive fragrance. These essential oils are often extracted for use in perfume, cosmetics, food, medicine and house cleaning products.

You will use the lab directions below to extract essential oils from a lemon using steam distillation. This essential oil is called d-limonene and is represented by the chemical structure shown below:

[](http://en.wikipedia.org/wiki/File:Limonene-2D-skeletal.png)

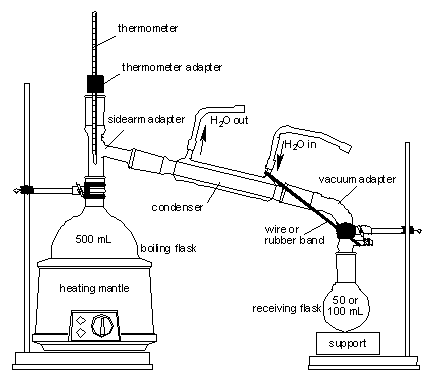
Chemical structure of d-limonene

Boiling point = 176˚C

**Steam Distillation:**

Steam distillation is a technique used to isolate or extract compounds at temperatures below their boiling points. Some compounds, such as essential oils, tend to decompose at their boiling temperature. By adding water or steam to the compound, the boiling point of the compound is reduced, allowing it to evaporate at a lower temperature so as to avoid decomposition during extraction.

The evaporated compounds are in their gaseous phase once heated, but condense back into their liquid phase upon contact with a cold surface (such as a condenser with cold water surrounding it). The compound in liquid form is then collected into a receiving flask. The diagram below shows the distillation set-up.



**Procedure:**

1. Collect the following apparatus from the supply area:

* + Lemon
  + Scissors (grating 25 grams worth of lemon skin will make for a much longer class period than 60 minutes)
  + Watt’s Up Meter (optional)
  + Scale
  + Weigh boat or weigh paper
  + Spatula
  + Deionized water (DI H2O)
  + Distillation apparatus
    - 2 ring stands with clamps
    - 1 hot plate
    - Distilling flask (can use an Erlenmeyer flask)
    - Condenser
    - Joint adapters
    - Thermometer
    - Collection flask, pre-marked with permanent marker at 20 mL (will need cork ring for support if using a round bottom collection flask)
    - Cold water source
    - Tubing

1. Review the data sheet below to ensure that you are recording data at certain phases of the experiment.
2. Set up the distillation equipment as shown in the diagram attaching the Watt’s Up Meter to the hot plate.
3. Determine the mass of the collection flask and record it on the student data sheet.
4. Peel the lemon and cut the lemon rind into small pieces (1-2 cm big)Place 25 g of the lemon rind in the distilling flask.
5. Add 25 mL DI H2O into the distilling flask.
6. Place the distilling flask on a hot plate.
7. Turn on the hot plate and allow the flask to heat up. Record the time that you turned on the hot plate. Monitor the temperature at which the water in the distilling flask begins to boil.
8. Collect the essential oil distillate in the collection flask. Record the time at which you are able to see the essential oil appear in the collection flask.
9. Allow the contents in the distilling flask to boil until approximately 20 mL of the distillate is collected. Record the time at which 20 mL of the essential oil distillate is collected.
10. Determine the mass of the essential oil collected.
11. Calculate the percent recovery of the essential oil compared to the mass of the rind.
12. Complete the student data sheet for steam distillation and answer all questions in full.

**Liquid CO2 and the Comparison of Essential Oil Extraction Methods**

**Student Data Sheet – Steam Distillation**

1. Mass of the dry, empty collection flask \_\_\_\_\_\_\_ grams
2. Mass of the collection flask with 20 mLs essential oil distillate \_\_\_\_\_\_\_ grams
3. Mass of the essential oil collected \_\_\_\_\_\_\_ grams
4. Volume of the essential oil collected \_\_\_\_\_\_\_\_ mL
5. Mass of the lemon rind placed into the distilling flask \_\_\_\_\_\_\_ grams
6. Temperature at which the water in the distilling flask boils? \_\_\_\_\_\_\_ ˚C
7. Color of the essential oil you have created \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
8. What was the percent recovery of the essential oil compared to the mass of the rind used with the steam distillation extraction?

*To calculate the percent recovery:*

*(mass of essential oil/mass of rind) x 100*

1. Time at which hot plate is turned on: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. Time that essential oil begins to appear in collection flask: \_\_\_\_\_\_\_\_\_\_\_\_\_
3. Time that 20 mL of essential oil distillate is collected: \_\_\_\_\_\_\_\_\_\_\_\_\_\_
4. How many minutes does it take to collect approximately 20 mL of the distillate?

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

1. How many minutes did the entire process take, from turning the hot plate on to collecting 20 mL of the essential oil distillate? \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. In a brief paragraph, consider the properties of the essential oil you have created – paying special attention to the toxicity and impact on the environment of the d-limonene.
3. In a brief paragraph, analyze this process against the 12 principles of green chemistry. Which principles does this process not adhere to and why?

1. Draw the chemical structure of the D-limonene you have extracted below:

**Liquid CO2 Lemon Oil Extraction**

**Student Lab Procedure**

Scientists have discovered the use of carbon dioxide (CO2) at high pressure as an alternative method of extracting essential oils. CO2 is the gas exhaled by humans during respiration, is consumed by plants during photosynthesis and exists in the environment in abundance from human activity such as fossil fuel combustion.

CO2 can be used as an alternative solvent for the extraction of essential oils. Because CO2 does not have high reactivity with essential oils, which can lead to the breakdown of the essential oil, its use in essential oil extraction is gaining popularity. Currently, CO2 is used to remove caffeine from coffee beans to produce decaffeinated coffee and as replacement for perchloroethlyene in dry cleaning applications. In this experiment you will be generating liquid CO2 to act as a solvent for extraction of essential oil.

**NOTE: For this experiment to work well, grate only the COLORED portion of the lemon peel.**

**Procedure:**

1. Collect the following apparatus from the supply area:
   * 3 x pairs of gloves
   * 2 x lemons
   * 1 x lemon peel grater –medium grade. Zesters do not work well.
   * 2 x weigh boats
   * 1 x spatula
   * 5 x 15 mL polypropylene centrifuge tubes with caps (Corning Catalog #430052)
   * 1 x 100 mL or larger plastic graduated cylinder
   * 1 lb dry ice crushed dry ice
     + *Dry ice should be crushed as fine as possible. Chunkier ice takes a longer time to react and with limited success.*
   * 1 x balance
   * 10 x parafilm pieces cut into 1” x 2” squares
   * Kimwipes or non-absorbent toilet paper
   * 1 x pair of tweezers
2. Put on safety gloves and glasses.
3. Fill plastic graduated cylinder with room temperature water (about ¾ full).
4. Find the mass of the empty centrifuge tube (without the cap) and record it on the student data sheet.
5. Using the medium grate opening on a food grater, **grate only the colored part** of the lemon peel. Collect 2 grams of the grated material in a weigh boat to minimize waste and mess.
6. Loosely place the 2 grams colored lemon peel into a kimwipe.
7. Wrap the kimwipe into a small bundle so that no lemon peel can escape.
8. Place the kimwipe bundle into the tube, and using the spatula, push it down towards the bottom of the tube. It should not enter the conical end of the tube.
9. Wear the oven mitts (these gloves should be worn every time dry ice is handled).
10. Using a mortar and pestle or a hammer, crush the dry ice into very fine pieces. The finer the pieces, the better. Use right away or the dry ice will sublime or reform into one big solid piece .
11. Fill the centrifuge tube with freshly, finely crushed dry ice all the way to the top. Tap the bottom of the tube against the lab bench to pack as much dry ice in as possible.
12. Place the cap on the centrifuge tube. Tighten the cap carefully. It is crucial to the process that you get a tight seal, but do not over tighten so that you over screw the cap and go from tight to loose.
13. Wrap two pieces of parafilm around the cap to ensure a good seal. Peel the white paper backing off of the parafilm. Hold one end of the parafilm to the tube using your thumb. Stretch and wrap the remaining end tightly around the tube 3-4 times. Repeat with 2nd piece of parafilm.
14. Transfer the tube to water in the plastic graduated cylinder. Record the start time of the reaction at this point.

**STAND BACK FROM THE EXPERIMENT AS TOPS CAN POP OFF.**

You may hear the hissing sound of CO2 gas escaping, which is expected. You will be able to see bubbles escaping from beneath the parafilm as the CO2 gas escapes. Soon after, you should be able to see liquid CO2 (this is the liquid CO2) forming and bubbling inside your tube.

1. Allow the reaction to continue until all of the liquid CO2 stops bubbling and disappears. Record the end time of the experiment.

NOTE: if you see the solid dry ice shrinking in diameter (it gets narrow, but has the same height), you need to re-do the set-up and try again. You should see the dry ice level get lower, but have the same thickness. Right after the dry ice lowers, you will see liquid CO2 liquid bubbling inside the tube.

1. Remove the tube from the water and slowly uncap it. Always point the tube away from your face and body.
2. Add more crushed dry ice and repeat the process a few times until you can see a liquid at the bottom tip of the centrifuge tube. This pale yellow liquid is the essential oil d-limonene.
3. Carefully remove the kimwipe bundle trap using tweezers.

NOTE: Keep the tube upright to avoid any loss of the oil. There should be nothing in the tube at this point except for the essential oil collected in the tip of the tube.

1. Dry the outside of the tube with a paper towel, weigh the tube with the essential oil in it. Record this mass.
2. Determine the mass of the essential oil.
3. Calculate percent recovery based upon the yield of the product compared to the mass of rind used.

**Liquid CO2 and the Comparison of Essential Oil Extraction Methods**

**Student Data Sheet – Liquid CO2**

1. Mass of the empty tube (without cap) \_\_\_\_\_\_\_\_\_\_\_\_\_ grams
2. Mass of lemon peel placed in the tube \_\_\_\_\_\_\_\_\_\_\_\_\_ grams
3. Mass of the tube and essential oil \_\_\_\_\_\_\_\_\_\_\_\_\_ grams
4. Mass of the essential oil collected \_\_\_\_\_\_\_\_\_\_\_\_\_ grams
5. What was the percent recovery of the essential oil compared to the mass of the rind used with the steam distillation extraction?

*To calculate the percent recovery:*

*(mass of essential oil/mass of rind) x 100*

1. Start time of experiment: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. End time of experiment: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
3. How many minutes did it take to collect the essential oil? \_\_\_\_\_\_\_\_\_\_ minutes
4. Estimate the volume of the essential oil collected \_\_\_\_\_\_\_\_\_\_\_\_ mL
5. Using your answers from questions 8 and 9, estimate how long would it take to collect 20 mL of the essential oil \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ minutes
6. What is the temperature of the water used in the process? \_\_\_\_\_\_\_\_\_\_\_˚C
7. What is the color of the essential oil you have created?\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
8. In a brief paragraph, consider the properties of the essential oil you have created – paying special attention to the waste, energy usage, procedure efficiency and hazards.

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

**Comparison of Essential Oil Extraction Methods – Student Sheet**

Not all principles will apply to this process – put N/A (not applicable) in any cells that do not apply to this process.

* REMEMBER: think about all of these processes both on a laboratory level and as an industrial process where large quantities of product need to be produced.
* USE: the information that you gathered using the laboratory data sheets to inform your decision on this grid.

|  |  |  |  |
| --- | --- | --- | --- |
| Principle | Traditional Solvent Extraction | Steam Distillation Extraction | Liquid CO2 extraction |
| #1 Pollution prevention |  |  |  |
| #2 Atom Economy |  |  |  |
| #3 Less hazardous synthesis |  |  |  |
| #4 Design safer chemicals |  |  |  |
| #5 Safer solvents and auxilaries |  |  |  |
| #6 Energy efficiency |  |  |  |
| #7 Renewable feedstocks |  |  |  |
| #8 Reduce Derivatives |  |  |  |
| #9 Cataylsis |  |  |  |
| #10 Design for degradation |  |  |  |
| #11 Real-time analysis |  |  |  |
| #12 Accident prevention |  |  |  |

**Comparison of Essential Oil Extraction Methods – Answer Key**

Not all principles will apply to this process – put N/A (not applicable) in any cells that do not apply to this process.

* REMEMBER: think about all of these processes both on a laboratory level and as an industrial process where large quantities of product need to be produced.
* USE: the information that you gathered using the laboratory data sheets to inform your decision on this grid.

|  |  |  |  |
| --- | --- | --- | --- |
| Principle | Traditional Solvent Extraction | Steam Distillation Extraction | Liquid CO2 extraction |
| #1 Pollution prevention | Pollution in the final product could be hazardous to human health as well as solvents in the waste stream | N/A | N/A |
| #2 Atom Economy | Lemon rind without oil is left over – but this is benign waste which can be composted | Lemon rind without oil is left over – but this is benign waste which can be composted | Lemon rind without oil is left over – but this is benign waste which can be composted |
| #3 Less hazardous synthesis | Hazardous solvents are used – methylene chloride is a carcinogen. The solvents used in this process are not considered safe | N/A | N/A |
| #4 Design safer chemicals | Final product can be hazardous to human health if there are trace solvents present. | The trace solvent left over will be water therefore it is benign. | N/A |
| #5 Safer solvents and auxilaries | The solvents used in this process are not considered safe | Water is a safer solvent however the steam might be hazardous to human health especially on an industrial scale | CO2 is non toxic to humans. On an industrial scale pressurized gas is used and it is constantly reused. |
| #6 Energy efficiency | Energy efficient. No use of intense heat. | This process uses a lot of energy in the heating of the water and subsequent cooling of the water when used on an industrial scale. | There is energy used in this process to heat the water but the water does not have to be as hot as in the case of steam distillation. Also on an industrial scale there is no water used but the CO2 is pressurized and heated although not to extreme temperatures. |
| #7 Renewable feedstocks | The solvents used are petroleum derivatives. Petroleum is a non-renewable resource. | This process utilizes large amounts of water. Although technically water is a renewable resource, on an industrial scale water is taken from rivers and put back into the watershed at temperatures that are not consist with the health of the rivers. Although often this water was recycled. | CO2 is a renewable resource |
| #8 Reduce Derivatives | N/A | N/A | N/A |
| #9 Catalysis | N/A | N/A | N/A |
| #10 Design for degradation | Methylene chloride and other organic solvents are not readily biodegradable – many will be persistent in the environment | Water naturally degrades and recycles in the environment, so this is a better solvent. | Carbon Dioxide is not persistent in the environment. On an industrial scale, the same CO2 is used over and over again. |
| #11 Real-time analysis | N/A  This is not applicable because this process is an extraction not a reaction | N/A  This is not applicable because this process is an extraction not a reaction | N/A  This is not applicable because this process is an extraction not a reaction |
| #12 Accident prevention | Workers are exposed to carcinogens and they can get severe burns. Some solvents can also be flammable. | Steam and water heated to a high temperature can be very hazardous both in the classroom and in an industrial setting | Liquid CO2/dry ice can be hazardous in the form you see it in the classroom. It can burn when handled. In an industrial setting there is increased pressure which will always hold a risk of explosion but in an industrial setting this would always be enclosed due to the way the equipment is designed. |

**Additional Teacher Background Information and Safety Considerations**

**Safety concerns**

The most serious safety concerns in this experiment involve the possibilities of cap discharge (most common occurrence) or vessel rupture (rarely observed). During the testing phase of this experiment, caps blew off during approximately 4% of the extractions. All caps were directed upward by the containment cylinders. Caps remained on during all extractions when students 1) tightened the cap as tightly as possible and 2) did not use caps that were stripped. **If the cap cannot be completely tightened and continues to turn, the stripped cap should be discarded and replaced.** Due to variations in the centrifuge tubes and caps, a tight seal is not always formed at their junction. In this case, the CO2 does not liquefy, and retightening of the cap or replacement of the cap or tube may be required. Although many modifications of the sealing process have been proposed, such as the use of Teflon tape or parafilm on the threads, it is important that the cap seal well enough to induce liquefaction but not so tightly that the gas cannot escape. The cap must allow the gaseous CO2 to escape slowly during the extraction and also must function as a safety valve. During experiment development, attempts were made to observe the transition from the liquid to the solid phase by opening the cap while the CO2 was liquid. In two of these cases, the centrifuge tube ruptured, and plastic shards were propelled several feet from the demonstrator. Although no injuries occurred, it is recommended that the tubes always remain in containment cylinders during liquefaction. It is important to note that in our experience vessel rupture *only* occurred while attempting to release the pressure from the vessel when liquid CO2 was present.

**Industrial methods for obtaining D-limonene.**

Traditionally essential oils have been extracted through the use of steam distillation or organic solvent extraction. During the past two decades, great strides have been made in technology that uses supercritical or liquid carbon dioxide in place of organic solvents. Carbon dioxide (CO2) is useful as a green alternative solvent because it provides environmental and safety advantages; it is nonflammable, relatively nontoxic, readily available, and environmentally benign. Processing with CO2 also results in minimal liability in the event of unintentional release or residual solvent in the product. Although CO2 is a greenhouse gas, when used as a solvent it is captured from the atmosphere, not generated, resulting in no net environmental harm. Large-scale CO2 processing has had commercial success in many separation and extraction processes. The tunable solubility properties, low toxicity, and ease of removal of CO2 have led to well established CO2 technology for the extraction of various food products, including essential oils and hops, and for the decaffeination of coffee and tea.

Another major benefit of using carbon dioxide as a solvent is its accessible phase changes. Unlike other gases, relatively low temperatures and pressures can be used to form supercritical and liquid CO2. CO2 sublimes (goes directly from a solid to a gas) at normal atmospheric pressure of 1.01 bar. Looking at a phase diagram of CO2, you can see the temperature and pressure at which the CO2 must be reached in order to see a transition from solid (dry ice) to liquid. In this experiment the supercritical point of CO2 will not be reached, this is not feasible with the equipment used in this simple experiment. In industry, pressure vessels are used to achieve the triple point, above which the supercritical point is reached.