Caffeine Extraction
STUDENT HANDOUT

EQUIPMENT
Hot plate, milligram balance, centrifuge, sand bath.

SUPPLIES
Safety goggles, tea bag, 50 mL glass beaker, 100 mL beaker, glass stirring rod, 1M Na$_2$CO$_3$, distilled water, aluminum foil, beaker tongs, ice bath, hexane/isopropanol solution with dispensette, 15 mL screw cap centrifuge tubes, Pasteur pipets with bulb, numbered glass screw cap vial, Na$_2$SO$_4$, 10-mL graduated pipette with green pump.

CAUTION
Hexane and isopropanol are EXTREMELY FLAMMABLE. Do not use near open flame! Hot plates can cause burns, be careful. Caffeine is poisonous in large quantities, exercise caution when handling it and wash your hands when you leave the laboratory area. Goggles are mandatory whenever you are in the lab area and laboratory work is being done. See the MSDS sheets for additional health and safety concerns.

PURPOSE
To extract and measure the caffeine in a tea bag.

PROCEDURE
1. Put on your goggles. Check that your lab station has all supplies as listed above.

2. Find the mass of a tea bag with tea. Record the mass. Copy the mass of the empty tea bag from the chalkboard.

3. Place 10 mL of a 1 M Na$_2$CO$_3$ solution in a 50 mL beaker. Add tea bag, cover with foil until solution boils, then remove foil. Boil for 5 minutes. Note: Add distilled water from wash bottle as needed to maintain 10 mL level. Keep the bag off the bottom of the beaker to avoid burning a hole in the bag.

4. Remove beaker from hot plate carefully, using tongs; let air cool for a few minutes; then cool beaker in ice bath; squeeze out excess fluid from tea bag being careful not to break bag. Discard the tea bag. At this point you should have no more than 10 mL of solution. If you have more, boil off some of the water to concentrate the solution. (Make sure 50 mL beaker is dry on the outside before putting it back on the hotplate). Cool the extract (liquid) for one minute in ice bath.

5. Pour the cooled extract into a 15 mL centrifuge tube.

6. Now, using the dispensette, pump 3 mL of hexane/isopropanol into your centrifuge tube and screw on the cap. Gently shake the tube for 10 seconds then relieve pressure by partially unscrewing the cap. If tube is shaken vigorously and opened quickly, contents may spew out of vial. Retighten the cap and shake gently for at least 30 seconds. Relieve pressure in tube every ten seconds by partially unscrewing the cap.
7. Dry the outside of the tube and tighten the cap. Place tube in centrifuge. **Note the centrifuge number for your tube.** Centrifuge the mixture for three to four minutes, being sure to balance the centrifuge with another tube of approximately equal volume.

8. Carefully remove the clear top layer of liquid with a glass Pasteur pipet and place it in your numbered glass vial. Avoid putting the pipet tip into the lower aqueous phase – you want the top layer (or organic phase). **Squeeze the bulb of the pipet before putting the tip of the pipet into the liquid.** Put the pipet tip just below the surface of the top layer in the centrifuge tube and release the bulb slowly. This will be a mixture of your product and hexane/isopropanol. Repeat this process until most of the top layer has been transferred into your group’s numbered vial.

9. Repeat the extraction process (See steps 5-8) **two** more times using 3 mL of fresh hexane/isopropanol each time. After each subsequent extraction be sure to pipet the clear top layer into your vial. Avoid taking any of lower aqueous layer. Screw the cap on your vial and note and record the number of the vial.

10. After the third extraction is completed, discard remaining lower dark layer into a waste container provided by your teacher. Save your vial with its contents for the next part of the lab.

---STOP-HERE-IF-YOU-DO-NOT-HAVE-TIME-TO-COMplete-THE-LAB-TODAY---

11. Add a small amount (pea-sized) of Na₂SO₄ crystals to your vial of solvent and caffeine. Screw cap on vial and swirl gently. The crystals will clump. Continue to add small amounts of crystals to the vial and swirl it until the addition of new crystals does not produce more clumping. You should observe individual crystals of Na₂SO₄ in your vial as you swirl it.

12. Using the stirring rod, decant the fluid portion of your vial into the 100 mL beaker. Avoid getting any crystals of Na₂SO₄ into the beaker.

13. After the decanting process is completed the hexane/isopropanol mixture must be carefully and slowly evaporated leaving only the crude caffeine behind. To evaporate the hexane/isopropanol mixture, place the 100 mL beaker into a sand bath heated to 90°C in the fume hood. Gently swirl the beaker in the sand bath. Just as the solvent has evaporated, remove the beaker. The residue can be slightly moist. The crude caffeine will coat the bottom of the beaker.

**Caution: DO NOT BOIL THE BEAKER DRY!**

14. Assemble the sublimation apparatus as shown in Figure 1. The outside of the inner 50 mL beaker must be very clean and dry. Do not add ice until after the beaker is placed on the hotplate.

15. Place the sublimation apparatus on a hot plate and begin heating. Fill the small beaker with chips of ice. Be careful not to let ice or water fall into the larger beaker.
16. Continue heating until the sublimation process is complete. When heated, the pure caffeine will sublime and then be deposited on the outside of the 50 mL inner beaker.

17. Once sublimation is complete, remove the apparatus from the hot plate with tongs and allow to cool. Remove the inner beaker very cautiously and carefully pour off the ice water making certain no water comes into contact with the sublimed caffeine crystals.

18. Dry the inside of the 50 mL beaker with Kimwipes and find the mass of this beaker with the caffeine on its bottom. Record this mass. Make careful observation about the physical properties of caffeine. Scrape the caffeine from the beaker and turn the product in to the instructor.

19. Carefully clean and dry the beaker you used. Find the mass of your clean, dry 50 mL beaker and record.

20. Find the difference in masses and record as the mass of caffeine obtained.
Caffeine Extraction

DATA COLLECTION

Mass of the tea bag with tea

Mass of the empty tea bag from board

Mass of tea

Type of tea used

Vial #

Mass of dry beaker with caffeine

Mass of clean, dry 50 mL beaker

Mass of caffeine

Color and appearance of the caffeine

Use this space for sample calculations.
QUESTIONS

1. What is the solubility of caffeine in water? In the hexane/isopropanol mixture?

2. Why is the hexane/isopropanol mixture a better solvent for this extraction than water?

3. How does sublimation differ from vaporization?

4. What was the purpose of the sublimation step of this lab?

5. It has been suggested by medical experts that caffeine, when consumed in excess, is detrimental to human health. Based on your knowledge of caffeine explain the reasons for this concern and possible effects on the body.

6. In your local grocery store you will find decaffeinated coffee and tea. Based on what you learned in this experiment propose a method by which the decaffeination process occurs.