Discussion

The operating guide for Liesegang Rings is organized slightly differently than other operating guides. This is because there are recipes here for 5 different reactions. It is recommended that one reaction of each recipe is set up, then all 5 reactions may be displayed. The general directions for all the recipes are given, then the specific recipes follow.

In general, a Liesegang reaction follows the following format. First, a chemical is dissolved in a gel solution. This gel solution is either made: 1) of the chemical dissolved with gelatin in water, or 2) of equal parts of 0.5 M acetic acid (with chemical dissolved) and 1.06 specific gravity silica gel. By either method, the gel is allowed to set overnight or longer. Then, a second chemical is layered on top of the gel. Over a period of weeks, reaction rings or crystals develop, with new reaction rings appearing progressively lower down the reaction tube.

Liesegang reactions are optimum under a few conditions. 1) The density of the gel material is very important. If the gel is too dense, the layered chemical cannot diffuse through, and a reaction occurs only at the interface between the two chemicals. If the gel is not dense enough, diffusion can happen two quickly and distinct rings are not seen. 2) When pouring the gel, be careful not to trap air bubbles, and do not disturb the container once the gel has formed. If the gel surface is wavy, or has been interrupted with a bubble or fracture, the rings will reflect this. 3) The concentration of the chemical in the gel must make it the limiting reagent in the reaction. The formation of rings depends upon the chemical in a local area being used.

The recipes listed here come from various sources:

Recipe 1 – OMSI original operating guide, Liesegang Rings Type 1
Recipe 2 – Sharbaugh, A. III and Sharbaugh, A. H. Jr. Journal of Chemical Education, 1989, v66, p589. This replaces the incorrect recipe from OMSI original operating guide, Liesegang Rings Type 2.
Recipe 3 – OMSI original operating guide, Liesegang Rings Type 3
Recipe 4 – Schibeci, R. A. and Carlsen, C. Journal of Chemical Education, 1988, v65, p365.
Recipe 5 – Stanley, Norm www.sas.org/tcs/weeklyIssues/2004-04-30/chem, written 4/30/2004, accessed 12/13/2005.
Recipe for sodium silicate solution, density 1.06 g/mL – Forman, J. E. Journal of Chemical Education, 1990, v67, p720.

For information on how Liesegang Rings are formed, see the following:

Hughes, E.B. Biochemistry Journal, 1934, 28, 1086-1196
Kanniah, N; Gnanam, F.D.; Ramasamy, P. Indian Acadamy of Science, Chemical Science, 1984, 93, 801-811.
Ostwald, Wo. Kolloid Zeit, 1926, 36, 380-390.
Sharbaugh, A. III and Sharbaugh, A. H. Jr. Journal of Chemical Education, 1989, v66, p589.

MATERIALS

- 250mL sodium silicate solution 40% w/v (water glass)
- 600mL vinegar (household, 5% acetic acid)
- stirring plate
- magnetic stir bar
- 2L beaker
- 1L beaker
- 250mL graduated cylinder
- 1L graduated cylinder
- 1.5L storage bottle
- 1L storage bottle
- plexiglas barrier
- cross-section samples of agate with rings
- 5 250mL graduated cylinders or other suitable containers
- other chemicals and equipment are listed in Materials Prep in the specific recipe

Setup/Takedown Procedures

ORIGINAL SETUP

- Prepare stock solutions (see Materials Prep).
- Prepare one Liesegang reaction of each recipe (see Materials Prep)
- Label agate samples.
- Label Liesegang reactions.

WEEKLY SETUP

- Set out public copy in Plexiglas holder.
- Set out cylinders of Liesegang reactions
- Set out agate samples.
- Place Plexiglas barrier in front of display

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WEEKLY TAKEDOWN

- Return Liesegang reactions and rock samples to glass cabinet storage.
- Return public copy to file drawer.

RUNNING SUGGESTIONS

- Make certain cylinders are tightly sealed. With tight sealing, Liesegang reactions have been known to last for as long as 30 years (Sharbaugh and Sharbaugh). If gels begin to look dehydrated, or otherwise less visually stimulating, replace the cylinder with a new reaction.
- Any container that is tall and narrow will work. A 250mL graduated cylinder is the best shape, but the graduation lines may interfere with the observation of rings. Test tubes work, but should be at least 10 inches in length. For any container, make certain you have a sealing lid or stopper.

EXTENSIONS

- **How Liesegang Rings are formed.** There are two reacting chemicals, one suspended in the gel, and one layered on top. As the solution on top diffuses through the gel, it reacts with the surrounding chemical. For a precipitate to form, there must be a certain concentration of chemical in the gel. As the reaction occurs, the chemical in the gel is used up, and there is no longer a high enough concentration. Thus, the diffusing chemical must continue to migrate to an area of greater concentration of chemical in the gel. Then the reaction occurs again. (Ostwald, Wo Kolloid Zeit 1926, 36, 38-390, cited in Sharbaugh and Sharbaugh).
- **How does this relate to Environmental Chemistry?** The Liesegang reactions take place in a silica gel. This silica gel has the same composition as sand. Agates form when water rich in silicate and other minerals fill a space in rock. Eventually a soft gel forms, and hardens into stone. Chemicals diffusing in the silicate gel likely form the rings formed in agates.

Safety & Disposal

Health: 1 **Flammability: 0** **Reactivity: 0** **Contact: 2**	sodium silicate 40% (water glass) is not considered hazardous. Mildly irritating to skin. Practice standard lab procedures. See MSDS for further information.
Health: **Flammability:** **Reactivity:** **Contact:**	5% acetic acid (household vinegar) is not considered hazardous. Practice standard lab procedures. See MSDS for further information.
Health: 3 **Flammability: 0** **Reactivity: 0** **Contact: 1**	**Caution: $CoCl_2$*$6H_2O$ (cobalt chloride hexahydrate) is poisonous and may be fatal if swallowed.** Wear gloves and goggles, do not breathe dust. Measure in hood. Wash thoroughly after handling. See MSDS for further information.
Health: 1 **Flammability: 0** **Reactivity: 1** **Contact: 1**	$MnSO_4$*H_20 (manganese sulfate monohydrate) in not considered hazardous. Practice standard lab procedure. See MSDS for further information.
Health: **Flammability:** **Reactivity:** **Contact:**	$(NH_4)_2HPO_4$ (ammonium phosphate, dibasic) is not considered hazardous. Practice standard lab procedures. See MSDS for further information.
Health: 3 **Flammability: 0** **Reactivity: 2** **Contact: 3**	**Caution: K_2CrO_4 (potassium chromate) is highly toxic by ingestion and inhalation.** Irritating to body tissues. Known carcinogen. Wear gloves and goggles. Measure in hood. Wash thoroughly after use. See MSDS for further information.
Health: 2 **Flammability: 0** **Reactivity: 0** **Contact: 2**	**Caution: $CuSO_4$*$5H_2O$ (cupric sulfate pentahydrate) causes skin irritation and is harmful if swallowed.** Wear gloves and goggles. See MSDS for further information.
Health: 1 **Flammability: 1** **Reactivity:1** **Contact: 1**	$(CH_2COOH)_2$ (succinic acid) is not considered hazardous. Practice standard lab procedures. See MSDS for further information.

Health: 3 **Flammability: 0** **Reactivity: 1** **Contact: 3**

Caution: NH_4OH (concentrated ammonia, ammonium hydroxide) is extremely damaging to eyes. Harmful if swallowed or inhaled. Corrosive and causes burns on skin contact. Fumes are harmful to mucous membranes. Wear gloves and goggles. Measure in hood. See MSDS for further information.

Health: 1 **Flammability: 0** **Reactivity: 1** **Contact:**

Caution: $K_2Cr_2O_7$ (potassium dichromate) is toxic by ingestion and inhalation. May be toxic by skin absorption. Contains chromium and chromium compounds are classed as known carcinogens by IARC and NTP. Wear gloves and goggles. See MSDS for further information.

Health: 3 **Flammability: 0** **Reactivity:3** **Contact: 3**

Caution: $AgNO_3$ (silver nitrate) solid is highly toxic by ingestion and inhalation. Corrosive to body tissues. Ingestion may be fatal; as little as 2 grams can be fatal to humans. Wear gloves and goggles. Measure in hood. Wash thoroughly after handling. See MSDS for further information.

Unused solutions of vinegar and sodium silicate may be poured down the drain. Gelled sodium silicate and gelatin may be thrown in the trash.

All other chemicals used in these experiments are hazardous and must be disposed of professionally. Store waste chemicals in a tightly sealed, labeled container.

MATERIALS PREP

To prepare **sodium silicate 1.06 g/ml** stock solution:

1. Measure 1360mL of water. Add this to a 2L beaker on a stir plate. Add a magnetic stir bar, and set it stirring at medium speed.
2. Before measuring 40% sodium silicate, stir it well. It will be a thick, syrupy liquid, and tends to settle out.
3. Measure 250mL 40% sodium silicate solution.
4. Dropwise, add the sodium silicate to the stirring water. This will take a long time. Adding large amounts of sodium silicate at once risks the water glass sinking to the bottom, and not being uniformly mixed.
5. After all sodium silicate is added, stir well.
6. Pour this solution into a 1.5L bottle. Label this bottle “sodium silicate stock solution 1.06 g/mL”

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To prepare **0.5M acetic acid** stock solution:

1. Measure 240 mL 5% acetic acid (household vinegar). Pour into a 600mL beaker.
2. Add water to a total volume of 400mL.
3. Pour this solution in to a 500mL bottle. Label this bottle “0.5 M acetic acid stock solution”

RECIPE 1

Adapted from OMSI Operating Guide, Liesegang Rings, Type 1
This recipe makes a pink or purple gel with bands of blue or purple. Over the course of a year, small amethyst-colored crystals form.

Materials:

Health: 3
Flammability: 0
Reactivity: 0
Contact: 1

Caution: $CoCl_2$*$6H_2O$ (cobalt chloride hexahydrate) is poisonous and may be fatal if swallowed. Wear gloves and goggles, do not breathe dust. Measure in hood. Wash thoroughly after handling. See MSDS for further information.

- 0.6g $CoCl_2$ * $6H_2O$ (cobalt chloride hexahydrate)
- 0.2g $MnSO_4$*H_2O (manganese sulfate monohydrate)
- 8g $(NH_4)_2HPO_4$ (ammonium phosphate, dibasic)
- 125mL sodium silicate stock solution 1.06 g/mL (recipe above)
- 125mL 0.5M acetic acid stock solution (recipe above)
- 400mL beaker
- 250mL graduated cylinder (for measuring)
- 250mL graduated cylinder (for display)
- small pipette

Procedure:

1. Fill the 400mL beaker with approximately 100mL 0.5M acetic acid stock solution.
2. Measure 0.6g $CoCl_2$ * $6H_2O$ (cobalt chloride hexahydrate) and add to the beaker.
3. Measure 0.2g $MnSo_4$*H_2O (manganese sulfate monohydrate) and add to the beaker.

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4. Stir until solids dissolve. Solution will range in color from clear to light bluish pink.
5. Add 0.5M acetic acid to a final volume of 125mL.
6. Measure 125mL sodium silicate stock solution 1.06g/mL. Add this to the acetic acid solution while stirring. Stir briefly to mix.
7. Quickly, but carefully, transfer the mixture into the 250mL graduated cylinder, avoiding excessive air-bubble formation.
8. Leave the cylinder undisturbed for at least 24 hours.
9. The next day, gently tilt the cylinder to see whether the gel has solidified. A slightly hazy, bluish-pink solid gel should have formed.
10. If the gel has not solidified, let it sit for one more day. If a solid gel has still not formed, use the pipette to add 4 to 6 drops of vinegar on the gel surface.
11. IT IS IMPERATIVE THAT THE NEXT STEP IS TAKEN ONLY AFTER A FIRM, SOLID GEL HAS FORMED IN THE GRADUATED CYLINDER.
12. Measure 8g $(NH_4)_2HPO_4$ (ammonium phosphate, dibasic) crystals. Make certain the crystals are free flowing and are not clumping. Gently pour these crystals uniformly over the top surface of the silica gel in the 250mL cylinder.
13. Using a pipette, add 8mL H_2O onto the surface. Stopper the cylinder, tape it shut, and set aside.
14. Within a few hours, a solid blue-purple band, about ½ inch thick, will form at the top of the silica gel. Then, for a number of days, alternate bands of Liesegang rings in varying shades of blue and purple will continue to appear through the main body of the silica gel mass. During a period of two to three weeks, a few tiny amethyst-colored crystals will begin to appear in different areas between the blue bands. During the course of a year, small amethyst-colored crystals will continue to grow into large crystal formations, eventually consuming most of the original band formations.

RECIPE 2

Adapted from Sharbaugh, A. III and Sharbaugh, A. H. Jr. Journal of Chemical Education, 1989, v66, p589.
This recipe makes a yellow to lime green gel with bands of brown or tan.

Health: 3
Flammability: 0
Reactivity: 2
Contact: 3

Materials:

Caution: K_2CrO_4 (potassium chromate) is highly toxic by ingestion and inhalation. Irritating to body tissues. Known carcinogen. Wear gloves and goggles. Measure in hood. Wash thoroughly after use. See MSDS for further information.

Health: 2
Flammability: 0
Reactivity: 0
Contact: 2

Caution: $CuSO_4$*$5H_2O$ (cupric sulfate pentahydrate) causes skin irritation and is harmful if swallowed. Wear gloves and goggles. See MSDS for further information.

- 4.5g K_2CrO_4 (potassium chromate)
- 1.6g $CuSO_4$*$5H_2O$ (cupric sulfate pentahydrate)
- 125mL sodium silicate stock solution 1.06 g/mL (recipe above)
- 125mL 0.5 M acetic acid stock solution (recipe above)
- 400mL beaker
- 250mL graduated cylinder (for measuring)
- 250mL graduated cylinder (for display)
- small pipette

Procedure:

1. Fill the 400mL beaker with approximately 100mL 0.5M acetic acid stock solution.
2. Measure 4.5g K_2CrO_4 (potassium chromate) and add to the beaker.
3. Stir until solids dissolve. Solution will be bright lemon yellow
4. Add 0.5M acetic acid to a final volume of 125mL.
5. Measure 125mL sodium silicate stock solution 1.06g/mL. Add this to the acetic acid solution while stirring. Stir briefly to mix.
6. Quickly, but carefully, transfer the mixture into the 250mL graduated cylinder, avoiding excessive air-bubble formation.
7. Leave the cylinder undisturbed for at least 24 hours.
8. The next day, gently tilt the cylinder to see whether the gel has solidified. A slightly hazy, yellow gel should have formed.
9. If the gel has not solidified, let it sit for one more day. If a solid gel has still not formed, use the pipette to add 4 to 6 drops of vinegar on the gel surface.
10. IT IS IMPERATIVE THAT THE NEXT STEP IS TAKEN ONLY AFTER A FIRM, SOLID GEL HAS FORMED IN THE GRADUATED CYLINDER.
11. Measure 1.6g $CuSO_4$*$5H_2O$ (cupric sulfate pentahydrate) crystals. Make certain the crystals are free flowing and are not

clumping. Gently pour these crystals uniformly over the top surface of the silica gel in the 250mL cylinder.

12. Add 8mL H_2O onto the surface. Stopper the cylinder, tape it shut, and set aside.
13. Within a few hours, a thin brown band will form at the top of the silica gel. In about six to eight hours, separate lines of Liesegang rings will start forming. Along with the disk formation, the color of the silica gel will change from lemon yellow to pale green. Alternate bands of black crystals of copper dichromate (CuCr2O7) may form over a period of weeks.

RECIPE 3

Adapted from OMSI Operating Guide, Liesegang Rings, Type 3
This recipe makes a clear to hazy gel with blue crystals.

Materials:

$(CH_2COOH)_2$ (succinic acid) is not considered hazardous. Practice standard lab procedures. See MSDS for further information.

Health: 2
Flammability: 0
Reactivity: 0
Contact: 2

Caution: $CuSO_4$*$5H_2O$ (cupric sulfate pentahydrate) causes skin irritation and is harmful if swallowed. Wear gloves and goggles. See MSDS for further information.

- 17.5g $(CH_2COOH)_2$ (succinic acid)
- 8g $CuSO_4$*$5H_2O$ (cupric sulfate pentahydrate)
- 125mL water
- 125mL sodium silicate stock solution 1.06 g/mL (recipe above)
- 400mL beaker
- 250mL graduated cylinder (for measuring)
- 250mL graduated cylinder (for display)
- small pipette

Procedure:

1. Fill the 400mL beaker with approximately 100mL water.
2. Measure 17.5g $(CH_2COOH)_2$ (succinic acid) and add to the beaker.
3. Stir until solids dissolve. If solids do not dissolbe, place it on a hot plate on low heat. Solution will be colorless.
4. Add water to a final volume of 125mL.

5. Measure 125mL sodium silicate stock solution 1.06g/mL. Add this to the acetic acid solution while stirring. Stir briefly to mix.
6. Quickly, but carefully, transfer the mixture into the 250mL graduated cylinder, avoiding excessive air-bubble formation.
7. Leave the cylinder undisturbed for at least 24 hours.
8. The next day, gently tilt the cylinder to see whether the gel has solidified. A slightly hazy gel should have formed.
9. If the gel has not solidified, let it sit for one more day. If a solid gel has still not formed, use the pipette to add 4 to 6 drops of vinegar on the gel surface.
10. IT IS IMPERATIVE THAT THE NEXT STEP IS TAKEN ONLY AFTER A FIRM, SOLID GEL HAS FORMED IN THE GRADUATED CYLINDER.
11. Measure 8g $CuSO_4*5H_2O$ (cupric sulfate pentahydrate) crystals. Make certain the crystals are free flowing and are not clumping. Gently pour these crystals uniformly over the top surface of the silica gel in the 250mL cylinder.
12. Add 8mL H_2O onto the surface. Stopper the cylinder, tape it shut, and set aside.
13. Within two to three days, distinct dark ultramarine blue crystals of copper succinate will start growing. They will continue to grow for several weeks.

RECIPE 4

Adapted from Schibeci, R. A. and Carlsen, C. Journal of Chemical Education, 1988, v65, p365.
This recipe makes a pink to red gel with bright blue bands.

Materials:

Health: 3
Flammability: 0
Reactivity: 0
Contact: 1

Caution: $CoCl_2*6H_2O$ (cobalt chloride hexahydrate) is poisonous and may be fatal if swallowed. Wear gloves and goggles, do not breathe dust. Measure in hood. Wash thoroughly after handling. See MSDS for further information.

Health: 3
Flammability: 0
Reactivity: 1
Contact: 3

Caution: NH_4OH (concentrated ammonia, ammonium hydroxide) is extremely damaging to eyes. Harmful if swallowed or inhaled. Corrosive and causes burns on skin contact. Fumes are harmful to mucous membranes. Wear gloves and goggles. Measure in hood. See MSDS for further information.

- 250mL water
- 400mL beaker

- 7.5g gelatin
- 18g $CoCl_2*6H_2O$ (cobalt chloride hexahydrate)
- 20mL concentrated ammonia
- 250mL graduated cylinder (for measuring)
- 250mL graduated cylinder (for display)
- small pipette

Procedure:

1. Fill the 400mL beaker with approximately 200mL water. Heat the water just to boiling.
2. Measure 7.5g gelatin and add it to the beaker. Continue to heat and stir until gelatin is completely dissolved.
3. Measure 18g $CoCl_2*6H_2O$ (cobalt chloride hexahydrate) and add to the beaker.
4. Stir until solids dissolve. Solution will be pink to light red.
5. Add water to a final volume of 250mL.
6. Quickly, but carefully, transfer the mixture into the 250mL graduated cylinder, avoiding excessive air-bubble formation.
7. Leave the cylinder undisturbed for at least 24 hours.
8. The next day, gently tilt the cylinder to see whether the gel has solidified. A pink to red gel should have formed.
9. IT IS IMPERATIVE THAT THE NEXT STEP IS TAKEN ONLY AFTER A FIRM, SOLID GEL HAS FORMED IN THE GRADUATED CYLINDER.
10. In the hood, measure 20mL concentrated ammonia. Gently pipette this liquid uniformly over the top surface of the silica gel in the 250mL cylinder.
11. Stopper the cylinder, tape it shut, and set aside.
12. Within an hour, a bright blue band about ½-inch thick should form. Over the next few days, tightly layered bright blue bands will continue to form down the length of the cylinder.

RECIPE 5

Stanley, Norm www.sas.org/tcs/weeklyIssues/2004-04-30/chem, written 4/30/2004, accessed 12/13/2005.
This recipe makes a yellow to gold gel with dark brown bands.

Materials:

Health: 3
Flammability: 0
Reactivity: 0
Contact: 1

Caution: $K_2Cr_2O_7$ (potassium dichromate) is toxic by ingestion and inhalation. May be toxic by skin absorption. Contains chromium and chromium compounds are classed as known carcinogens by IARC and NTP. Wear gloves and goggles. See MSDS for further information.

Health: 3
Flammability: 0
Reactivity:3
Contact: 3

Caution: $AgNO_3$ (silver nitrate) solid is highly toxic by ingestion and inhalation. Corrosive to body tissues. Ingestion may be fatal; as little as 2 grams can be fatal to humans. Wear gloves and goggles. Measure in hood. Wash thoroughly after handling. See MSDS for further information.

- 250mL water
- 400mL beaker
- 10g gelatin
- 0.25g $K_2Cr_2O_7$ (potassium dichromate)
- 20mL water
- 50mL beaker
- 2g $AgNO_3$ (silver nitrate)
- 250mL graduated cylinder (for measuring)
- 250mL graduated cylinder (for display)
- small pipette

Procedure:

1. Fill the 400mL beaker with approximately 200mL water. Heat the water just to boiling.
2. Measure 10g gelatin and add it to the beaker. Continue to heat and stir until gelatin is completely dissolved.
3. Transfer hot gelatin solution to the hood. In the hood, measure 0.25g $K_2Cr_2O_7$ (potassium dichromate) and add to the beaker.
4. Stir until solids dissolve. Solution will be yellow to dark yellow.
5. Add water to a final volume of 250mL.
6. Quickly, but carefully, transfer the mixture into the 250mL graduated cylinder, avoiding excessive air-bubble formation.
7. Leave the cylinder undisturbed for at least 24 hours.
8. The next day, gently tilt the cylinder to see whether the gel has solidified. A yellow to dark yellow gel should have formed.

9. IT IS IMPERATIVE THAT THE NEXT STEP IS TAKEN ONLY AFTER A FIRM, SOLID GEL HAS FORMED IN THE GRADUATED CYLINDER.
10. Fill the 50mL beaker with approximately 15 mL of water.
11. In the hood, measure 2g $AgNO_3$ (silver nitrate) and add it to the beaker. Stir until dissolved. Add water to final volume of 20mL.
12. Gently pipette this liquid uniformly over the top surface of the gel in the 250mL cylinder.
13. Stopper the cylinder, tape it shut, and set aside.
14. Within an hour, a bright red band about ¼-in thick should form at the top surface of the gel. Over the next few weeks, dark brown bands will appear. The spacing between these bands increases as more bands are formed.

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