

CHAPTER

20

CARBON MONOXIDE

CARBON MONOXIDE IS A COLORLESS, ODORLESS AND TASTELESS GAS formed from the incomplete combustion of carbon.



It is flammable in air and burns with a characteristic blue flame to produce carbon dioxide:



Carbon monoxide is produced whenever combustion of a carbonaceous material occurs with insufficient oxygen. It was first isolated by Joseph Priestley in the 1770s. Pure CO(g) is produced by passing CO₂(g) over charcoal heated to redness:

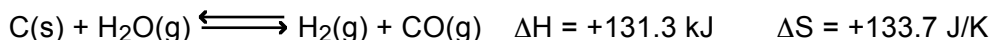


This disproportionation equilibrium between CO(g) and C(s) and CO₂(g) exists at all temperatures but is kinetically slow except at temperatures between 400 - 700 °C. The forward reaction is favored by entropy and at temperatures above 981 K (708 °C) the reaction shifts to the right as CO-formation becomes thermodynamically spontaneous. At 1000 °C, the product mixture is 99% CO(g).

Mixtures of CO(g) with other gases are produced by passing air over red hot coke, C(s). Because air is used, the resulting mixture is 70% N₂(g) and about 25% CO(g):



Steam can also be blown over red hot coke to produce mixtures of H₂(g) and CO(g). This mixture is called **water-gas**:



Carbon monoxide mixtures such as water-gas are used as a gaseous fuel. Gaseous fuels are much easier to transport than solid or liquid fuels. Gaseous fuels can be transported great distances through pipelines.

Carbon monoxide has a melting point of $-205.1\text{ }^{\circ}\text{C}$ and a boiling point of $-191.5\text{ }^{\circ}\text{C}$. The density of CO(g) is about 3% less than air. Carbon monoxide is negligibly soluble in water. Only 0.0270 g CO dissolve per L at $20\text{ }^{\circ}\text{C}$, or 100 mL of water will dissolve 2.3 mL CO.

Suitability

Both advanced chemistry (2nd year) high school and university-level chemistry students, with the skills listed below, have a suitable background to do these experiments. Experiments 3, 5, 7 and 9 make better demonstrations than laboratory experiments. Experiment 5 involves the use of an expensive substance, PdCl_2 .

Experiment 1. Blue jets!

Experiment 2. Wimpy soap bubble explosions and rockets

Experiment 3. Carbon monoxide detectors

Experiment 4. Reduction of copper(II) oxide with carbon monoxide

Experiment 5. Reduction of palladium and silver ions with carbon monoxide

Experiment 6. Carbon monoxide and potassium permanganate react

Experiment 7. Carbon monoxide poisoning

Experiment 8. Formation of a simple organometallic compound

Experiment 9. The Water-gas shift reaction

Experiments 1 and 2 are useful in demonstrating the flammable nature of CO(g) . Experiment 3 demonstrates how a carbon monoxide detector works. Experiments 4 – 6 are based on the ability of carbon monoxide to function as a mild reducing agent. Experiment 7 demonstrates the formation of the iron-CO complex that explains how CO functions as a poison in blood. In Experiment 8, an organometallic compound between Cu^+ and carbon monoxide is produced. Experiment 9 demonstrates the water-gas shift reaction, a useful reaction for the conversion of carbon-rich materials such as coal into gaseous fuels.

Background skills required

Students should be:

- ❖ able to manipulate syringes from previous experience with the In-Syringe method.
- ❖ familiar with the Thermal Method.
- ❖ able to prevent unintentional discharge of gas by using reduced pressure whenever opening the system.
- ❖ understand fundamental concepts of high school chemistry so that observations can be interpreted.

Time required

The experiments in this chapter can be performed as a combination of laboratory activities and classroom demonstrations. Carbon monoxide is relatively easy to prepare by individuals who have experience with the technique from the previous chapter. Experiments 1, 2, 4 and 6 are short laboratory activities that could be accomplished during the same laboratory period as that used for generating the gas.

Before students arrive: Assemble the equipment required

Generating gases by the Thermal Method requires the Microscale Gas Chemistry Kit as well as the special "Thermal Method Equipment" listed on the next page.

Preparation of carbon monoxide in the microwave oven

Samples of CO(g) also can be prepared conveniently in a microwave oven. See Chapter 24 for details.

Gas reaction catalyst tube

Three interesting reactions involving carbon monoxide and the Gas Reaction Catalyst Tube were given in Chapter 18, including:

- C. Oxidation of carbon monoxide with air
- G. Carbon monoxide and nitrogen dioxide
- J. Nitrous oxide and carbon monoxide

Website

This chapter is available on the web at website:

http://mattson.creighton.edu/Microscale_Gas_Chemistry.html

Instructions for your students

For classroom use by teachers. Copies of all or part of this document may be made for your students without further permission. Please attribute credit to Professors Bruce Mattson and Mike Anderson of Creighton University and this website.

GENERATING CARBON MONOXIDE¹

General Safety Precautions

Always wear safety glasses. Gases in syringes may be under pressure and could spray liquid chemicals. Follow the instructions and only use the quantities suggested.

Toxicity

Carbon monoxide has a relatively high toxicity. The molecule binds to hemoglobin about 300 times better than oxygen, thus disabling the ability of hemoglobin to carry oxygen to tissues. Symptoms of carbon monoxide poisoning include headache, mental dullness, weakness, nausea and vomiting. If any of these symptoms are noted, seek fresh air. Recovery from mild levels is rapid and complete with no cumulative effects. Higher levels of exposure can lead to unconsciousness and death.

Chemical caution: sulfuric acid

Concentrated sulfuric acid is an exceptionally dangerous chemical that causes severe chemical burns upon contact. If contact with the acid is suspected, wash area with plenty of water. Contact with the eyes may cause permanent damage and possible blindness. Wash the eyes with plenty of water and seek immediate medical attention.

Thermal Method Equipment

The following "Thermal Method Equipment" is used to generate gases by the Thermal Method. It is the same equipment that was used to produce HCl(g) in the previous chapter. You will also need the basic Microscale Gas Chemistry kit (Chapter 1).

18 x 150 mm test tube (in addition to the one in the Microscale Gas Chemistry Kit)
two-hole #1 stopper fitted with two short lengths (2 cm) of glass tubing
two pieces of tubing, 1/8-inch (3.175 mm) ID, 5 cm length
pinch clamp or hemostat
ring stand and a suitable clamp to hold test tube
wooden dowel or aluminum rod, 1 cm diameter x 15 cm length with suitable clamp
three heavy duty rubber bands
small Bunsen burner
matches or a lighter
marker pen
balance capable of measuring to 0.01 g

¹ Content for this chapter first appeared as "Microscale Gas Chemistry, Part 12. Experiments with Carbon Monoxide" Mattson, B. M., Anderson, M. P., Catahan, R., Bansal, M., Khandhar, P., Mattson, A., Rajani, A., Obendrauf, V., Vaitkus, R., *Chem13 News*, **274**, March, 1999.

MAKING CARBON MONOXIDE

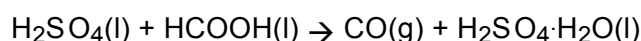
Carbon monoxide is generated by the Thermal Method, described in detail in the previous chapter and summarized here. The two chemicals to be heated together are concentrated sulfuric acid and formic acid. Very little heat is required to cause the reaction to proceed.

Chemicals required for each preparation

8 drops concentrated sulfuric acid and

8 drops (0.23 g; 5 mmol) formic acid

This quantity of reagents will produce approximately 60 mL of CO(g). The production of gas is quite fast and requires very little heat. The reaction is:



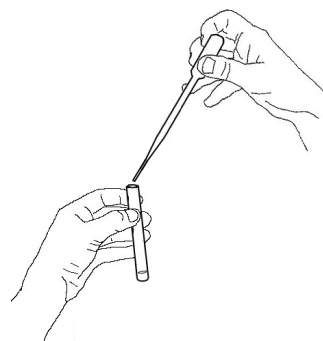
1. Wear your safety glasses!

2. Check the plunger and barrel

Make sure the syringe plunger moves easily in the syringe barrel. If it does not, try another combination of plunger and barrel.

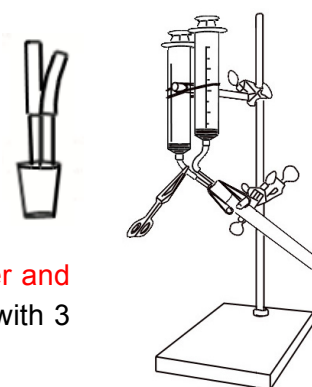
3. Measure out the liquid reagents

Add eight drops of each reagent together in the 18 x 150 mm test tube.



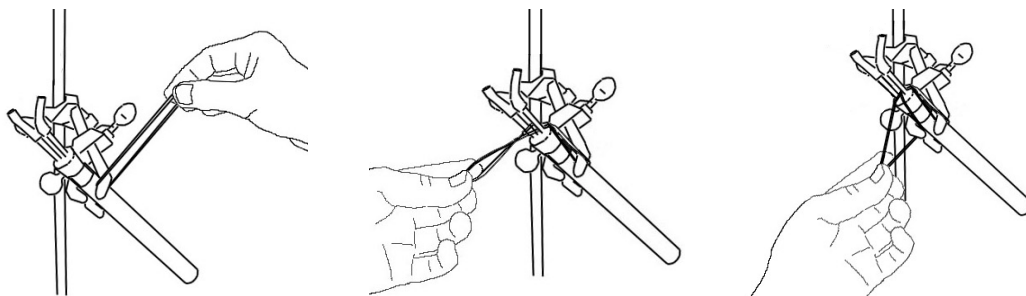
4. Assemble the apparatus

The fully assembled apparatus is shown at right. Start by clamping the test tube in position at a 45° angle. Insert a two-hole stopper equipped with two short lengths of glass tubing (the glass tubing must form an air-tight seal with the stopper). **CAUTION! Use extreme caution when inserting glass into the rubber stopper. Lubricate the stopper and glass with alcohol to make it easier.** Equip the glass tubes with 3 cm lengths of rubber tubing.



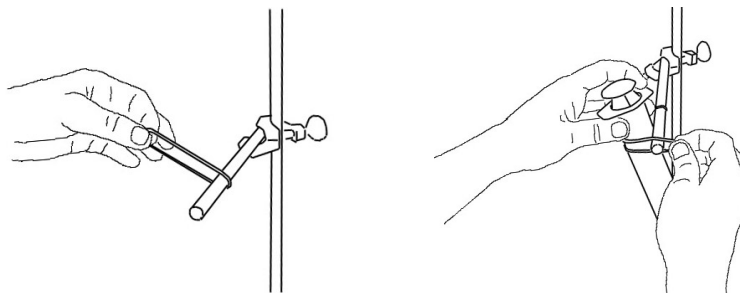
5. Secure the stopper in the test tube

Insert the rubber stopper firmly and snugly into the test tube. The stopper is held in place with a rubber band as shown in the following sequence: (Left) Hook the rubber band around one of the clamp's fingers; (Center) Maneuver the rubber band between the two glass tubes; and (Right) Hook the rubber band onto another clamp finger. The rubber band should be taut.



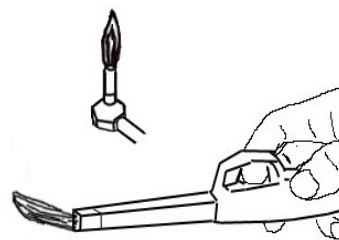
6. Position the syringes

Clamp the wooden dowel or aluminum rod in position about 15 cm above the lower clamp that holds the test tube. The two syringes are held in position by rubber bands around the dowel/rod as shown below: (Left) Slip two rubber bands (only one shown in figure) over the wooden dowel or aluminum rod; (Right) place the syringe next to the dowel/rod and then slip the rubber band around the syringe and over the dowel/rod; and then repeat with the other syringe. The use of rubber bands is preferable to using clamps because it is easier and faster to removed/replaced syringes.



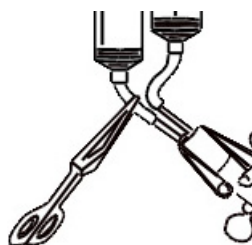
7. Burner

Prepare a burner for use. The amount of heat required depends on the gas being produced. For example, hydrogen chloride requires the use of a small burner. Carbon monoxide requires very little heat — the flame from a butane lighter is sufficient. Read gas-specific instructions carefully.



8. Hemostat/pinch clamp

A hemostat or pinch clamp is used to pinch closed the left rubber tube as shown in the close-up figure.

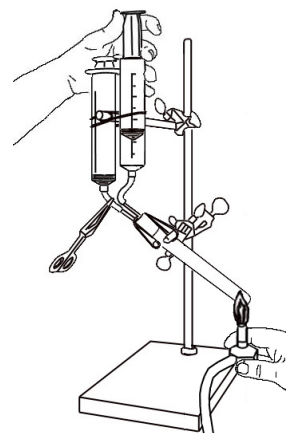


left tube clamped shut

9. Perform the reaction in three steps

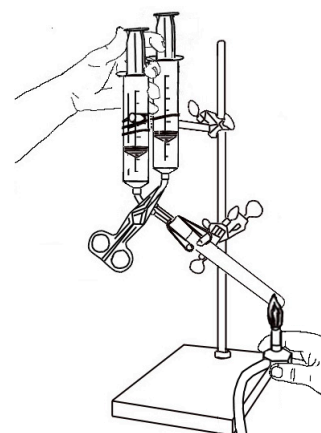
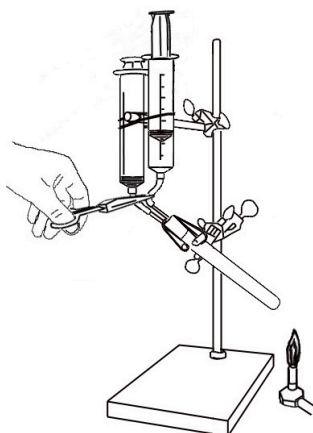
Step A. Collect air and gas in first syringe

The first syringe full of the gas collected is mostly air originally present in the test tube. Hold the heat source with one hand while helping the plunger upward — maintain reduced pressure. Gas soon will be produced and the plunger of the syringe should begin to move. It is necessary to assist the sliding movement of the plunger up the barrel of the syringe during the reaction. Continue to heat while gently assisting the plunger's movement.



Step B. Collect the gas

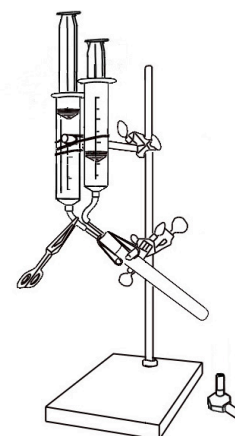
After at least 30 mL of waste gas (air) has been collected in the right syringe, switch the location of the pinch clamp (left figure) to the right rubber tube so that relatively pure product gas can be collected in the left syringe. Assist the movement of the plunger as before (right figure). Continue to collect pure gas until at least 50 mL has been collected.



Step C. Stop heating

Switch the pinch clamp back to the tubing connected to the pure gas collection syringe and remove the heat source.

While holding the plunger slightly outward in order to establish a slightly reduced pressure, remove the tube from the gas collection syringe and immediately cap the syringe. The syringe contents are > 95% pure and ready for experiments.



Allow the apparatus to cool. The plunger in the remaining syringe may move outward at first because gas generation may continue for several seconds after the test tube is removed from the flame and then move inward as the apparatus cools.

10. (optional) Collecting multiple samples

If more than the prescribed amounts of reagents were used, it would be possible to collect two or more syringes full of CO(g). Simply replace the “clamped-off” syringe (the left one in the figure above) with a clean, dry empty syringe. Switch the hemostat and continue heating.

11. Washing the gas is unnecessary

It is unnecessary to wash gases collected by the thermal method because the gas sample was collected in a clean, dry syringe.

Disposal

Unwanted samples of CO(g) including the contents of the waste syringe can be discarded in a fume hood or out of doors. The liquid remaining in the test tube is partially hydrated H₂SO₄ which can be dissolved by adding about 10 mL water or every 1 mL waste to the test tube and discarded as acidic wastes. The test tubes can be reused unless they have been damaged.

Teaching tips

1. This reaction is much simpler than the HCl reaction. The stoppers do not get slippery and pop out as easily. Nevertheless, use rubber bands to keep the system under reduced pressure and the risk of popping stoppers is eliminated.
2. After the reaction is complete, the liquid inside the test tube can be diluted with water and treated as acid waste.

Questions

1. How many moles of CO(g) can be expected from 8 drops (0.23 g) formic acid, the limiting reagent?
2. Referring to Question 1, what volume of CO(g), in mL, is expected if the temperature is 298 K and standard pressure?
3. Did you notice bubbles forming as soon as the two acids were mixed? What would happen if we did not heat the reaction?
4. How do you know that formic acid is the limiting reagent? What is the role of sulfuric acid?
5. If we used considerably more formic acid than sulfuric acid, would the reaction work as well?
6. Sketch a reaction profile for this reaction and another for the generation of HCl(g) as per the previous chapter. Which one has the larger energy of activation?

EXPERIMENTS WITH CARBON MONOXIDE

EXPERIMENT 1. BLUE JETS!

Equipment

Microscale Gas Chemistry Kit
Thermal Method Equipment (see list)
candle, affixed to a coin
glass disposable pipet
large pickle jar (for Part C)

Chemicals

CO(g), 50+ mL, several syringes
full

Suitability

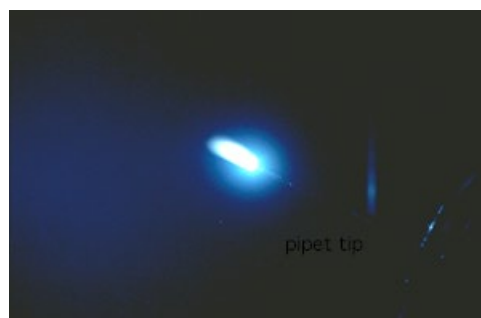
advanced high school chemistry lab, university lab, and classroom demonstration

Applications, Topics, Purpose

chemical properties of gases, combustion reactions, household chemicals

Instructions

In this experiment we will burn samples of pure CO(g) and observe the flame. Fit a 15 cm piece of tubing into a pipet and connect the other end to a syringe of CO(g). **Part A.** While holding the end of the pipet 2 - 3 cm from a candle flame, discharge 30 mL CO(g) over a period of 10 seconds through the flame. A jet of blue fire should appear on the opposite side of the candle.



Part B. While holding the end of the pipet <1 cm from the flame, slowly discharge CO(g) near the flame in order to ignite the pipet tip. Remove the tip of the pipet from the flame and the CO(g) will burn with a gentle blue flame that can be sustained by carefully discharging the CO(g) at a slow rate.

Part C. Place a candle in a large (> 1 L) vessel that can be sealed, such as a pickle jar or a desiccator. Light the candle and cover the vessel by placing the cover upside down over the opening, creating a fairly closed system. (Do not create a completely air-tight closed system because the increase in pressure from the heat may burst the bottle.) Darken the room. The candle flame will diminish in size and lose its characteristic yellow color as the O₂(g) from air is depleted. As the flame becomes smaller, note the increased size of the blue region especially near the lower part of the flame. The blue color is attributed to the combustion of CO(g) which replaces CO₂ as the dominant product of combustion and is itself combustible.

Teaching tips

1. This is probably best done as a demonstration at the high school level.
2. Remember how to discharge a few mL at a time. See Chapter 1

Questions

1. Write the reaction for the combustion of CO to produce carbon dioxide.
2. What color is produced when CO burns?
3. A burning candle produces a flame that has a region of blue near its base. What gas is present in that part of the flame to cause the blue color?
4. If carbon monoxide can burn, why does carbon dioxide not also burn?

EXPERIMENT 2. WIMPY SOAP BUBBLE EXPLOSIONS AND ROCKETS

Equipment

Microscale Gas Chemistry Kit
Thermal Method Equipment (see list)
large capacity, wide stem plastic
pipet
rocket launcher, Appendix C

Chemicals

CO(g), 50+ mL
O₂(g), 60 mL (See: Chapter 4 for
instructions)
3% dish soap solution

Suitability

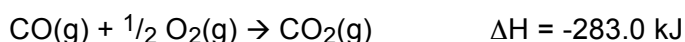
high school chemistry lab, university lab, and classroom demonstration

Applications, Topics, Purpose

chemical properties of gases, combustion, flammability of carbon monoxide

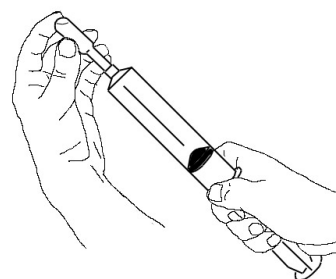
Instructions

Generate a syringe filled with O₂(g) and a syringe of CO(g). Transfer CO(g) and O₂(g) to a clean third syringe with the aid of a short length of tubing. A 2:1 volume ratio of CO(g) and O₂(g) gives the best results because it follows the stoichiometry for the reaction:



This mixture can be “exploded”: **Part A.** Form a mound of soap bubbles using dish soap solution and ignite it with a match or lighter.

Part B. Produce CO + O₂ rockets and launch them with the use of a piezoelectric sparker. You may want to review this procedure; a step-by-step color photo sequence on filling and launching rockets is available at our website. The “explosions” are mild. The rocket, for example, produces a bright flash of light but only travels a few dm. Sometimes the gas mixture will not leave the launcher but does produce a flash of light.

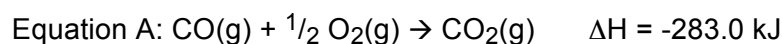


Teaching tips

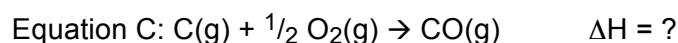
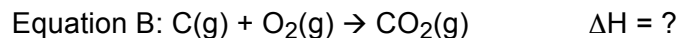
1. These demonstrations will be very tame compared to previous experience with flammable gas mixtures. The experiment provides for a useful discussion of comparisons between the flammability of various carbon-based fuels.
2. The rocket usually flies a short distance proving that a reaction has taken place. The bubbles almost seem to “pop” from the flame or spark without an obvious chemical reaction.
3. Exploding various gas mixtures such as $\text{C}_2\text{H}_2/\text{O}_2$, H_2/O_2 , and CO/O_2 would provide a useful comparison and reveal the relative reactivities of these gases.

Questions

1. Describe the reaction between carbon monoxide and oxygen. What other flammable gas mixtures have you studied with either bubbles or rockets? How does the carbon monoxide/oxygen system compare?
2. The reaction featured in this experiment is exothermic:



Look up the value for ΔH for the following two reactions and use Hess's law and the values of ΔH determined for Equations B and C to calculate ΔH for Equation A. How does it compare to the value given?



EXPERIMENT 3. CARBON MONOXIDE DETECTORS

Equipment

Microscale Gas Chemistry Kit
Thermal Method Equipment (see list)
carbon monoxide detector
4 L (1 gallon) sealable plastic bag

Chemicals

CO(g) , 5 mL

Suitability

classroom demonstration

Applications, Topics, Purpose

health, poisons, chemicals in the home

Instructions

Carbon monoxide detectors are designed to sound a warning when the CO(g) concentration exceeds a certain level for a sustained period of time. This level is

between 30 - 100 ppm. One syringe full of CO(g) discharged into 1 m³ of air produces 50 ppm CO. You will need 5 mL CO for this experiment. It is advisable to know how to reset the alarm prior to performing this demonstration. Place a digital CO(g) detector in a 1 gallon (4 L) sealable plastic bag and zip shut with plenty of air locked inside. Plug the device into the electrical service by pushing the prongs through the plastic bag. Poke a small hole with a pencil and work the 15 cm length of tubing through the hole. Connect the tubing to the syringe of CO(g) and slowly inject 1 - 5 mL of CO(g) into the bag. Most detectors need about three minutes to register CO(g) so the display will not change right away. The level of CO should be 250 ppm for 1 mL CO(g) diluted into 4 L air.

Teaching tips

1. Read the directions that come with the CO detector. Most are designed to sound an alarm after a sustained level of CO has been detected. This is to prevent cigarette smoke from setting off the alarm.
2. The digital detectors are more expensive, but worth it because the exact level is displayed — not just the annoying noise.
3. One part per million, for gases, means 1 molecule CO(g) per 1 million molecules of air. (When referring to solutions and mixtures, it means 1 mg/kg or 1 mg/L).
4. The maximum allowable level of continuous exposure to CO(g) for an 8 hour period is 50 ppm according to the Occupational Safety and Health Administration (OSHA). A sustained level of 200 ppm will produce a slight headache, dizziness, fatigue and nausea after 2 - 3 hours.

Questions

1. Why is the detector designed to wait until a high level of CO(g) is sustained before sounding the alarm?
2. Given that 1 ppm CO(g) corresponds to 1 mL CO(g) per 1,000,000 mL air, calculate the concentration in ppm of CO(g) if 5 mL of CO(g) were discharged into a gallon (4 L) bag.
3. If a syringe containing 50 mL CO(g) were discharged into a room measuring 10 m × 10 m × 3 m, and the gas were evenly distributed, what is the concentration of CO(g) in units of ppm? Hint: 1 m = 10 dm and 1 dm³ = 1 L = 1000 mL

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## EXPERIMENT 4. REDUCTION OF COPPER(II) OXIDE WITH CARBON MONOXIDE

### Equipment

Microscale Gas Chemistry Kit  
Thermal Method Equipment (see list)  
small Bunsen burner  
glass Pasteur pipet  
wooden stick such as a kabob skewer to position the copper wool into the pipet

### Chemicals

CO(g), 50+ mL  
copper wool such as a ChoreBoy kitchen scrubbing pad (without soap)  
limewater

### Suitability

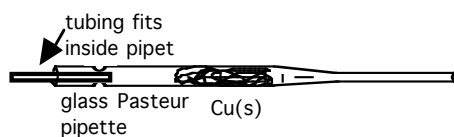
high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

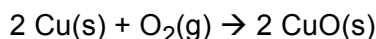
chemical properties of gases, production of metals from their ores

### Instructions

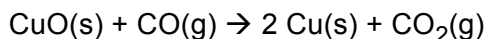
Use a wooden stick to position a 0.50-g plug of copper wool into a glass Pasteur pipet. Position the pipet horizontally using a clamp and ring stand. Fasten the clamp near the end with the tubing.



The reaction uses copper wool such as that sold as a kitchen scrubber and 50 mL CO(g). In the first part, the oxidation reaction between copper and oxygen (from air) to form black copper(II) oxide readily takes place:



The CuO(s) can be reduced back to copper with carbon monoxide gas:



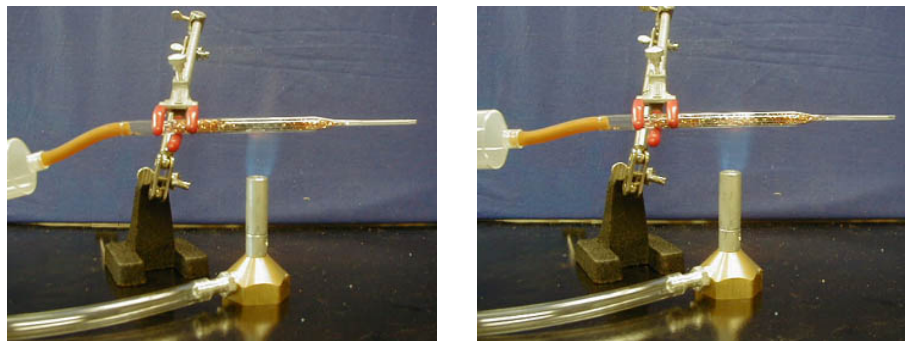
### Part A.

Connect an air-filled syringe to the pipet using a short length of tubing. Heat the Cu/pipet for 30 s and then slowly pass the air through the pipet. The copper will quickly turn black. The oxide coating is very thin but binds tightly to the surface of the copper metal.

### Part B.

Connect the CO-filled syringe to the pipet using a short length of tubing. Position a small test tube over the open tip of the pipet to collect the gas that is discharged. Heat the CuO/pipet and then slowly pass the carbon monoxide gas through the pipet. The black oxide will quickly convert to shiny metallic copper metal and will appear unchanged

from its original form (prior to oxidation in Part A). After all of the carbon monoxide has been passed through the pipet, remove the heat and allow the pipet to cool. If the copper cools in the presence of carbon monoxide, it will retain its shiny copper color. Add 2 mL of limewater to the test tube. Place your finger over the test tube opening and shake to confirm the presence of carbon monoxide.



### Teaching tips

1. The first time the copper wool is used, you may notice some smoke due to an oily film on the Cu.
2. Carbon monoxide is well known for its ability to function as a reducing agent. Metal oxides can be reduced with  $\text{CO(g)}$ . Several important industrial metallurgical processes, including the production of iron and steel in the blast furnace, are based on this reaction. This reaction demonstrates carbon monoxide's ability to function in this regard.

### Questions

1. Balance the equation for the reaction between Cu and  $\text{O}_2$ .
2. Why is it necessary to heat the Cu in order for it to react?
3. Balance the equation for the reaction between copper(II) oxide and carbon monoxide to produce copper metal and carbon dioxide.
4. Use oxidation numbers to identify the oxidizing agent and reducing agent in the equation in Question 3.
5. Calculate  $\Delta H$  for the reaction being discussed in Questions 3 and 4.

## EXPERIMENT 5. REDUCTION OF PALLADIUM AND SILVER IONS WITH CARBON MONOXIDE

### Equipment

Microscale Gas Chemistry Kit  
Thermal Method Equipment (see list)  
disposable plastic cups  
sealable (air-tight) plastic bags

### Chemicals

CO(g), 50 mL  
0.01 M PdCl<sub>2</sub>(aq) (0.177 g/100 mL)  
0.50 M PdCl<sub>2</sub>(aq) (0.89 g/10 mL)  
1 g AgNO<sub>3</sub>

### Suitability

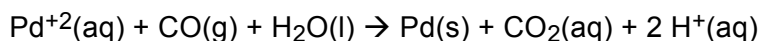
high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

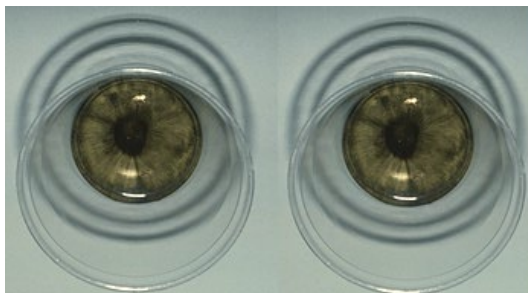
carbon monoxide as a reducing agent, formation of elemental metal from aqueous ion solutions

### Instructions

**Part A.** In the previous experiment, we saw the ability of CO(g) to function as an reducing agent. Aqueous solutions of metal ions can also be reduced by CO(g) even though the gas is only sparingly soluble in water. Solutions of 0.01 M PdCl<sub>2</sub>(aq) form black metallic elemental Pd(s) within minutes of exposure to CO(g). The reaction takes place at the surface where CO's limited solubility in water allows interaction with the metal ions. The reaction is carried out in a disposable plastic cup in a sealable (air-tight) plastic bag. More concentrated Pd<sup>+2</sup>(aq) solutions (0.5 M) form a shiny metallic surface that looks similar to nickel. The clear aqueous solution beneath the surface is acidic as expected by the balanced oxidation-reduction reaction:



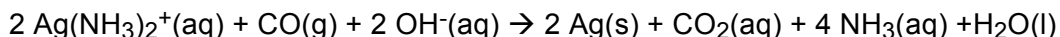
This reaction is used by medical examiners to test for the presence of CO in samples of blood from individuals suspected of CO-poisoning. The appearance of a metallic luster of palladium confirms the presence of CO.<sup>2</sup>



**Part B.** Solutions of 0.1 M AgNO<sub>3</sub> are not reduced by CO(g) unless the diammine silver(I) complex [Ag(NH<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> is first formed. Dissolve 1 g AgNO<sub>3</sub>(s) in 50 mL distilled water and slowly add concentrated ammonium hydroxide solution. A brown precipitate will initially form and then disappear

<sup>2</sup> This curious effect was made by the reduction of Pd<sup>+2</sup> as described above. Subtle vibrations on the countertop caused the crystals to migrate into the center of the plastic cup. PhotoShop was used to duplicate the image, creating a "pair of eyes"

as more (2 - 3 mL) ammonium hydroxide is added. The clear, colorless solution of  $\text{Ag}(\text{NH}_3)_2^+$  complex will react with  $\text{CO}(\text{g})$  as described for  $\text{Pd}^{+2}(\text{aq})$ . The reaction is:



### Disposal

Dispose of heavy metal samples according to local regulations. Do not discard them down the drain.

### Teaching tips

1. The website contains some spectacular pictures of this reaction. They were obtained by tapping the outside of the cup containing the suspended palladium heterogeneous solution. This created an unusual solution that looks like an eye.

### Questions

1. Are  $\text{Pd}^{+2}$  and  $\text{Ag}^+$  ions easily reduced to their elements?
2. Are metals soluble in water?
3. Could you test for carbon dioxide, one of the products of the reaction? How?
4. In the reduction of  $\text{Pd}^{+2}$  (Part A), does the pH increase or decrease as the reaction proceeds?
5. Describe the appearance of the silver formed in Part B.

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## EXPERIMENT 6. CARBON MONOXIDE AND POTASSIUM PERMANGANATE REACT

### Equipment

Microscale Gas Chemistry Kit  
Thermal Method Equipment (see list)  
test tube, 18 x 150 mm with suitable stopper

### Chemicals

$\text{CO}(\text{g})$ , 50 mL  
3 mL faintly purple ( $1 \times 10^{-4}$  M)  $\text{KMnO}_4$  solution (Appendix D)

### Suitability

high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

carbon monoxide as a reducing agent

### Instructions

Carbon monoxide reduces dilute solutions of  $\text{KMnO}_4$  to produce either colorless  $\text{Mn}^{+2}$  or brown, solid, suspended  $\text{MnO}_2$ . Transfer about 3 mL of a faintly purple  $\text{KMnO}_4$  solution to a test tube. With the 15 cm length tubing connected to the syringe, fill the test



tube with CO(g) and stopper. Upon shaking the test tube, the solution will slowly lose its pink color as the permanganate is reduced.

### Teaching tips

1. If the solution turns colorless,  $\text{Mn}^{+2}$  has been formed; if it turns brown, solid, suspended  $\text{MnO}_2$  has been formed. Both are possible.

### Questions

1. Did your reaction yield  $\text{Mn}^{+2}$  or  $\text{MnO}_2$ ?
2. Balance the appropriate oxidation-reduction reaction that took place in your experience.
3. Identify the oxidizing agent, the reducing agent, the species oxidized and the species reduced.

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## EXPERIMENT 7. CARBON MONOXIDE POISONING

### Equipment

Microscale Gas Chemistry Kit  
Thermal Method Equipment (see list)  
test tubes, 18 x 150 mm, several, with suitable stoppers

### Chemicals

CO(g), 50 mL  
absorbent pad from a package of fresh beef, pork or poultry soaked with red meat juices (keep frozen until ready for use)

### Suitability

high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

health, poisons, chemicals in the home

### Instructions

Carbon monoxide is toxic. It binds to hemoglobin about 300 times better than oxygen, thus inhibiting the ability of hemoglobin to carry oxygen to tissues. It is possible to observe the bright red color due to the CO-hemoglobin complex by using the red meat juices that soak into the absorbent pads provided in grocery store packages of meat.

Lay the pad flat in a plastic bag and freeze for easier handling. While frozen, cut the pad into strips that will fit into test tubes. Keep the strips frozen. Fill a series of

stoppered test tubes with various gases such as  $\text{N}_2$ ,  $\text{O}_2$ , air, and  $\text{CO}$ . Place one frozen strip in each test tube. Place the test tubes in a rack and store them at room temperature. Observe the test tubes over the next hour and again the next day. The sample in the  $\text{CO}$  atmosphere will become very bright red. The others will become gray. Discard all samples without handling.

### Teaching tips

1. This reaction takes several hours to develop a noticeable color change.
2. In the past, it was possible to perform experiments on samples of human blood. These days this practice is inadvisable and possibly illegal due to the risks associated with blood-borne diseases.
3. Discard all samples without handling.

### Questions

1. What color is associated with the  $\text{CO}$ -hemoglobin complex?
2. Individuals who may be suffering from  $\text{CO}$  poisoning can often recover if they are given oxygen or plenty of fresh air to replace the  $\text{CO}$  attached to their hemoglobin. Does this suggest that  $\text{CO}$  binds irreversibly or reversibly to hemoglobin?
3. What would happen to an individual who lived in a house where some  $\text{CO}$  leakage was occurring — such as from a leaking furnace system? How might that person feel?



## EXPERIMENT 8. FORMATION OF A SIMPLE ORGANOMETALLIC COMPOUND

### Equipment

Microscale Gas Chemistry Kit  
Thermal Method Equipment (see list)  
250 mL flask with suitable stopper

### Chemicals

CO(g), 30 mL  
100 mL of 3 M HCl or 25 mL concentrated HCl  
16-g anhydrous copper(I) chloride, CuCl

### Suitability

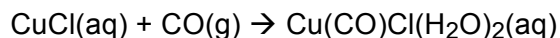
advanced high school chemistry lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

quantitative analysis of gases, organometallic compounds, Lewis acids and bases, Lewis dot structures, formal charges

### Instructions

A solution of copper(I) chloride reacts with carbon monoxide to form water soluble  $\text{Cu}(\text{CO})\text{Cl}(\text{H}_2\text{O})_2$ :



This reaction allows for the quantification of CO(g) in gas mixtures.

### Copper(I) chloride solution

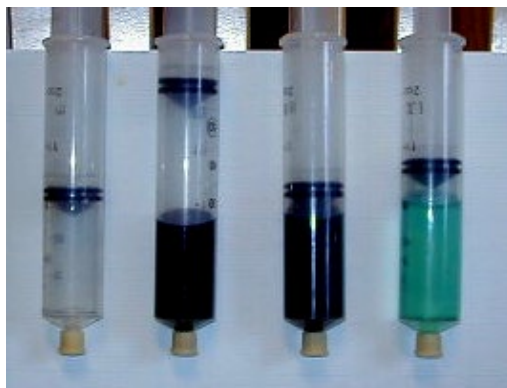
Prepare a stock solution of 1.6 M CuCl(aq) in 3 M HCl. To prepare 100 mL of the solution, prepare 100 mL of 3 M HCl by diluting 25 mL concentrated HCl to 100 mL with distilled water. To this solution, dissolve 16-g anhydrous copper(I) chloride, CuCl. The solution will be deep forest green in color. This solution will react with CO(g) in a 1:1 volume ratio: 1 mL solution will react with 1 mL CO(g).

### Part A. Quantification

Prepare a syringe filled with 30 mL pure CO(g). To this syringe draw in 30 mL 1.6 M CuCl(aq) solution. Attach the syringe cap and shake the contents *vigorously*. Within 30 s the CO(g) will have completely reacted with the solution. For quantification purposes, record the volume of gas originally present by noting the position of the plunger's rubber seal. Record the volume of gas again after the reaction is over. The volume of CO(g) originally present is equal to the difference in these two values.

### Part B. Color of Complex

In order to observe the color of the complex  $\text{Cu}(\text{CO})\text{Cl}(\text{H}_2\text{O})_2(\text{aq})$ , repeat the reaction above using 30 mL 0.4 M CuCl(aq) solution (prepared by dilution of the stock solution with water in a ratio of 1:3). Under these conditions, the complex can be observed to have a blue-green color.



from left: (a) syringe containing  $\text{CO(g)}$ ; (b) immediately after  $\text{CuCl(aq)}$  is drawn into syringe; (c) after shaking -- all of the  $\text{CO}$  reacts leaving air (d) dilution shows the color of  $\text{Cu(CO)Cl(H}_2\text{O)}_2$

### Teaching tips

1. Vigorous shaking is necessary because  $\text{CO(g)}$  is nearly insoluble in water.
2. The amount of gas that did not dissolve in the solution represents gases other than  $\text{CO(g)}$ .

### Questions

1. Sketch the Lewis dot structure of  $\text{CO}$  and assign formal charges to each atom.
2. Carbon monoxide is a reasonably good Lewis base. Which electron pair is most easily donated to a Lewis acid?
3. Consider the carbon monoxide complex formed,  $\text{Cu(CO)Cl(H}_2\text{O)}_2$ . Determine the oxidation number on copper. Is it a Lewis acid or base? Sketch the interaction between  $\text{CO}$  and the copper ion.
4. From your answers to the previous questions, how do you think carbon monoxide interacts with hemoglobin which contains an  $\text{Fe}^{+2}$  that is used to transport oxygen,  $\text{O}_2(\text{g})$ ? Sketch the Lewis dot structure of  $\text{O}_2$ , assign formal charges, and compare  $\text{O}_2$  with  $\text{CO}$  in terms of their abilities as Lewis bases.

## EXPERIMENT 9. THE WATER-GAS SHIFT REACTION

### Equipment

Microscale Gas Chemistry Kit  
 Thermal Method Equipment (see list)  
 glass tubing, suitable diameter to form a snug fit in hole of stopper, 2 cm shorter than test tube (approximately 13 cm)  
 small Bunsen burner  
 matches or a lighter

### Chemicals

$\text{CO(g)}$ , two syringes filled with 50 mL  
 fine (#00) steel wool, 2 g

## Suitability

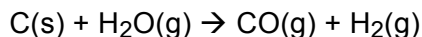
university lab and classroom demonstration

## Applications, Topics, Purpose

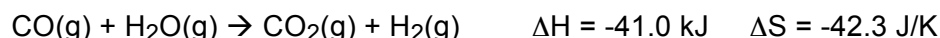
water-gas shift reaction, fuels from coal, hydrocarbon fuels

## Instructions

**Water-gas** is the name given to a mixture of  $\text{H}_2(\text{g})$  and  $\text{CO}(\text{g})$  produced by passing steam over red-hot coke:

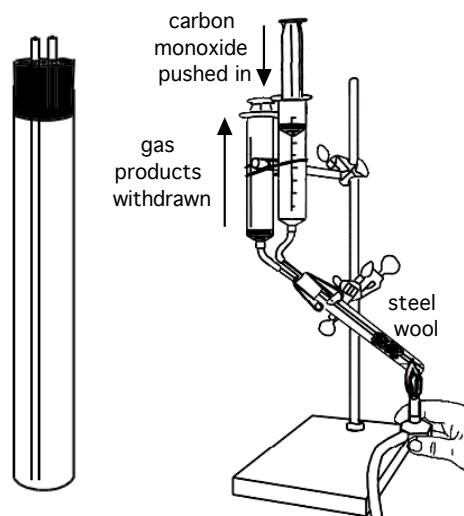


The  $\text{CO}/\text{H}_2$  mixture can be burned directly as a fuel or reacted further at a lower temperature to convert the  $\text{CO}(\text{g})$  to more desired products by means of the **water-gas shift reaction**:



The water-gas shift reaction, demonstrated in this experiment, is catalyzed by iron or  $\text{Fe}_3\text{O}_4$  at elevated temperatures.

The apparatus used for this reaction is shown in the figures. The 15 x 180 mm test tube is fitted with a 2-hole stopper equipped with a long glass tube that extends to 2 mm from the bottom of the test tube and a shorter glass tube. The latter is connected to the gas collection syringe by a short piece of tubing. An additional gas collection syringe is necessary. Lubricate the plungers of the gas collection syringes; they must move easily. Steel wool comes coated with a protective film to reduce oxidation. Unfold a 4 - 5 g piece of fine (#00) steel wool. Hold the steel wool with crucible tongs and ignite it from the bottom with a small burner (butane lighter) flame. The organic film will burn with the production of considerable smoke, leaving behind  $\text{Fe}_3\text{O}_4$ . Remove the stopper from the test tube just far enough to wrap a 2.0-g piece of the burned steel wool around the long glass tube and work it down into the test tube. The steel wool should be densely packed. Re-seat the stopper. *The glass tube should run through the steel wool as shown in the figure.* With a pipet, add 1 mL  $\text{H}_2\text{O}(\text{l})$  to the test tube via the long glass tube. The end of the glass tube should be under the surface of the water.



Push 30 mL CO(g) through the system via the long glass tube. This purges the system of air. Switch syringes so that a full syringe of CO(g) is available for the reaction. Heat the test tube directly below the steel wool to near its softening temperature as indicated by the flame becoming yellow-orange in color (ca. 400 °C) Do not heat the water. Radiant heat will cause the water to start to boil. When the water begins to boil, slowly deliver the CO(g) from the syringe. As it bubbles through the water, it sweeps H<sub>2</sub>O(g) with it through the steel wool, where the reaction is catalyzed. In order to prevent steam burns, do not switch syringes while the system is hot.

The gas collection syringe contains CO<sub>2</sub>(g) and H<sub>2</sub>(g) along with unreacted CO(g) and H<sub>2</sub>O(g, l). The contents of the syringe can be qualitatively tested for CO<sub>2</sub>(g) using limewater.

As a control, repeat the experiment without heating the catalyst. No CaCO<sub>3</sub>(s) will appear in the limewater test.

### Teaching tips

1. This experiment requires skill and experience. The results are worth the perseverance and the topic is important.

### Questions

1. What is water-gas? What is the water-gas shift reaction?
2. Review signs of  $\Delta H$  and  $\Delta S$  for the water-gas shift reaction. Is the reaction enthalpy favored? Entropy favored?
3. Use the Gibbs-Helmholtz equation to determine the temperature at which  $\Delta G = 0$  for the water-gas shift reaction. What is the significance of this temperature?
4. Why is the water-gas shift reaction useful?

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### Clean-up and storage.

At the end of the experiments, clean the syringe parts, caps and tubing with water. Rinse all parts with distilled water if available. Be careful with the small parts because they can easily be lost down the drain. **Important:** Store plunger out of barrel unless both are completely dry.

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## SUMMARY OF MATERIALS AND CHEMICALS NEEDED FOR CHAPTER 20. EXPERIMENTS WITH CARBON MONOXIDE

### Equipment required

| Item                                         | For demo | For 5 pairs | For 10 pairs |
|----------------------------------------------|----------|-------------|--------------|
| Microscale Gas Chemistry Kit (See Chapter 1) | 1        | 5           | 10           |
| Thermal Method Equipment (see list)          | 1        | 5           | 10           |
| top-loading balance                          | 1        | 2 - 3       | 3 – 5        |
| candle, affixed to a coin                    | 1        | 5           | 10           |
| pipet, plastic transfer                      | 1        | 5           | 10           |
| pipet, glass Pasteur                         | 2        | 10          | 20           |
| small Bunsen burner                          |          |             |              |
| wooden stick such as a kabob skewer          | 1        | 5           | 10           |
| disposable plastic cups                      | 1        | 5           | 10           |
| test tube, 18 x 150 mm with suitable stopper | 5        | 5           | 10           |
| small Bunsen burner                          | 1        | 5           | 10           |
| 250 mL flask with suitable stopper           | 1        | demo        | demo         |
| glass tubing*                                | 1        | demo        | demo         |
| large pickle jar                             | 1        | demo        | demo         |
| carbon monoxide detector                     | 1        | demo        | demo         |
| 4 L (1 gallon ) sealable plastic bag         | 2        | demo        | demo         |
| piezo sparker (Appendix C)                   | 1        | 2 - 3       | 3 - 5        |

\* suitable diameter to form a snug fit in hole of stopper, 2 cm shorter than test tube (approximately 13 cm)

## Materials required

| Item                                                                                    | For demo | For 5 pairs | For 10 pairs |
|-----------------------------------------------------------------------------------------|----------|-------------|--------------|
| matches or a lighter                                                                    | 1        | 1           | 1            |
| ChoreBoy kitchen scrubbing pad                                                          | 1        | 1           | 1            |
| 3% dish soap solution                                                                   | 10 mL    | 50 mL       | 100 mL       |
| absorbent pad from a package of fresh beef, pork or poultry soaked with red meat juices | 1        | demo        | demo         |
| fine (#00) steel wool                                                                   | 2 g      | demo        | demo         |
| large pickle jar                                                                        | 1        | demo        | demo         |
|                                                                                         |          |             |              |

## Chemicals required

| Item                                                  | For demo | For 5 pairs | For 10 pairs |
|-------------------------------------------------------|----------|-------------|--------------|
| sulfuric acid, concentrated                           | 10 mL    | 25 mL       | 50 mL        |
| formic acid, HCOOH                                    | 10 mL    | 25 mL       | 50 mL        |
| limewater                                             | 5 mL     | 25 mL       | 50 mL        |
| hydrogen peroxide, H <sub>2</sub> O <sub>2</sub> , 6% | 10 mL    | 50 mL       | 100 mL       |
| potassium iodide, KI                                  | 0.5 g    | 1 g         | 2 g          |
| potassium permanganate, KMnO <sub>4</sub>             | a        | a           | a            |
| palladium chloride, PdCl <sub>2</sub>                 | 1 g      | demo        | demo         |
| silver nitrate, AgNO <sub>3</sub>                     | 1 g      | demo        | demo         |
| hydrochloric acid, 3 M HCl                            | 100 mL   | demo        | demo         |
| copper(I) chloride, CuCl, anhydrous                   | 16-g     | demo        | demo         |