

## CHAPTER

# 13

# AMMONIA

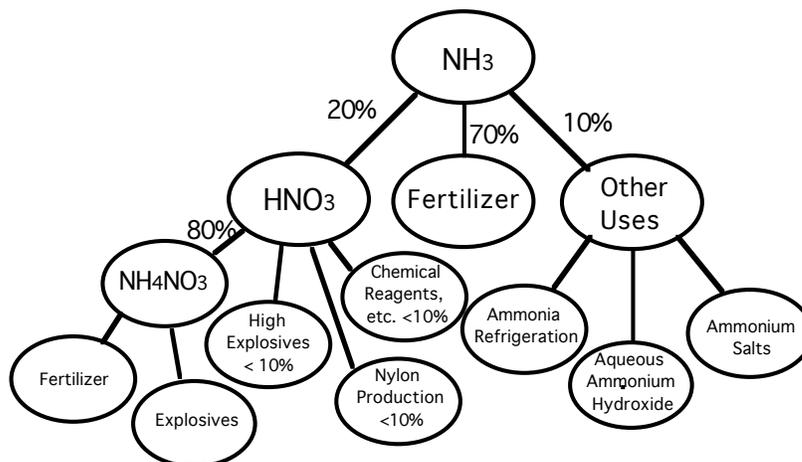
**AMMONIA IS A COLORLESS GAS WITH A SHARP, IRRITATING ODOR.** It is extremely soluble in water and is lighter than air. Ammonia was first isolated by Joseph Priestley in 1774 with the aid of a pneumatic trough filled with mercury instead of water.

Ammonia is produced by the anaerobic decay of organic material. Prior to the 20th century, ammonia was made by destructive distillation of animal parts such as hoofs, horns, etc. and was called *Spirits of Hartshorn*.

In 1913, Haber worked out a synthetic route to produce ammonia from the elements. The Haber process is still used today and operates between 400 – 500 °C and 200 – 300 atmospheres pressure.



Ammonia production is the largest consumer of nitrogen and is the gateway compound to most other nitrogen compounds produced industrially. Enormous quantities of ammonia are produced worldwide. Much of the ammonia produced in the USA is used directly for fertilizer. The following figure shows the various uses of ammonia.



*Principal Uses of Ammonia.*

The majority of all ammonia and ammonium nitrate produced is for use as fertilizer. Approximately 70% of all  $\text{NH}_3$  produced is directly used for fertilizer. Farmers inject the pressurized liquid ammonia into the soil. Ammonia is so soluble in water and has such a high affinity for polar groups such as hydroxyl groups that the ammonia is almost instantly taken up by the soil and very little is lost to the atmosphere.

Other important uses of ammonia include the production of nitric acid, use as a refrigerant, and use as a non-aqueous solvent. Ammonia's use as a refrigerant comes from the fact that it is a condensable liquid. Ammonia is currently commonly used as the refrigerant in large industrial refrigerators and freezers. Its main disadvantage in residential use stems from its toxicity and flammability should a leak develop in the system. Liquid ammonia is also the most common, widely used non-aqueous solvent. Ammonia's boiling point,  $-33.3\text{ }^\circ\text{C}$ , requires that vessels be refrigerated or pressurized. Ammonia is used in the manufacture of numerous other chemicals and products ranging from dyes to plastics.

Ammonia's melting point is  $-77.7\text{ }^\circ\text{C}$  and boiling point is  $-33.3\text{ }^\circ\text{C}$ . Ammonia can be liquefied with dry ice and used extensively as a non-aqueous solvent in synthesis. Liquid ammonia solubilizes metallic sodium to make a beautiful deep blue solution of "free electrons" and  $\text{Na}(\text{NH}_3)_x^+$  ions. The density of  $\text{NH}_3$  is  $0.6826\text{ g/L}$  at  $25\text{ }^\circ\text{C}$  and  $1\text{ atm}$ . This is about half the density of air so it tends to rise in a still room.

Ammonia is extremely soluble in water. As much as  $89.9\text{ g}$  dissolve per  $\text{L}$  at  $0\text{ }^\circ\text{C}$ . This means that  $1\text{ mL}$  of water will dissolve  $1183\text{ mL}$  ammonia! At  $100\text{ }^\circ\text{C}$ , the solubility drops to  $7.4\text{ g}$  ammonia /  $100\text{ mL H}_2\text{O}$  ( $113\text{ volumes NH}_3$  per  $1\text{ volume water}$ .)

## Suitability

All of these experiments are suited for use as classroom demonstrations or laboratory activities for high school and university-level students. Experiment 6 requires the most skill and hence, is better suited for use as a classroom demonstration. The following experiments are included in this chapter.

Experiment 1. Ammonia is a base

Experiment 2. Ammonia fountain

Experiment 3. Acid-base reactions with fruit juices

Experiment 4. Ammonia is more soluble at low temperature

Experiment 5. Gaseous ammonia reacts with gaseous hydrogen chloride

Experiment 6. Ammonia forms nitric oxide in the Ostwald process

Experiment 7. Ammonia forms complex ions with transition metals

Generally, the production of ammonia and the experiments that go with this gas should be conducted by individuals familiar with and experienced with gas production using the syringe method. Ammonia has properties that make its proper use and handling more important than was the case for carbon dioxide, hydrogen and oxygen.

All of these experiments serve to review basic concepts of chemistry including chemical properties, chemical changes, writing and working with chemical formulas, chemical reactions, and writing balanced chemical equations. In addition to these review topics, ammonia forms an aqueous solution that is common/familiar household chemical. Most of these experiments touch on these important chemistry concepts: intermolecular forces, solubility, solutions, solution equilibrium, chemical equilibrium, acids and bases.

Experiment 1, "Ammonia is a base" is used to introduce acid/base properties of gases and acid/base solution equilibrium. Experiment 3 is very similar, but allows students to bring samples of fruit juices from home for testing. Results for Experiment 3 will vary; many fruit juices do not function as indicators and thus will not change colors.

Experiment 2, "Ammonia fountain" will be everyone's favorite! Lessons learned touch on the strength of intermolecular forces, gas solubility, physical changes, polar molecules dissolve in polar solvents, and the dissolving process.

Experiment 4, "Ammonia is more soluble at low temperature" explores the effect of temperature on gas solubility.

Experiment 5, "Gaseous ammonia reacts with gaseous hydrogen chloride" is an excellent demonstration of the reaction between a Lewis acid and base. It is also spectacular because of the formation of a solid from two gases.

Experiment 6, "Ammonia forms nitric oxide in the Ostwald process", is best used as a demonstration. The formation of nitric oxide with the use of a metal catalyst provides a much needed demonstration of gas-phase catalysis.

Like Experiment 5, Experiment 7, "Ammonia forms complex ions with transition metals", is based on the formation of a Lewis acid-base adduct (complex ion formation).

### **Background skills required**

Students should be able to:

- ❖ generate a gas as learned in Chapter 1.
- ❖ know how to prevent accidental/unintentional discharge of gas (described in the Preparation of Ammonia instructions).
- ❖ understand fundamental concepts of high school chemistry so that observations can be interpreted.

## Time required

These experiments require more than one laboratory period if most are to be done by the students. Splitting the experiments between classroom demonstration and laboratory experiment is also a possibility. For example, during one 45-minute laboratory period, students could do:

Preparation of Ammonia

Experiment 1. Ammonia is a base

Experiment 2. Ammonia fountain

Experiment 4. Ammonia is more soluble at low temperature

The remaining experiments could be performed as classroom demonstrations or selected experiments could be performed during a second laboratory period. Experiments 6 and 7 are the most difficult in terms of concept understanding.

## Gas reaction catalyst tube

Two interesting reactions involving ammonia and the Gas Reaction Catalyst Tube were given in Chapter 18. Refer to these experiments:

E. Catalytic oxidation of ammonia

I. Nitrous oxide and ammonia

## Website

This chapter is available on the web at website:

[http://mattson.creighton.edu/Microscale\\_Gas\\_Chemistry.html](http://mattson.creighton.edu/Microscale_Gas_Chemistry.html)

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## 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.

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# PREPARATION OF AMMONIA<sup>1</sup>

## 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

Ammonia has a pungent irritating odor and is a poisonous gas. Exercise caution when working with poisonous gases and vacate areas that are contaminated with unintentional discharges of gas.

## Equipment

Microscale Gas Chemistry Kit (Chapter 1)  
400 mL beaker  
hot water bath or heat source

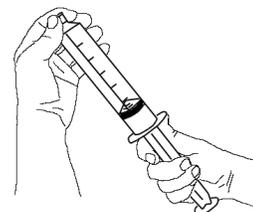
## Chemicals

3 mL concentrated ammonium hydroxide solution

## Preventing unwanted discharge of ammonia

Ammonia is a noxious gas and must not be discharged into breathable air. The use of syringes to generate such gas samples works exceptionally well and far better than any other method in preventing undesired discharges. There are two simple considerations to keep in mind whenever handling noxious gases:

(1) When opening the syringe (by removing the syringe cap), do so with the plunger slightly withdrawn so the contents are under reduced pressure. Use your thumb to maintain the plunger in this position as shown in the drawing. This will allow some air to enter the syringe but no noxious gas to escape.



(2) After the gas sample has been generated, discharge the used reagents into a large cup of water to dilute solution.

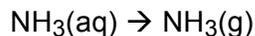


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<sup>1</sup> Content for this chapter first appeared as "Microscale Gas Chemistry, Part 6. Experiments with Ammonia" Mattson, B. M., *Chem13 News*, **256**, March, 1997.

## Generating ammonia gas samples

The following procedure will produce approximately 60 mL of  $\text{NH}_3$ . The production of  $\text{NH}_3$  is relatively slow and it typically takes between 20 - 60 seconds to fill a syringe with  $\text{NH}_3$ . The reaction is:



Fill a cup with water and set it aside. Draw 3 mL concentrated ammonium hydroxide into a clean dry 60 mL syringe. Fit the syringe cap over the syringe fitting. Place the syringe in 400 mL beaker of hot (60 - 70 °C) water for several minutes. **Care must be taken to stop the gas generation after the syringe is full by removing the syringe cap while it is directed upwards.** Rotate the syringe 180° in order to discharge the aqueous ammonia solution and then recap the syringe. **CAUTION!** The liquid will vigorously spray out of the syringe. In order to control the spray and minimize the ammonia odor, discharge the liquid at close range above the surface of the cup of water.

Several syringes of  $\text{NH}_3(\text{g})$  can be generated at the same time if a larger beaker (1 L) of hot water is used, but it is best to stagger their starting times in the hot bath so that they are not all ready to come out at the same time. After the liquid has been discharged, store the  $\text{NH}_3$ -filled syringes in the hot water bath until needed.

## Preparation of Ammonia in the microwave oven

Samples of  $\text{NH}_3(\text{g})$  also can be prepared conveniently in a microwave oven. See Chapter 24 for details.

## Washing ammonia samples is not possible

Do not wash the  $\text{NH}_3$ -filled syringes. Ammonia is extremely soluble in water. Instead, it is possible to transfer the  $\text{NH}_3(\text{g})$  sample to a clean, dry syringe via a short length of tubing — although for the experiments described in this chapter, this is unnecessary.

## Disposal of ammonia samples

Unwanted  $\text{NH}_3(\text{g})$  samples can be destroyed by bubbling the gas through a cup of water.

## Teaching tips

1. Several syringes can be made at the same time provided a large enough water bath is used. Stagger their starting times so that they are not all ready to come out of the bath at the same time.
2. The solubility of a gas in water increases as the temperature decreases. Keep the samples warm or transfer them to a dry syringe.

3. Ammonia has a very pungent odor and is used as smelling salts.

### Questions

1. To generate your  $\text{NH}_3(\text{g})$ , why did you place the syringe with 3 mL of concentrated ammonium hydroxide solution in a hot water bath?
2. Why does the volume of  $\text{NH}_3(\text{g})$  decrease upon standing at room temperature?



## EXPERIMENTS WITH AMMONIA

### EXPERIMENT 1. AMMONIA IS A BASE

#### Equipment

Microscale Gas Chemistry Kit

#### Chemicals

$\text{NH}_3(\text{g})$ , 60 mL

3 mL universal indicator solution (or 10 mL cabbage juice)

1 drop 2 M  $\text{HCl}(\text{aq})$

#### Suitability

high school lab, university lab, and classroom demonstration

#### Applications, Topics, Purpose

acid/base properties of gases, chemical properties of gases, indicators, household chemicals, properties of ammonia

#### Instructions

Ammonia is a weak base and in this experiment, its basic nature is demonstrated. To 100 mL water add 1 drop of 2 M  $\text{HCl}(\text{aq})$  and one of the following: (a) 3 mL universal indicator solution or (b) 10 mL cabbage juice solution. Shake to mix. Remove the syringe cap from an ammonia-filled syringe and equip it with a 15 cm length of tubing. Slowly discharge the ammonia gas just above the surface of the universal indicator solution. As the ammonia comes in contact with the water, it will dissolve and raise the pH in the vicinity of the surface. A pH gradient of several colors often develops upon standing.

#### Teaching tips

1. The universal indicator solution, adjusted to a low pH with 1 M  $\text{HCl}$ , becomes basic when exposed to gaseous ammonia.

2. Provide a chart of indicator color vs. the corresponding pH to your students.

Indicator Colors		
pH	Universal	Red Cabbage
4.0	Red	Red
5.0	Orange Red	Purple
6.0	Yellow Orange	Purple
7.0	Dark Green	Purple
8.0	Light Green	Blue
9.0	Blue	Blue-Green
10.0	Reddish Violet	Green
11.0	Violet	Green
12.0	Violet	Green
13.0	Violet	Green-Yellow
14.0	Violet	Yellow

### Questions

1. What was initial the pH of the solution before you discharged any  $\text{NH}_3(\text{g})$  over the solution?
2. What was the pH of the solution after you discharged the  $\text{NH}_3(\text{g})$  over the surface? Did the pH of the solution increase or decrease? Does that imply that at solution is more acidic or less acidic?
3. Was  $\text{NH}_3(\text{g})$  soluble in the water solution? Explain based on your observations. Did you detect any ammonia odor?
4. Is  $\text{NH}_3(\text{g})$  an acidic or basic compound? Explain based on your observations.
5. Why was the color of the solution different at the top than at the bottom?



## EXPERIMENT 2. AMMONIA FOUNTAIN

### Equipment

Microscale Gas Chemistry Kit

### Chemicals

$\text{NH}_3(\text{g})$ , 60 mL (one syringe for each experiment)

1 mL phenolphthalein solution

### Suitability

high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

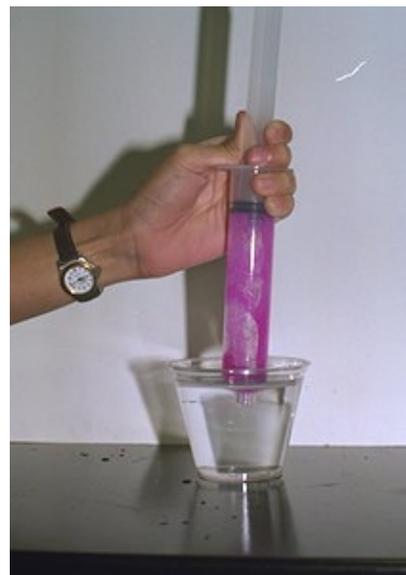
indicators, household chemicals, intermolecular forces, solubility, physical changes, properties of ammonia, polar molecules, solutions, the dissolving process, solution equilibrium, chemical equilibrium, acids and bases

### Instructions

Add 1 mL phenolphthalein solution to a cup filled with water. Remove the syringe cap from an ammonia-filled syringe. **Firmly** grasp the syringe plunger so that it cannot slip into the syringe barrel. Plunge the syringe's fitting under the surface of the water and observe the immediate fountain formed. There is a considerable force pulling the plunger inward.

### Teaching tips

1. Draw the Lewis structures for both  $\text{NH}_3$  and  $\text{H}_2\text{O}$ . Label the more electronegative atoms (N and O) with a  $\delta^-$  and the less electronegative atoms (hydrogen) with a  $\delta^+$ .
2. Sketch the Lewis structure of one water molecule and one ammonia molecule with the oxygen atom of water close to a hydrogen atom of ammonia. Draw a dotted line from the water's oxygen atom ( $\delta^-$ ) to a hydrogen atom ( $\delta^+$ ) on ammonia. This picture will illustrate one possible attractive force between these two molecules.
3. Ammonia is very soluble in water because both molecules are polar covalent and form hydrogen bonding attractive forces with each other. The  $\delta^-$  oxygen of water forms a hydrogen bond attractive force with the  $\delta^+$  hydrogen atoms on the  $\text{NH}_3$  molecule while the  $\delta^+$  hydrogen atoms on the  $\text{H}_2\text{O}$  molecule form attractive forces to the  $\delta^-$  nitrogen on ammonia. There are several possible  $\delta^-$  to  $\delta^+$  attractions.



## Questions

1. Explain the reason for fountain seen inside the syringe.
2. Based on what you learned in this experiment, how would you describe the solubility of ammonia gas in water?
3. Draw the Lewis structure of ammonia. Draw the Lewis structure of water. What attractive forces (dipole-dipole, London dispersion or hydrogen bonding) would ammonia molecules form with water molecules?
4. Hydrogen gas is collected in the laboratory by bubbling it through water. Is it possible to collect  $\text{NH}_3(\text{g})$  in this manner? Explain why or why not.

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## EXPERIMENT 3. ACID-BASE REACTIONS WITH FRUIT JUICES

### Equipment

Microscale Gas Chemistry Kit

### Chemicals

$\text{NH}_3(\text{g})$ , 60 mL

variety of fruit drinks (students bring samples)

### Suitability

high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

indicators, household chemicals, properties of ammonia, acids and bases

### Instructions

Place 3 mL of a fruit juice in a cup and add 20 mL water. Draw the fruit juice solution into the ammonia-filled syringe. Cranberry juice will turn purple and then a deep green. Grape juice turns forest green.

### Teaching tips

1. Ask students to bring fruit juice samples from home. Results for this experiment will vary; many fruit juices do not function as indicators and thus will not change colors.
2. Once in the lab, fruit juices should be considered to be chemical reagents and must never be tasted.
3. It will take an entire syringe full of ammonia for each fruit drink because the concentration of the juices is quite high. Try diluting the juices.

4. This is probably an experiment for the whole class. Suggest that each student or team use a different fruit juice, work out the details and then share their results in a short presentation to the rest of the students.

5. The fruit juices are acidic and have a sour taste. Bases have a bitter taste and the  $\text{NH}_3(\text{g})$  is basic. This is an example of an acid - base neutralization reaction.

### Questions

1. Acids have a sour taste and bases have a bitter taste. Are (unsweetened) fruit juices acidic or basic?
2. Is  $\text{NH}_3(\text{g})$  an acid or a base?
3. Did the pH of the fruit juice increase or decrease after you added the  $\text{NH}_3(\text{g})$ ?
4. What is the relation between the pH and acid concentration?
5. What type of chemical reaction occurred when you added  $\text{NH}_3(\text{g})$  to fruit juice, acid and base, redox, or precipitation?

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## EXPERIMENT 4. AMMONIA IS MORE SOLUBLE AT LOW TEMPERATURE

### Equipment

Microscale Gas Chemistry Kit  
large beaker (600 - 800 mL) or cut off 2 L plastic beverage bottle

### Chemicals

Concentrated ammonium hydroxide,  $\text{NH}_3(\text{aq})$ , 3 mL  
ice

### Suitability

high school lab, university lab, and classroom demonstration

### Applications, Topics, Purpose

household chemicals, intermolecular forces, solubility, physical changes, properties of ammonia, polar molecules, solutions, the dissolving process, solution equilibrium

### Instructions

Generate ammonia as per the general instructions, but *do not discharge the water present*. Prepare a 600 mL cold water bath with a temperature of 0 - 10 °C. Place the  $\text{NH}_3(\text{g})$ -filled syringe into the cold bath. (Do not remove the syringe cap.) Within 5 minutes the syringe will appear as it did before gaseous ammonia was generated: simply 3 mL liquid in a syringe with the plunger completely in. The process can be repeated by placing the syringe in the hot water bath again. The equilibrium reaction is:



### Teaching tips

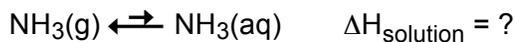
1. Students can regenerate the ammonia gas and use it for another experiment.
2. All gases are all more soluble at low temperatures. (Think of carbon dioxide in carbonated beverages.)

### Questions

1. How quickly did the ammonia re-dissolve in the water present?
2. Why is  $\text{NH}_3(\text{g})$  so water-soluble?
3. Would re-heating the  $\text{NH}_3(\text{aq})$  produce  $\text{NH}_3(\text{g})$  again?
4. Sketch a qualitative plot of ammonia's solubility (y-axis) vs. temperature (x-axis)?
5. Given the value for  $\Delta H$  for the following equilibrium, sketch a reaction profile. When sketching the activation energy, should you use a large or small energy of activation "hill"?



6. Calculate  $\Delta H_{\text{solution}}$  for the following reaction:



7. Given  $\Delta H^\circ$  and  $\Delta S^\circ$  in for the reaction under investigation, calculate  $\Delta G^\circ$ .

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## EXPERIMENT 5. GASEOUS AMMONIA REACTS WITH GASEOUS HYDROGEN CHLORIDE

### Equipment

Microscale Gas Chemistry Kit

### Chemicals

$\text{NH}_3(\text{g})$ , 60 mL

concentrated hydrochloric acid,  $\text{HCl}(\text{aq})$ , a few drops (See Precaution!)

### Suitability

university lab and classroom demonstration

### Precaution

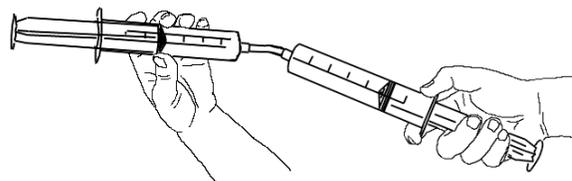
Concentrated hydrochloric acid,  $\text{HCl}$ , is a strong acid and dangerous chemical capable of causing severe chemical burns. In the event of dermal contact, immediately wash area with plenty of water and seek medical attention. Do not inhale vapors.

## Applications, Topics, Purpose

Lewis acids and bases, physical and chemical changes, properties of ammonia, energy and chemical change, chemical formulas, writing balanced chemical equations, classifying chemical changes, chemical reactivity of ammonia, chemical bonding, molecular structure, polar molecules, kinetic theory of gases, rates of chemical reactions (chemical kinetics), precipitation reactions

## Instructions

Equip a syringe with a 15 cm length of tubing. Fill the syringe with gaseous hydrogen chloride by drawing the head space fumes of  $\text{HCl(g)}$  from a bottle of concentrated hydrochloric acid. (The contents of this syringe will be mostly air with varying amounts of gaseous  $\text{HCl}$ .) Connect the  $\text{HCl(g)}$ -filled syringe with an  $\text{NH}_3$ -filled syringe as shown in the figure. Slowly push on the plunger of the ammonia syringe to produce plumes of white  $\text{NH}_4\text{Cl(s)}$  in the  $\text{HCl}$ -filled syringe. Variant: Tap firmly on the plunger of the  $\text{NH}_3$ -filled syringe so that the plunger moves inward 1 - 2 mL at a time. This will produce interesting "smoke rings" in the  $\text{HCl}$ -filled syringe.



## Teaching tips

1. Review safety precautions regarding concentrated hydrochloric acid.

## Questions

1. What is the formula of the salt produced?
2. Write the chemical reaction that took place.
3. Do you expect this salt to be soluble in water?
4. Why did the plunger(s) move inward?
5. Why did the plunger(s) stop moving inward? How would things be different if you were to use pure (100%)  $\text{HCl(g)}$  and  $\text{NH}_3(g)$ ?



## EXPERIMENT 6. AMMONIA FORMS NITRIC OXIDE IN THE OSTWALD PROCESS

### Equipment

Microscale Gas Chemistry Kit  
fume hood  
glass stir rod or pencil  
Bunsen burner  
matches or lighter  
pliers or tongs

### Chemicals

NH<sub>3</sub>(g), 30 mL  
O<sub>2</sub>(g), 30 mL (See Chapter 4 for in-syringe preparation, or make oxygen in a gas bag — See Chapter 5)  
30 cm length of 20 gauge copper

### Suitability

university lab and classroom demonstration

### Applications, Topics, Purpose

catalysts and catalysis, chemical changes, properties of ammonia, energy and chemical change, chemical formulas, chemical reactions, writing balanced chemical equations, oxidation-reduction reactions

### Instructions

One of the primary industrial uses of ammonia is in the Ostwald process in which NH<sub>3</sub> is oxidized to NO:



The nitric oxide is converted to NO<sub>2</sub>(g) and then to nitric acid as we discussed in Chapter 12. In the actual Ostwald process, screens made from platinum are used as the catalyst.

Transfer 30 mL O<sub>2</sub>(g) from the O<sub>2</sub>-filled syringe to the NH<sub>3</sub>-filled syringe with the aid of a 3 cm length of tubing. The gases do not react at room temperature so that it is necessary to maintain the same total volume by pulling the NH<sub>3</sub> plunger outward as the O<sub>2</sub> plunger is pushed inward. Remove the tubing from the syringe filled with the gas mixture and cap the syringe until needed.

The reaction should be done in a working fume hood. In this experiment we will use a coil of copper wire. Construct a coiled copper wire as shown in the figure by winding a 30 cm length of 20 gauge copper around a glass stir



rod or pencil. The coils should be close to one another. Light a Bunsen burner and adjust it to make a hot flame. Set the coiled wire and a set of pliers or tongs near the burner for use later.

Remove the syringe cap from the gas mixture. While holding the copper wire with the pliers or tongs, heat the coiled portion of the wire at the top of the flame's inner cone until it glows brightly red. Remove the coil from the flame and quickly start to discharge the gas mixture at "point blank" range to the red hot copper (but not touching it). The coils will glow as the wire catalyzes the reaction. The heat given off from the reaction maintains the copper at red heat. More than one attempt may be necessary before this works properly. As the reaction proceeds, note the reddish colored  $\text{NO}_2$  gas generated by the reaction of  $\text{NO}(\text{g})$  with the  $\text{O}_2(\text{g})$  present. **CAUTION:** The syringes are easily damaged by heat so avoid contact between the hot coil and the syringe barrel.

### Teaching tips

1. Work in a fume hood in order to prevent exposure to fumes of nitrogen oxide gases.
2. In the actual Ostwald process, screens made from platinum are used as the catalyst.
3. Catalysts change the mechanism or pathway a chemical takes place. The altered mechanism has a lower energy of activation and the reaction is faster.

### Questions

1. Why are red fumes observed if  $\text{NO}(\text{g})$  is being produced?
2. How is  $\text{NO}(\text{g})$  used to form nitric acid? What are the next steps?
3. What is a catalyst?
4. Why does the copper wire stay hot as long as the ammonia/oxygen mixture is being discharged over its surface?



## EXPERIMENT 7. AMMONIA FORMS COMPLEX IONS WITH TRANSITION METALS

### Equipment

Microscale Gas Chemistry Kit  
12-or 24-well plate

### Chemicals

$\text{NH}_3(\text{g})$ , 60 mL (one syringe full of ammonia per solution)  
 $\text{CoCl}_2(\text{aq})$ , 3 – 5 mL, faintly pink       $\text{CuSO}_4(\text{aq})$ , 3 – 5 mL, faintly blue  
 $\text{NiCl}_2(\text{aq})$ , 3 – 5 mL, faintly green       $\text{AgNO}_3(\text{aq})$ , 3 – 5 mL, dilute

### Suitability

university lab and classroom demonstration

### Applications, Topics, Purpose

chemical properties of ammonia, Lewis acid-base compounds, complex ion formation, chemical formulas, chemical reactions, writing balanced chemical equations, chemical bonding, molecular structure, chemical equilibrium

### Instructions

The following reactions are performed in a 12-or 24-well plate. Prepare the following reagents in separate wells before generating  $\text{NH}_3(\text{g})$ .

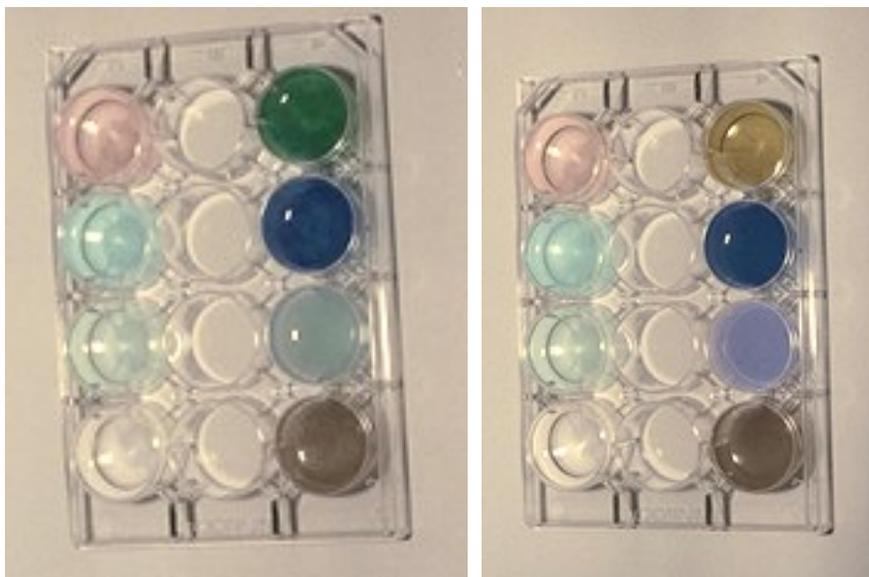
<b>Well:</b>	<b>Contains:</b>	<b>Results:</b>
1	$\text{CoCl}_2(\text{aq})$	Initially forms green-blue precipitate; then forms soluble orange $[\text{Co}(\text{NH}_3)_6]\text{Cl}_2(\text{aq})$
2	$\text{CuSO}_4(\text{aq})$	Initially forms pale blue-green precipitate; then forms soluble deep blue-purple $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4(\text{aq})$
3	$\text{NiCl}_2(\text{aq})$	Forms soluble purple $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2(\text{aq})$
4	$\text{AgNO}_3(\text{aq})$	Initially forms brown precipitate; then forms soluble colorless $[\text{Ag}(\text{NH}_3)_2]\text{NO}_3(\text{aq})$

One syringe full of  $\text{NH}_3(\text{g})$  may be necessary for each of these experiments. Equip the syringe with a 15 cm length of tubing. Into each well **slowly** discharge enough  $\text{NH}_3(\text{g})$  through the solution to achieve the desired results, namely a transparent solution of the stated color. If the precipitate has not disappeared by the time the volume of  $\text{NH}_3(\text{g})$  is reduced to 10 mL, hold the tubing in the metal ion solution until the solution moves up the tubing and into the syringe body.

Dispose of metal ion solutions according to local regulations.

## Teaching tips

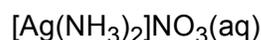
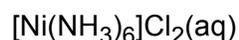
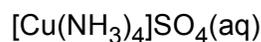
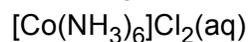
1. Prepare 50 mL of the following 0.1 molar metal ion solutions by dissolving:
  - a. 1.19 g  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  in  $\text{H}_2\text{O}$  to make 50 mL
  - b. 1.25 g  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in  $\text{H}_2\text{O}$  to make 50 mL
  - c. 1.19 g  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  in  $\text{H}_2\text{O}$  to make 50 mL
  - d. 0.85 g  $\text{AgNO}_3$  in  $\text{H}_2\text{O}$  to make 50 mL
2. If time is short, use one well plate for the entire class and have each student bubble their  $\text{NH}_3(\text{g})$  into a well.
3. The following changes take place in the different wells.
  - a. The faintly pink  $\text{CoCl}_2(\text{aq})$  initially forms a green-blue precipitate; then forms soluble orange  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_2(\text{aq})$  after adding excess  $\text{NH}_3(\text{g})$ .
  - b. The faintly blue  $\text{CuSO}_4(\text{aq})$  initially forms pale blue-green precipitate; then forms soluble deep blue-purple  $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4(\text{aq})$  after adding excess  $\text{NH}_3(\text{g})$ .
  - c. The faintly green  $\text{NiCl}_2(\text{aq})$  forms soluble purple  $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2(\text{aq})$  after adding excess  $\text{NH}_3(\text{g})$ .
  - d. Dilute  $\text{AgNO}_3(\text{aq})$  initially forms brown precipitate; then forms soluble, colorless  $[\text{Ag}(\text{NH}_3)_2]\text{NO}_3(\text{aq})$  after adding excess  $\text{NH}_3(\text{g})$ .



Left: Before addition of ammonia. Right: After exposure

## Questions

The following compounds were produced in this experiment:



1. Write the formula of each complex ion produced in this experiment. Include the square brackets and charge. The first one is:  $[\text{Co}(\text{NH}_3)_6]^{+2}(\text{aq})$
2. Determine the oxidation state for the metal in each complex ion. The first one is:  $\text{Co}^{+2}$
3. What is the coordination number of the complex ion in each compound? For  $\text{Co}^{+2}$ , the answer is six.

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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 13. EXPERIMENTS WITH AMMONIA

### Equipment required for Part 1: Student Experiments (Experiments 1 – 5)

Item	For Demo	For 5 pairs	For 10 pairs
Microscale Gas Chemistry Kit (See Chapter 1)	1	5	10
400 mL beaker	1	2 - 3	4 - 5
800 mL beaker*	1	2 - 3	4 - 5

\* or cut off 2 L plastic beverage bottle

### Materials required for Part 1: Student Experiments (Experiments 1 – 5)

Item	For Demo	For 5 pairs	For 10 pairs
fruit juices, various	a	a	a

a. students bring various samples

### Chemicals required for Part 1: Student Experiments (Experiments 1 – 5)

Item	For Demo	For 5 pairs	For 10 pairs
concentrated ammonium hydroxide solution	15 mL	50 g	100 g
universal indicator solution*	3 mL	15 mL	30 mL
hydrochloric acid, 2 M HCl(aq)	1 drop	1 mL	2 mL
hydrochloric acid, conc., HCl(aq)	1 drop	1 mL	2 mL
phenolphthalein solution	1 mL	5 mL	10 mL
ice	1 cup	5 cups	10 cups

\* or cabbage juice (generally, you will need to use more cabbage juice than universal indicator)

### Equipment required for Part 2: Advanced Experiments and Demonstrations (Experiments 6 – 7)

Item	For Demo	For 5 pairs	For 10 pairs
Microscale Gas Kit (See Chapter 1)	1	5	10
Bunsen burner, small	1	5	10
fume hood	1	1 – 3	2 - 5
glass stir rod or pencil	1	5	10
matches or lighter	1	5	10
12-or 24-well plate	1	5	10

### Materials required for Part 2: Advanced Experiments and Demonstrations (Experiments 6 – 7)

Item	For Demo	For 5 pairs	For 10 pairs
20 gauge copper wire	30 cm	5	10
pliers or tongs	1	5	10

### Chemicals required for Part 2: Advanced Experiments and Demonstrations (Experiments 6 – 7)

Item	For Demo	For 5 pairs	For 10 pairs
potassium iodide, KI, powder	1 g	2 g	5 g
6% H <sub>2</sub> O <sub>2</sub> (aq)*	10 mL	50 mL	100 mL
CoCl <sub>2</sub> (aq), 0.1 M	3 – 5 mL	1 g solid	2 g solid
CuSO <sub>4</sub> (aq), 0.1 M	3 – 5 mL	1 g solid	2 g solid
NiCl <sub>2</sub> (aq), 0.1 M	3 – 5 mL	1 g solid	2 g solid
AgNO <sub>3</sub> (aq), 0.1 M	3 – 5 mL	1 g solid	2 g solid

\* 3% H<sub>2</sub>O<sub>2</sub>(aq) will also work, but not quite as well