CHAPTER 24

MAKING GASES IN A MICROWAVE OVEN

A SIMPLE MICROWAVE OVEN PROVIDES AN INTERESTING ALTERNATIVE for generating some of the gases previously described in this series. The microwave oven provides distinct advantages over traditional laboratory methods. Gases can be generated faster, the use of open flames is eliminated, and the quantities of reagents can be reduced. In this article we will describe the generation of six different gases in the microwave oven. Ammonia, oxygen, carbon monoxide, sulfur dioxide, methane and hydrogen chloride are conveniently generated in a microwave oven.

Preparation of Gases

Details for each gas are given below and summarized in the Table. When using a microwave oven to generate gases, the progress of the reaction should be checked periodically during the allotted time. Each microwave oven is different, so the times given may vary.

Table. Summary of Gas Preparation Conditions for Microwave Methods.

Gas	Time	Microwave Method	Chemicals	Microwave Setting
NH_3	30 - 40 s	inside syringe	1 mL conc NH_4OH	500 W
O_2	7 - 10 s	inside syringe	5 mL H_2O_2 + KI crystal	500 W
CO	< 5 s	inside syringe	10 drops HCOOH + 10 drops H_2SO_4	500 W
SO_2	5 - 7 s	inside syringe	0.25 g $NaHSO_3$ + 3 mL 6 M HCl	500 W
CH_4	2 - 3 min	medium test tube	0.5 g $NaC_2H_3O_2$ + 0.5 g NaOH	400 W
HCl	2 - 3 min	small test tube	2 g $NaHSO_4$(anhyd) + 1 g NaCl	500 W

Suitability

All of these experiments are suited for use as classroom demonstrations. Individuals attempting these experiments should be experienced with the simpler syringe/gas techniques. Advanced students or students with special laboratory skills could be allowed to perform these experiments under close supervision by the instructor.

Background skills required

Students should be:

- ❖ able to manipulate syringes from previous experience with the In-Syringe method.

More on microwave ovens

The microwave oven has been used throughout science in many ways throughout its 50-year history. Our website contains numerous links to the use of microwave ovens in science.

Website

This chapter is available on the web at website:

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

Instructions for your students

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GENERATING GASES IN A MICROWAVE OVEN

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

Four of the six gases described here, ammonia, carbon monoxide, sulfur dioxide, and hydrogen chloride, have relatively high toxicities. Of these four, only CO has no odor; it is also the least toxic considering the small quantity produced. Do not discharge these gases into the room. Oxygen and methane are non-toxic in the quantities used. Methane is flammable in air and forms explosive mixtures with air. Use appropriate caution when working with sulfuric acid.

Equipment

The following equipment is used in this chapter:

microwave oven
several 60 mL plastic syringes
several syringe caps
tubing, 1/8-inch (3.175 mm) ID, 15 cm length and 4 cm length
gallon (4 L) sealable storage bags (such as "Ziploc" or equivalent)
silicone oil
glass Pasteur pipet
Additional equipment necessary for generating $CH_4(g)$ and HCl(g): 250 mL beaker (or similar), medium test tube (18 x 150 mm), small test tube (13 x 100 mm); each test tube should be fitted with a one-hole rubber stopper and each stopper should be fitted with a 4 cm length of glass tubing (must be a air-tight fit — use alcohol to lubricate rubber stopper and glass before inserting glass into stopper)

INSIDE SYRINGE MICROWAVE METHOD

The preparation of NH_3, O_2, CO, and SO_2 are performed by this method. In each case, the appropriate amount of reagent(s) is/are added to the syringe, and the syringe is capped. As a precautionary measure to protect the microwave oven, the syringe is placed inside a 4 L sealable plastic bag. Position the syringe diagonally inside the bag so that the plunger can move outward unimpeded by the bag.

Preparation of gaseous ammonia in a microwave oven

In Chapter 13, gaseous ammonia was generated by placing a syringe containing 3 mL concentrated ammonium hydroxide in a hot water bath (60 - 70 ^{o}C) for 20 - 60 seconds. The reaction is:

$$NH_3(aq) \rightarrow NH_3(g)$$

The same reaction can be performed in a microwave oven (on high setting, 500 W) in 30 - 40 seconds. Draw 3 mL concentrated ammonium hydroxide into a syringe and cap the syringe. Place the syringe diagonally in a sealable 4 L plastic bag and set the bag in the microwave oven. Start the microwave oven and check the extent of gas production every 10 seconds. After 50 - 60 mL $NH_3(g)$ has been collected, remove the syringe from the bag and remove the syringe cap while the cap is directed upwards. Rotate the syringe 180^{o} and discharge the warm ammonia solution at close range above the surface of a large (> 250 mL) container of water (to prevent excessive ammonia odor.) Recap the syringe. ***CAUTION!*** The liquid will vigorously spray out of the syringe.

Preparation of oxygen in a microwave oven

The generation of oxygen is typically slow when done by the method described in Chapter 4. Oxygen is generated by the decomposition of 6% hydrogen peroxide with a small quantity of potassium iodide serving as a catalyst. Using a microwave oven, the reaction takes only a few seconds compared to over a minute by our original method.

Lubricate the plunger's rubber seal with silicone oil. Drop a few crystals of solid KI into the syringe barrel and install the plunger fully into the barrel. Draw 5 mL 6% $H_2O_2(aq)$ into the syringe and immediately cap the syringe with the syringe cap. Place the syringe inside a sealable storage bag and seal the bag. The syringe must be positioned so that the plunger can move outward unobstructed by the bag. Heat the syringe/bag assembly in the microwave oven on the highest (500 W) setting for 7 - 10 seconds. Remove the bag and note the volume of gas produced. Additional time in the microwave may be necessary.

After the plunger has reached the desired mark (usually 50 mL), position the syringe with the syringe cap upward and carefully remove the syringe cap. Turn the syringe 180^{o} and discharge the excess liquid reagents into a plastic cup filled with water. Wash the gas in order to remove traces of aqueous chemicals from the inside surfaces of the syringe before the gases are used in experiments. To do this, draw 5 mL water into the syringe without discharging any gas, cap the syringe and shake the syringe to splash the rinse water on every inside surface. Remove the cap (cap end up!) and discharge the water but not any of the gas. Repeat if necessary.

Preparation of carbon monoxide in a microwave oven

The ease of generating carbon monoxide is substantially improved by the use of a microwave oven. In Chapter 20, we described a method in which 8 - 10 drops each of formic acid and concentrated sulfuric acid are heated with a small flame in a test tube connected to syringes in order to generate CO(g) according to the reaction:

$$H_2SO_4(l) + HCOOH(l) \rightarrow CO(g) + H_2SO_4 \cdot H_2O(l)$$

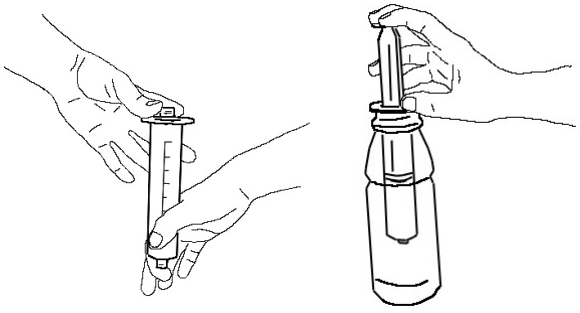
The same general approach is used when making carbon dioxide in a microwave oven. Premix 8 - 10 drops of each HCOOH and H_2SO_4(conc) in a small plastic weighing dish and draw the mixture into a syringe. Cap the syringe and place in a sealable plastic bag. Heat in the microwave oven for 3 - 5 seconds or until at least 50 mL CO(g) has been generated. Wash the gas as described for oxygen.

Preparation of sulfur dioxide in a microwave oven

As with oxygen, the generation of sulfur dioxide is especially slow by the traditional in-syringe method (Chapter 15). This is because SO_2(g) is highly soluble in water. In Chapter 15, we called for the use of 2-g sodium bisulfite (placed in an over-sized vial cap) and 5 mL 6 M HCl in order to generate 50 mL SO_2(g) according to the reaction:

$$NaHSO_3(s) + HCl(aq) \rightarrow SO_2(g) + NaCl(aq) + H_2O(l)$$

When using a microwave oven, use only 0.25 g $NaHSO_3$(s) and 3 mL 6 M HCl(aq). Lower the solid reagent into the syringe barrel by flotation (left figure) and install the plunger (right figure). Draw the HCl(aq) into the syringe, cap and mix the solid and liquid by shaking the syringe. Generation of SO_2(g) will be slow.



Place the syringe inside a sealable storage bag and seal the bag. The syringe must be positioned so that the plunger can move outward unobstructed by the bag. Heat the syringe/bag assembly in a microwave oven on the high setting for 5 - 7 seconds. Note the volume of gas produced. Additional time in the microwave may be necessary. Sulfur dioxide is exceedingly soluble in water so the plunger may start moving inward soon after removal from the microwave oven. To minimize this, remove the liquid reagents as soon as possible: With the syringe in the cap-up position, remove the syringe cap. Rotate the syringe and discharge the excess liquid reagents into a plastic cup filled with water.

Sulfur dioxide cannot be washed because it is water-soluble. Instead, transfer the gas sample to a clean, dry syringe by connecting the two syringes with a short length of tubing.

Preparation of methane in a microwave oven

Heating together anhydrous sodium acetate and sodium hydroxide produces methane (CH_4) according to the reaction:

$$NaOH(s) + NaC_2H_3O_2(s) \rightarrow CH_4(g) + Na_2CO_3(s)$$

The microwave method is similar except that a microwave oven is used instead of the Bunsen burner flame. Add 0.50 g NaOH(s) and 0.50 $NaC_2H_3O_2$(s) to a medium test tube. Fit the test tube with the one-hole stopper/glass connector tube. Connect the test tube assembly to a clean, lubricated syringe with a 15 cm length of tubing and set the entire assembly in a 250 mL glass beaker. Place the entire assembly in a 4 L sealable plastic bag. Heat in a microwave oven using the 400-watt setting for 2 - 3 minutes. Check the progress periodically. After 50 mL CH_4(g) have been collected, remove the bag from the microwave oven. Methane generated in this way is ready to use.

Preparation of hydrogen chloride in a microwave oven

Samples of gaseous hydrogen chloride (HCl) can be generated by heating 2-g anhydrous sodium hydrogen sulfate (sodium bisulfate), $NaHSO_4$ (or 2.3 g of the monohydrate, $NaHSO_4 \cdot H_2O$) and 1.0-g sodium chloride, NaCl together in a test tube. The reaction is:

$$NaHSO_4(s) + NaCl(s) \rightarrow HCl(g) + Na_2SO_4(s)$$

In Chapter 19, we called for heating the mixture with a Bunsen burner flame. Similar results can be obtained with a microwave oven. The general procedure described for methane is followed, except that a smaller test tube is used. It typically takes 2 - 3 minutes in a microwave oven running on the highest setting to generate 50 mL HCl(g). Check the progress of the reaction every 30 seconds.

Disposal

Unwanted gas samples can be safely discharged into the fume hood or out of doors. Oxygen samples can be discharged into the room.

Teaching tips

1. Every microwave is different. Times will vary.
2. Use the sealable plastic bag to contain chemicals that would otherwise be spilled if the plunger pops out of the barrel.

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.

SUMMARY OF MATERIALS AND CHEMICALS NEEDED FOR CHAPTER 24. MAKING GASES IN MICROWAVE OVENS

Equipment required

Item	For demo	For 5 pairs	For 10 pairs
Microscale Gas Chemistry Kit (See Chapter 1)	1	5	10
microwave oven	1	2	3
top-loading balance	1	2 - 3	3 – 5
pipet, glass Pasteur	2	10	20
250 mL beaker*	1	5	10
one-hole rubber stoppers**	1 + 1	5 + 5	10 + 10

* additional equipment necessary for generating CH_4(g) and HCl(g):

** to fit the small and medium test tubes; each stopper should be fitted with a 4 cm length of glass tubing (must be a air-tight fit — use alcohol to lubricate rubber stopper and glass before inserting glass into stopper)

Materials required

Item	For demo	For 5 pairs	For 10 pairs
gallon (4 L) sealable storage bags	1	5	10

Chemicals required

Item	For demo	For 5 pairs	For 10 pairs
conc NH_4OH (for NH_3)	1 mL	5 mL	10 mL
H_2O_2 (for O_2)	5 mL	25 mL	50 mL
KI (for O_2)	< 1	2 g	4 g
HCOOH (for CO)	1 mL	5 mL	10 mL
H_2SO_4 (for CO)	1 mL	5 mL	10 mL
$NaHSO_3$ (for SO_2)	0.25 g	1 g	2 g
6 M HCl (for SO_2)	3 mL	15 mL	30 mL
$NaC_2H_3O_2$ (for CH_4)	0.5 g	2.5 g	5 g
NaOH (for CH_4)	0.5 g	2.5 g	5 g
$NaHSO_4$(anhyd) (for HCl)	2 g	10 g	20 g
NaCl (for HCl)	1 g	5 g	10 g