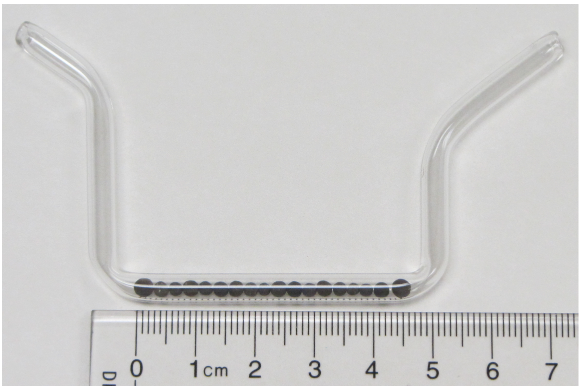
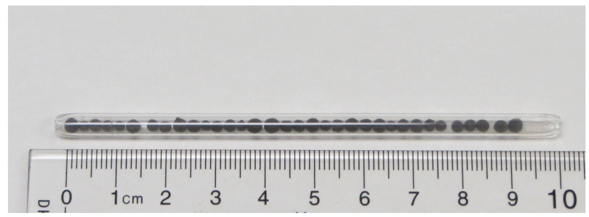
Gas Reaction Catalyst Tube

Bruce Mattson





Left: U-shaped catalyst used for sand bath high temperature work. Right: Straight design used for experiments conducted in flame.

Construction of the catalyst tube

A 25-cm length of borosilicate glass tubing, 6 mm OD, is fire-polished on one end until the opening becomes slightly constricted. A 90^{o} angle is formed in the tubing approximately 8 cm from one end with an oxygen/natural gas torch. If the bend slightly constricts the inside diameter, that can serve the useful purpose of preventing the Pd beads from escaping. At this point, the tubing should have the shape of the letter "L". Next place Pd-coated alumina beads into the tubing via the longer arm of the "L". A disposable wide-step plastic transfer pipet can be formed into a funnel by cutting off the stem so that it can be slipped over the "L"-shaped glass tubing. The top half of the bulb can be cut off to create the funnel. Wearing gloves, beads of 0.5% Pd on alumina are dropped, one-at-a-time, into the tube. In our experience, the larger beads do not fit in the tube. After the beads inside the glass tube extend a length of 4 – 8 cm, the second 90^{o} bend can be formed. Again, a slight constriction prevents the escape of the Pd beads. The second end of the U-shaped tube is fire-polished. Allow 30 minutes to construct the catalyst tube. Slight bends as shown serve to keep the syringes away from the heat rising from the sand bath. The catalyst tube can be used indefinitely. Prudent safety measures must be employed when open flames are used to bend glass.

Contents

PART ONE. GENERAL INFORMATION

About the Catalyst

The gas reaction catalyst tube consists of a coating of 0.5% palladium atoms dispersed over alumina support. The palladium present is of almost negligible mass yet is the material that actually catalyses the reactions described here.

A. Additional Equipment Required

In addition to the equipment provided in the kit, you will need the following equipment in order to perform the catalytic reaction:

• a Bunsen burner
• two ring stands with one three-prong clamp each (optional)

In addition, the various chemical tests of the gaseous products require equipment and chemicals that are not provided. These are described in the tests later in these instructions.

B. Setting Up the Apparatus

Two short pieces (approx. 2-cm) of Latex tubing connect the catalyst tube to the two syringes. The syringe on the left contains the reagent gas mixture ready to be passed through the catalyst. The plunger of the receiver syringe (right) must be able to move freely in the syringe barrel because it should move outward on its own as the plunger of the reactant syringe is pushed inward. Two ring stands and clamps hold the two syringes in the appropriate position above the burner's flame. The clamps should not hold the syringes tightly and must allow for free rotation of the syringes and catalyst tube for even heating. With some experience, we have found that it is easier to hold the syringes with one’s hands instead of using a ring stand.

C. Properly Heating the Catalyst Tube.

Heat from a Bunsen burner flame is capable of softening the glass portion of the catalyst tube. When the glass is soft, it is susceptible to deformations and even "blow holes" if the pressure inside the system is increased by moving the plunger of the syringe. To prevent overheating the glass, use only a cool Bunsen burner flame. Minimize the amount of air used so that the flame has a soft, ill-defined blue inner cone. Position the catalyst tube at least 1 cm above the tip of the inner cone. Watch for traces of red, orange or yellow in the flame above the catalyst tube. These colors indicate that the glass is softening. If this should happen, remove the flame and adjust the flame.

D. Explosion Risk! Please Read!

The oxidation reactions involving hydrocarbons (methane and ethene) described herein utilize air as a source of oxygen. Do NOT attempt these catalysis reactions using oxygen instead of air! An explosion will result!

E. Activating the catalyst

The ceramic catalyst will appear tan or brown until it is activated. "Activation" simply involves heating the catalyst tube in a cool flame until it turns dark, sometimes even black.

This takes less than a minute and can be done as part of the first experiment. Heat the catalyst tube evenly by rotating the syringes periodically in the flame.

F. Sources of Gases.

The gas catalysis experiments described here require samples of various gases. Compressed cylinders of gases are convenient and the purity is assumed to be quite good. Natural gas can be used as a source of methane. All of the reagent gases in this article can be prepared by simple methods we have described in our series in ***Chem13 News*** over the past several years. These gas preparations are also available in our book, ***Microscale Gas Chemistry,*** available from Educational Innovations and at our website (See Part Four). Gases prepared in this method contain small amounts of air.

G. Toxicity.

Manipulating gases in syringes is generally safe and unintentional discharges are not common. Nevertheless, such discharges are possible and it is important to read and understand the following information. Nitrogen dioxide has an irritating odor and is a poisonous gas. Concentrations of 100 ppm are dangerous. To put this in perspective, if the contents of one entire syringe of NO_2 (60 mL) were discharged into a volume of 1 m^3, the concentration of NO_2 would be 60 ppm. Ammonia has a pungent irritating odor and is highly poisonous. Although less toxic than ammonia and nitrogen dioxide, carbon monoxide is toxic but has no odor. Symptoms of carbon monoxide poisoning include headache, mental dullness, weakness, nausea and vomiting. Exercise caution when working with poisonous gases and vacate areas that are contaminated with unintentional discharges of gas.

H. Clean-up and Storage

After reactions, heat the catalyst for 30 seconds in the flame, remove the flame and purge the catalyst with a syringe filled with an inert gas such as nitrogen or argon. Air may be used if inert gases are not available. Allow the catalyst to cool. Store the gas reaction catalyst tube in a sealed plastic bag.

PART TWO. CATALYTIC REACTIONS

A. Oxidation of Methane with Air.

$$CH_4(g) + 2\ O_2(g) \rightarrow CO_2(g) + 2\ H_2O(g) \quad \Delta H = -803\ kJ$$

Fill the reagent syringe with 50 mL air (0.4 mmol O_2) and 10 mL methane (0.4 mmol.) Connect both the reagent and receiver syringes to the catalyst tube and assemble the apparatus as shown in the Figure. Pass about 10 mL of gas mixture through the catalyst tube to (a) check for leaks, (b) determine that the plunger in the receiver flask moves freely; and (c) displace air from the catalyst tube. (Option: Disconnect the receiver syringe from the catalyst tube, discharge the 10-mL contents from the receiver syringe and reconnect to the catalyst tube.) Heat the catalyst tube gently and evenly on all sides for a total of about 30 seconds.

With continued heating in a cooler flame (top of outer cone or less make-up air), slowly pass about half of the methane/air reagent gas mixture through the catalyst tube over the course of about 30 seconds. The volume of gases collected in the receiver syringe should approximately equal the volume decrease in the reagent syringe because the main component in the syringe is nitrogen from the air. After half of the gas mixture has been passed through the catalyst tube, remove the heat. Remove both syringes and cap them with Latex syringe caps. Label the syringes 'reactants' and 'products' with a marker pen.

You may wish to perform one or more of the following tests, described in Part Three, on the reagent gas mixture and product gas mixture:

(a) Limewater test for CO_2
(b) Flammability test
(c) Gas chromatography
(d) Water test

More information about this experiment along with color photographs can be found at our gas chemistry website (Part Four, below). This reaction has been described by Cooper and Wolf (Gilbert, G. L., Alyea, H. N., Dutton, D., and Dreisbach, D., *Tested Demonstrations in Chemistry and Selected Demonstrations from the Journal of Chemical Education*, Volume I; *Journal of Chemical Education* 1994, pp I-34 – 35, I-37.) and utilizes a Bunsen burner and a platinum wire.

B. Oxidation of Ethene with Air.

$$C_2H_4(g) + 3\ O_2(g) \rightarrow 2\ CO_2(g) + 2\ H_2O(g) \quad \Delta H = -1323\ kJ$$

Follow the general procedure described in Reaction 1 for methane. Use 50 mL <u>air</u> (0.43 mmol O_2) and 10 mL ethene (0.41 mmol.)

You may wish to perform one or more of the following tests, described in Part Three, on the reagent gas mixture and product gas mixture:

(a) Lime water test for CO_2
(b) Flammability test
(c) Gas chromatography
(d) Bromine-water test
(e) Water test

C. Oxidation of Carbon Monoxide.

$$2\ CO(g) + O_2(g) \rightarrow 2\ CO_2(g) \quad \Delta H = -566\ kJ$$

In this oxidation, either air or oxygen may be used as the oxidant. Follow the general procedure described above for methane. Use 45 mL air (0.38 mmol O_2) and 15 mL carbon monoxide (0.6 mmol) or 40 mL CO (1.6 mmol) and 20 mL O_2 (0.8 mmol).

You may wish to perform one or more of the following tests, described in Part Three, on the reagent gas mixture and product gas mixture:

(a) Lime water test for CO_2
(b) Gas chromatography
(c) If O_2 is used rather than air, the Flammability and Glowing Splint test may be performed.

D. Hydrogenation of Ethene

$$C_2H_4(g) + H_2(g) \rightarrow C_2H_6(g) \quad \Delta H = -137 \text{ kJ}$$

Fill the reagent syringe with 30 mL ethene (1.2 mmol) and 30 mL hydrogen (1.2 mmol.) Connect the reagent and receiver syringes to the catalyst tube as shown in the Figure. Pass about 10 mL of gas mixture through the catalyst tube to purge it of air. Remove the receiver, discharge the air and then reconnect as quickly as possible in order to minimize H_2-loss. Heat the catalyst tube evenly on all sides for about 30 seconds, then slowly pass about half of the C_2H_4/H_2 reagent gas mixture through the catalyst tube over the course of about 30 seconds. The volume of gases collected in the receiver syringe should be *less than* the volume decrease in the reagent syringe; 2 mol gaseous reactants become 1 mol of gaseous products if the reaction efficiency is 100%. In our experience, these experimental conditions cause hydrogenation with about 50% efficiency. Nearly complete hydrogenation can be achieved by activating the catalyst tube in a gentle flame with pure hydrogen (60 mL) prior to passing the mixture through the catalyst tube. After half of the gas mixture has been passed through the catalyst tube, remove the heat. Remove both syringes and cap them with Latex syringe caps. Label the syringes.

The Bromine-water test for C_2H_4 should confirm that there is less ethene in the product syringe than in the reactant syringe. Gas chromatography allows for a quantitative estimation of the extent of hydrogenation. See Part Three for details.

E. Catalytic Oxidation of Ammonia.

$$4\ NH_3(g) + 3\ O_2(g) \rightarrow 2\ N_2(g) + 6\ H_2O(g) \quad \Delta H = -1268 \text{ kJ}$$

This reaction has been the subject of numerous demonstrations involving glowing platinum or copper (See: (a) *A Demo A Day, A Year of Chemical Demonstrations*, Flinn Scientific 1995; pp 224; (b) Shakhashiri, B. Z.; *Chemical Demonstrations, A Handbook for Teachers of Chemistry*, Volume 2, University of Wisconsin Press, 1985; pp 214 – 215; (c) Gilbert, G. L., Alyea, H. N., Dutton, D., and Dreisbach, D., *Tested Demonstrations in Chemistry and Selected Demonstrations from the Journal of Chemical Education*, Volume I; *Journal of Chemical Education* 1994, pp I-34 - 35.). In each case, ammonia and air react at the surface of the metal that has been preheated to redness in a flame. The exothermic nature of the reaction sustains the red glow of the catalyst. In the reaction described here, the palladium catalyst operating at a lower temperature yields nitrogen rather than nitric oxide.

Fill the reagent syringe with 30 mL ammonia (1.2 mmol) and 30 mL oxygen (1.2 mmol.) In this proportion, NH_3(g) is the limiting reagent. Connect the reagent and receiver syringes to the catalyst tube as shown in the Figure. For this reaction, *do not* pass any of the gas mixture through the catalyst tube to displace air from the tube. Heat the catalyst tube evenly on all sides for a total of about 30 seconds. Slowly pass about half of the ammonia/oxygen reagent gas mixture through the catalyst tube over the course of about 30 seconds. A cloud or fog of condensing water vapor should be noticed in the receiver syringe. After half of the gas mixture has been passed through the catalyst tube, remove the heat. Remove both syringes and cap them with Latex syringe caps. Label the syringes with a marker pen.

The relative amount of ammonia in each syringe is determined as follows. Note the volume of gas in each syringe. Remove the syringe cap and place each syringe in a 250 mL beaker filled with water. Draw at least 20 mL water into each syringe; ammonia will quickly dissolve. After a minute, note the new volume of the gas in the syringe. The product syringe will contain little or no ammonia, so the volume of gas will be about the same as its original value. The reactant syringe had contained 50% ammonia so that the volume of gas remaining should be half of its original amount. One may add some universal indicator to the discharged water from each syringe in order to estimate the pH. The unreacted ammonia will increase the pH substantially, while the product syringe may remain neutral. If nitric oxide were produced as occurs with the reactions described in the literature *(see above),* it would immediately react with oxygen present to form red NO_2, an acid anhydride. Neither the red color of NO_2, nor the low pH that a solution of the gas would produce is observed.

F. Methane and Nitrogen Dioxide.

$$CH_4(g) + 2\ NO_2(g) \rightarrow N_2(g) + CO_2(g) + 2\ H_2O(g) \quad \Delta H = -869\ kJ$$

Fill the reagent syringe with 30 mL methane (1.2 mmol) and 30 mL nitrogen dioxide (1.2 mmol.) This proportion assures that NO_2 is the limiting reagent. The mixture is red-brown due to the nitrogen dioxide. Connect the reagent syringe to the catalyst tube and assemble the apparatus as shown in the Figure. Do not pass any of the gas mixture through the catalyst tube to displace air from the tube. Heat the catalyst tube evenly on all sides for a total of about 30 seconds. Slowly pass all of the CH_4/NO_2 reagent gas mixture through the catalyst tube over the course of about 30 seconds. The gases collected in the receiver syringe should not be red. Rather, a 'fog' of water vapor should be noted. It is possible that the red color will not completely disappear on the first pass. If that is so, simply reverse directions and pass the gas mixture back through the catalyst in the other direction.

In addition to detecting the reaction has taken place due to the disappearance of the red color, the product gases, described in Part Three, can be tested by the

(a) Limewater test
(b) Water test

More information about this experiment along with color photographs can be found at our gas chemistry website (See Part 4B.) This reaction is highly suited for a lecture demonstration because the red color of the reactants can be seen to disappear while a fog of water forms in the product syringe.

G. Carbon Monoxide and Nitrogen Dioxide.

$$4\ CO(g) + 2\ NO_2(g) \rightarrow N_2(g) + 4\ CO_2(g) \qquad \Delta H = -1198\ kJ$$

Fill the reagent syringe with 40 mL carbon monoxide (1.6 mmol) and 15 mL nitrogen dioxide (0.6 mmol.) This proportion assures that NO_2 is the limiting reagent. The mixture is red-brown due to the nitrogen dioxide. Connect the reagent syringe to the catalyst tube and assemble the apparatus as shown in the Figure. Do not purge the catalyst tube with the gas mixture before heating. Heat the catalyst tube evenly on all sides for a total of about 30 seconds. Slowly pass all of the CO/NO_2 reagent gas mixture through the catalyst tube over the course of about 30 seconds. The gases collected in the receiver syringe should not be red. Unlike Reaction 6, no 'fog' of water vapor will be noted. It is possible that the red color will not completely disappear on the first pass. If that is so, simply reverse directions and pass the gas mixture back through the catalyst in the other direction.

In addition to detecting the reaction has taken place due to the disappearance of the red color, the product gases can be tested by the Limewater test, described in Part Three.

This reaction will take place without the catalyst present if high temperatures are used; this can be demonstrated by performing the reaction with a control (empty tube). The reaction requires a catalyst at lower temperature.

H. Decomposition of Nitrous Oxide.

The thermal decomposition of nitrous oxide occurs above 300 ^{o}C. The reaction is:

$$2\ N_2O(g) \rightarrow 2\ N_2\ (g) + O_2\ (g) \qquad \Delta H = -164\ kJ$$

Fill the reagent syringe with 60 mL N_2O (2.4 mmol N_2O). Connect both the reagent and receiver syringes to the catalyst tube and assemble the apparatus as shown in the Figure. Pass about 10 mL of N_2O through the catalyst tube to displace the air present. Disconnect the receiver syringe from the catalyst tube, discharge the 10-mL gas from the receiver syringe and reconnect to the catalyst tube. Heat the catalyst tube evenly on all sides for a total of about 45 seconds. Slowly pass about half of the $N_2O(g)$ through the catalyst tube over the course of about 30 seconds. The catalyst may turn slightly tan due to oxidation caused by the oxygen produced by this reaction. After half of the gas mixture has been passed through the catalyst tube, remove the heat. Remove both syringes and cap them with Latex syringe caps. Label the syringes 'reactants' and 'products' with a marker pen.

Test the reagent gas mixture and product gas mixture by the following tests, described in Part Three:

(a) Glowing Splint test
(b) Gas Chromatography

I. Nitrous Oxide and Ammonia.

$$3\ N_2O(g) + 2\ NH_3(g) \rightarrow 3\ H_2O(g) + 4\ N_2(g) \quad \Delta H = -880\ kJ$$

Fill the reagent syringe with 15 mL ammonia (0.6 mmol) and 30 mL nitrous oxide (1.2 mmol.) In this proportion, $NH_3(g)$ is the limiting reagent. Connect the reagent and receiver syringes to the catalyst tube as shown in the Figure. Do not purge the catalyst tube with the reaction mixture. Heat the catalyst tube evenly on all sides for a total of about 30 seconds. Slowly pass about half of the NH_3/N_2O reagent gas mixture through the catalyst tube over the course of about 30 seconds. After half of the gas mixture has been passed through the catalyst tube, remove the heat. Remove both syringes and cap them with Latex syringe caps. Label the syringes with a marker pen.

Perform the tests, described in Part Three, on the on the reagent gas mixture and product gas mixture:

(a) Acidity test
(b) Ammonia test
(c) Water test

J. Nitrous Oxide and Carbon Monoxide

$$N_2O(g) + CO(g) \rightarrow CO_2(g) + N_2(g) \quad \Delta H = -365\ kJ$$

Fill the reagent syringe with 30 mL carbon monoxide (1.2 mmol) and 30 mL nitrous oxide (1.2 mmol.) Connect the reagent and receiver syringes to the catalyst tube as shown in the Figure. Pass about 10 mL of gas mixture through the catalyst tube to displace air from the tube. Remove the receiver syringe from the catalyst tube, discharge the contents and reconnect as before. Heat the catalyst tube evenly on all sides for a total of about 30 seconds. Slowly pass about half of the CO/N_2O reagent gas mixture through the catalyst tube over the course of about 30 seconds. Remove the heat. Remove both syringes and cap them with syringe caps. Label the syringes with a marker pen.

Perform the following tests, described in Part Three, on the reagent gas mixture and product gas mixture.

(a) Limewater
(b) Flammability
(c) Gas Chromatography

Do not perform the Glowing Splint test on unreacted $N_2O(g)/CO(g)$ mixture; this mixture of gases reacts explosively.

K. Nitrous Oxide and Methane

$$4\ N_2O(g) + CH_4(g) \rightarrow CO_2(g) + 4\ N_2(g) + 2\ H_2O(g)\ \Delta H = -1130\ kJ$$

Fill the reagent syringe with 40 mL N_2O (1.6 mmol) and 10 mL methane (0.41 mmol.) Cap the syringe and allow the gases to mix for several minutes. Connect the reagent syringe to the catalyst tube and assemble the apparatus as shown in the Figure. Pass about 10 mL of gas mixture through the catalyst tube. (Option: Remove the receiver syringe from the catalyst tube, discharge the 10-mL air from the receiver syringe and reconnect to the catalyst tube.) With a Bunsen burner on low heat (no sharp inner cone), heat the catalyst tube evenly on all sides for a total of about 30 seconds. Remove the heat; it is NOT necessary to continue to heat the catalyst. Slowly pass about half of the CH_4/N_2O reagent gas mixture through the catalyst tube over the course of about 30 seconds. The catalyst inside the tube may become red hot, in which case slow down the flow of gas. Small droplets of water may form on the glass near the receiver syringe. A cloud of condensing water vapor may also be noted in the receiver syringe. After half of the gas mixture has been passed through the catalyst tube, remove the heat. Remove both syringes and cap them with latex syringe caps. Label the syringes with a marker pen.

One or more of the following tests, described in Part Three, may be performed on the reagent gas mixture and product gas mixture:

(a) Limewater test for CO_2
(b) Flammability test:
(c) Gas chromatography.
(d) Water test

L. Trying Other Catalytic Reactions.

Use caution when attempting other reactions with the catalyst tube. Explosive mixtures, even on the millimole scale are dangerous. When trying reactions for the first time, dilute the gas mixture with an inert gas such as argon or nitrogen. For example, NO_2 and H_2 react explosively unless diluted. The catalyst glows red and then the explosion occurs. In our case, the plunger shot out of the syringe, but the glass catalyst tube could have just as easily exploded. When new reactions are being explored, they should be done so with considerable dilution (perhaps 90% argon and 10% reagents) until the nature of the reaction has been worked out. Never use pure oxygen as an oxidant unless you have determined it is safe to do so. This is done by a series of experiments in which the amount of O_2 is incrementally increased. We used this approach in working with Reactions C and E. Generally air can be used as ‘diluted oxygen;’ it is approximately 21% O_2 and the rest is inert N_2 and Ar.

PART THREE. CONFIRMATORY TESTS

A. Acidity test.

Prepare a universal indicator by dissolving 5 mL universal indicator in 50 mL distilled water. The concentration must be fairly high so that the colors are readily seen. Equip the syringe with a 15 cm length of latex tubing. Bubble 10 - 20 mL of the gas through the indicator solution. Remove the syringe and tubing. Notice color changes.

B. Ammonia tests.

Ammonia can be detected by odor. Discharge 3-mL of the gas about 1-ft (30 cm) in front of your face. With a cupped hand, waft the gas towards your nose. Ammonia can also be detected by the Cu^{+2} test. Place 5 mL 0.10 M $CuSO_4$ in a 15 x 180 mm test tube. Equip the syringe with a 15 cm length of latex tubing. Bubble 10 - 20 mL of the gas through the Cu^{+2} solution. Remove the syringe and tubing. Stopper the solution and shake to mix gaseous layer with Cu^{+2} solution. A deep blue solution indicates the presence of NH_3 as a result of the reaction:

$$Cu^{+2}(aq) + 4\ NH_3(g) \rightarrow [Cu(NH_3)_4]^{+2}(aq)$$

C. Bromine-water test.

Place 5 mL dilute bromine water (yellow, not orange) into a 15 x 180 mm test tube. Equip the syringe with a 15 cm length of latex tubing. Bubble 10 - 20 mL of the gas through the bromine water solution. Remove the syringe and tubing. Stopper the solution and shake to mix gaseous layer with bromine water solution. If alkenes are present, such as ethene, the yellow solution will turn colorless. The reaction is:

$$C_2H_4(g) + Br_2(aq) \rightarrow CH_2OHCH_2Br(l)$$

Other gases, including ethane, CO_2, and H_2 do not react with bromine water, so the solution will not discolor. (Bromine water is prepared from chlorine bleach and potassium bromide or sodium bromide. For detailed instructions, see our gas chemistry website (Part Four, below).)

D. Flammability test.

Fill a small weighing boat with 3% dish soap solution. Equip the gas syringe with the 15-cm length of latex tubing. Discharge 10-mL gas into the soap solution in order to produce a mound of several large bubbles. Try to ignite the bubbles with a match. If the bubbles contain hydrocarbons, they may burn or pop rather than simply break. (Dish soap solution, 3%, is prepared by dissolving 3 g dish soap per 100 g water.)

E. Gas chromatography.

We use gas chromatography to separate and detect syringe gases. Our choice of column is a Porapak N 80/100, 6-ft (180 cm), inside diameter = 0.085 inches (2.2 mm), available from Alltech Part Number 2716; telephone: 847-948-8600. We use a thermoconductivity detector and run the GC at room temperature. Carrier gas is helium, 30-mL/minute.

F. Glowing Splint.

A traditional test for oxygen is the glowing splint test. Only one other common gas, N_2O is capable of re-igniting a glowing splint. Connect the syringe to a glass pipet via a short length of latex tubing. Discharge 10 – 15 mL of the gas directly from the syringe onto the glowing splint. The discharge should be quick and as close to the glowing splint as possible. Pure O_2 and N_2O will re-ignite the splint into an open flame. Mixtures of these gases with other gases may prevent the splint from being re-ignited, however the splint will glow brightly while the gas is being discharged. In most cases, the splint will re-ignite, however.

G. Limewater test for CO_2

Place 3 - 4 mL lime water in a 15 x 180 mm test tube. Equip the syringe with a 13 cm length of latex tubing. Discharge 10 - 20 mL of the gas above the surface of the limewater solution. Remove the syringe and tubing. Stopper the solution and shake to mix gaseous layer with limewater solution. A cloudy solution indicates the presence of CO_2 as a result of the reaction:

$$Ca(OH)_2(aq) + CO_2(g) \rightarrow CaCO_3(s) + H_2O(l)$$

(Limewater is a clear colorless saturated $Ca(OH)_2(aq)$ prepared by mixing 1.5 g $Ca(OH)_2(s)$ per liter of water. Stir or shake vigorously and allow the solid to settle overnight. When using limewater, decant carefully to avoid transferring any solid or suspended $Ca(OH)_2(s)$.)

H. Water Test.

When water is formed, the product syringe often appears 'cloudy' from the aerosol of water. After a few minutes, the aerosol condenses into minuscule drops of water lining the inside of the syringe. By pushing the plunger inward by 5 - 10 mL and then retracting it back outward by the same amount, the water droplets are pushed along ahead of the plunger. This greatly assists in seeing the droplets. As chemical confirmation, remove the plunger just long enough to add a piece of blue-colored Dririte ($CoCl_2$ on an anhydrous $CaCl_2$ granule) to the syringe. Return the plunger or stopper the syringe barrel. The presence of water is confirmed if the blue granule turns pink-purple within a few minutes.

PART FOUR. SUPPORT

A. Our Gas Chemistry Website

We have published articles in ***Chem 13 News*** describing the microscale preparation and experimentation for a wide variety of gases. All of these articles are available on our gas chemistry website or in our book on microscale gas chemistry experiments which is at:

mattson.creighton.edu/Microscale_Gas_Chemistry.html

This work with the catalyst tube refers to four different chapters in this website or in our book on microscale gas chemistry experiments . Interested readers are encouraged to learn more about microscale gas chemistry by visiting this site. In all, over 140 experiments are described along with many spectacular photographs of the reactions.

B. Microscale Gas Chemistry

Own your own copy of all of the microscale gas chemistry experiments published in our ***Chem 13 News*** series. The book includes a detailed index, ordering information and information about each gas. Spiral bound, 270+ pages. Sold exclusively by Educational Innovations, Item #BK-590.

C. Ordering syringes, parts, etc. from Educational Innovations

- gas reaction catalyst tube only (Item # GAS-120)
- syringes, 60-mL plastic syringe with a LuerLOK fitting (Item # GAS-140)
- syringe cap fittings, latex LuerLOK (Item # GAS-160)
- plastic vial caps (Item # GAS-180)
- latex tubing, 1/8-inch (3.175 mm) ID, 5 ft (1.75 m) lengths (Item # GAS-220)
- metal locking hemostat (Item # GAS-200)

Safety Guidelines

Excerpted from the Division of Chemical Education's (American Chemical Society) Safety Guidelines for Chemistry Demonstrators

1. ***Wear appropriate eye protection*** for all chemical demonstrations.

2. Know the properties of the chemicals and the chemical reactions involved in all demonstrations presented.

3. Comply with all local rules and regulations.

4. Arrange to have a **fire extinguisher** at hand whenever the slightest possibility for fire exists.

5. Plan the demonstration so that harmful quantities of noxious gases (NO_2, SO_2, H_2S, etc.) do not enter the local air supply.

6. Provide safety shield protection wherever there is the slightest possibility that a container, its fragments or its contents could be propelled with sufficient force to cause personal injury.

7. Warn the members of the audience to cover their ears whenever a loud noise is anticipated.