

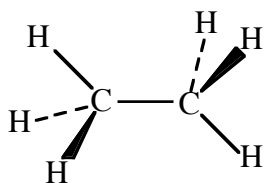
CHAPTER

14

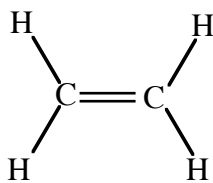
ETHYNE (ACETYLENE)

ETHYNE IS COMMONLY REFERRED TO AS ACETYLENE, the gas used by welders in oxy-acetylene torches. Ethyne is a colorless, highly flammable gas with a peculiar but characteristic odor. The odor of pure ethyne is not unpleasant, however, traces of phosphine (PH_3) are frequently present giving ethyne a disagreeable odor. The phosphines are produced from trace levels of calcium phosphide, Ca_3P_2 , present in calcium carbide, the compound used to generate ethyne. Pure ethyne (100%) does not burn. When mixed with air, even as little as 3%, ethyne forms explosive mixtures with air over a wide range of compositions.

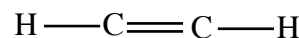
Ethyne is a linear molecule with a carbon-carbon triple bond. Interesting comparisons are noted between ethyne and the other two-carbon hydrocarbons, ethane (C_2H_6) and ethene (C_2H_4).



ethane



ethene

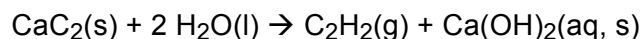


ethyne

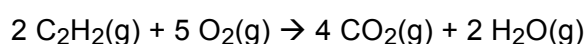
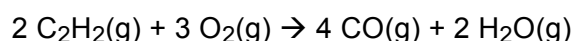
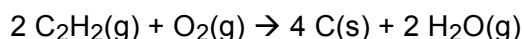
The following table summarizes the differences in bond length and strength between these two-carbon hydrocarbons. The carbon-carbon bond length becomes progressively shorter through the series ethane, ethene and ethyne. The carbon-carbon bond strength also becomes stronger through the series with the triple bond having a strength of 839 kJ/mol.

	C-C Bond	C-C Bond Length (pm)	C-C Bond (kJ/mol)
Ethane, C_2H_6	single	153	347
Ethene, C_2H_4	double	134	614
Ethyne, C_2H_2	triple	120	839

Ethyne occurs in small amounts in nature. It is found in swamp gases, and in coal and petroleum deposits. John Davy first prepared ethyne in 1836. Today it is manufactured by the reaction of calcium carbide with water, the same reaction that we will employ in this chapter. The reaction is:



Ethyne burns in air with the production of considerable amounts of soot. The reaction between ethyne and air is complex and depends on the amount of oxygen available. All three of these reactions take place:



When oxygen is limited, soot, C(s), is produced as given in the first reaction. Oxy-acetylene torches use pure oxygen. Under these conditions, an extremely hot and brilliantly white flame is produced and the products are almost exclusively CO₂ and H₂O as per the last equation.

Ethyne has a melting point and boiling point of -80.8 °C -84.0 °C, respectively, at 1 atm. The density of ethyne is 1.064 g/L at 25 °C which is 10% less than that of air. Ethyne is slightly soluble in water; 1 mL water will dissolve 1 mL ethyne. The gas also dissolves in a wide variety of organic solvents. For example, 1 volume of acetone (propanone) dissolves 25 volumes of ethyne!

Suitability

All of these experiments are suited for use as classroom demonstrations or laboratory activities for high school and university-level students. Experiment 3 is recommended as a demonstration because it requires mature and responsible behavior on the part of the students. The rocket experiment is the most fun for all, but is also the one that requires the most skill. The following experiments are included in this chapter.

- Experiment 1. Ethyne reacts with permanganate
- Experiment 2. Sooty combustibility of ethyne
- Experiment 3. Banging bubbles!
- Experiment 4. Ethyne/oxygen rockets
- Experiment 5. Ethyne reacts with aqueous bromine

Generally, the production of ethyne and the experiments that go with this gas should be conducted by individuals familiar with and experienced with gas production

using the syringe method introduced in Part 1. Ethyne forms explosive mixtures with air and due care must always be exercised.

All of these experiments serve to review basic concepts of chemistry including chemical changes/properties, writing and working with chemical formulas. In addition to these review topics, ethyne is a familiar and important organic compound. Experiments 1 and 5 demonstrate the chemical reactivity of the carbon-carbon triple bond. Experiment 1 is easy to set up and perform; Experiment 5 requires the preparation and use of aqueous bromine. Experiments 2, 3 and 4 all deal with the combustion of ethyne. Experiment 2 shows how ethyne undergoes incomplete combustion when an inadequate supply of air is present. In Experiments 3 and 4, ethyne is mixed with oxygen and loud and spectacular results are obtained.

Background skills required

Students should be able to:

- ❖ generate a gas as learned in Chapter 1.
- ❖ understand fundamental concepts of high school chemistry so that observations can be interpreted in intelligible ways.
- ❖ work responsibly with fire and flames.
- ❖ work responsibly with explosive mixtures of gases.
- ❖ know how to fill a test tube by the method of water displacement.
- ❖ fill and launch pipet mini-rockets as was done in the Chapter 4.

Time required

Most, if not all, of these experiments can be accomplished in one laboratory period, providing the students are prepared in advance for the experiments. Out-of-laboratory time will be needed to answer the questions. Splitting the experiments between classroom demonstration and laboratory experiment is a possibility.

Website

This chapter is available on the web at website:

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 ETHYNE¹

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

Ethyne is relatively non-toxic; however, it is a simple asphyxiant if inhaled in very large quantities.

Equipment

Microscale Gas Chemistry Kit (Chapter 1)

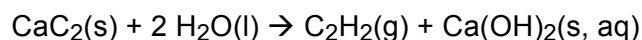
Chemicals

0.20-g calcium carbide, $\text{CaC}_2(\text{s})$ Note: Calcium carbide has a short shelf-life. See teaching Tip #1.

5 mL water

Generating ethyne gas samples

Approximately 60 mL of $\text{C}_2\text{H}_2(\text{g})$ will be prepared by the following instructions. The production of $\text{C}_2\text{H}_2(\text{g})$ is relatively fast and it typically takes 15 seconds to fill a syringe. The reaction is:



The C_2H_2 gas samples used in these experiments are generated by the method described in Chapter 1 and summarized below. Care must be taken to stop the gas generation after the syringe is full.

Step-by-step instructions for generating ethyne

1. Wear your safety glasses!
2. Make sure the syringe plunger and barrel are a good combination for each other and that the plunger moves with reasonable ease in the barrel without binding or sticking.
3. Measure out 0.20-g calcium carbide, $\text{CaC}_2(\text{s})$ directly into the vial cap.

¹ Content for this chapter first appeared as "Microscale Gas Chemistry, Part 7. Experiments with Hydrocarbons" Mattson, B. M.; Anderson, M.; Nguyen, J; Harrison, B., *Chem13 News*, **257**, April, 1997.

4. Fill the syringe barrel with water. Place your finger over the hole to form a seal.
5. Float the vial cap containing the solid reagent on the water surface.
6. Lower the cap by flotation. Release the seal made by finger to lower the cap into the syringe barrel without spilling its contents.
7. Install the plunger while maintaining the syringe in a vertical position, supported by the wide-mouth beverage bottle or flask.
8. Fill the weighing dish with water. Draw 5 mL water into the syringe.
9. Push the syringe fitting into the syringe cap.
10. Shake the device up and down in order to mix the reagents. Gently help the plunger move up the barrel.
11. Remove the syringe cap with the syringe held “cap-up”. Assume contents are under positive pressure.
12. Discharge the liquid reagent into the plastic cup. Immediately cap the syringe to prevent loss of gas.
13. Wash away contaminants. Ethyne-filled syringes must be “washed” in order to remove traces of unwanted chemicals from the inside surfaces of the syringe before the gases can be used in experiments. Remove the syringe cap and draw 5 mL water into the syringe. Cap the syringe and shake to wash inside surfaces. Remove cap and discharge water only (no gas), and recap the syringe. Repeat these washing steps if necessary.

Disposal of ethyne samples

Unwanted $C_2H_2(g)$ samples can be safely discharged outdoors or in a fume hood.

Teaching tips

1. Calcium carbide has a short shelf-life. It should normally look like dark chunks. With exposure to air from opening and closing the bottle, it turns into a tan powder that is not calcium carbide. Using the tan powder will not generate ethyne. To extend the shelf-life of calcium carbide, store the bottle under an inert atmosphere and in a desiccator if possible. A simple desiccator can be made from a wide-mouth jar (such as a jelly/jam/preserves jar with some anhydrous magnesium or calcium sulfate or calcium chloride as desiccant).
2. Practice generating ethyne before showing your students; it is a rapid reaction.
3. Remind students of the importance of the pressure-release procedure-Cap Up and OFF!
4. Ethyne is a highly flammable gas.

5. Ethyne forms explosive mixtures with air! When mixed with air, even as little as 3%, ethyne forms explosive mixtures and forms explosive mixtures with air over a wide range of compositions.

Questions

1. Write the balanced equation for the reaction that occurred in the syringe.
2. Determine the quantity of calcium carbide (in moles) that you used to prepare your $C_2H_2(g)$.
3. Determine the quantity of water used (in moles) in the experiment. Assume a density of 1.0 g/mL.
4. Which reactant was the limiting reactant, calcium carbide or water?
5. Use the limiting reagent to determine the theoretical yield of $C_2H_2(g)$.
6. What volume (mL) of $C_2H_2(g)$ is theoretically expected?

ETHYNE EXPERIMENTS

EXPERIMENT 1. ETHYNE REACTS WITH PERMANGANATE

Equipment

Microscale Gas Chemistry Kit

Chemicals

$C_2H_2(g)$, 60 mL

potassium permanganate, dilute aqueous solution (one small crystal per 50 mL water)

Suitability

high school lab, university lab, and classroom demonstration

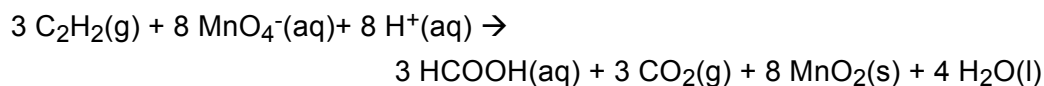
Applications, Topics, Purpose

chemical properties of ethyne, oxidation/reduction, organic chemistry, writing balanced chemical equations, chemical reactivity of ethyne

Instructions

Pour about 3 mL of the $KMnO_4$ solution into a small test tube and then discharge 10 mL $C_2H_2(g)$ into the syringe. Cap the test tube and shake the mixture vigorously. The pink color of permanganate will turn brown due to $MnO_2(s)$.

Ethyne and permanganate react with cleavage of the carbon-carbon triple bond and formation of the formate ion and the carbonate ion. The stoichiometry for the oxidation by permanganate under neutral or slightly acidic conditions is:



As a control the reaction between methane or propane and $\text{MnO}_4^-(\text{aq})$ can be tested. Unlike alkenes and alkynes, alkanes do not react with $\text{MnO}_4^-(\text{aq})$.

Teaching tips

1. The above reaction shows carbon dioxide formed. If the reaction were done inside the syringe by drawing dark purple potassium permanganate solution into the syringe and gently mixing gas with solution, tiny bubbles would be noticed. Unfortunately, this procedure stains the syringe brown.
2. This experiment illustrates the difference in chemical reactivity of alkynes (and alkenes) and alkanes. The alkenes and alkynes are much more chemically reactive than alkanes. The double and triple bonds are the reactive sites in these compounds.

Questions

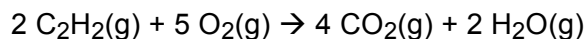
1. Draw the Lewis structure of ethyne.
2. Was the permanganate ion oxidized or reduced as it was converted into MnO_2 ?
3. From your answer to Questions 2, was the ethyne molecule oxidized or reduced?
4. Sometimes MnO_2 (a brown precipitate) is not produced; instead, a colorless solution of Mn^{+2} is observed. Did your reaction produce MnO_2 or Mn^{+2} ?
5. The carbon-carbon triple bond in ethyne is fairly reactive. Strong bonds tend to be less reactive, so we might suspect that the triple bond in ethyne is not especially strong. Carbon-carbon single, double, and triples bonds have bond strengths of 347, 614 and 839 kJ/mol, respectively. The double bond is *not* twice as strong as a single bond and can be thought of as increasing the overall bond strength by only 267 kJ/mol ($347 + 267 = 614$). In a similar way, how does a triple bond compare to a double bond?



EXPERIMENTS 2 - 5. ETHYNE/OXYGEN STOICHIOMETRY

Caution! Never mix ethyne and oxygen in the same syringe. The mixture is known to spontaneously explode.

The law of combining volumes states that the theoretically ideal ratio of ethyne and oxygen for use in Experiments 2 - 5 is two parts C₂H₂(g) for every five parts O₂(g).



EXPERIMENT 2. SOOTY COMBUSTION OF ETHYNE

Equipment

Microscale Gas Chemistry Kit
birthday candle affixed to a coin
match or lighter

Chemicals

C₂H₂(g), 60 mL

Suitability

high school lab, university lab, and classroom demonstration

Applications, Topics, Purposes

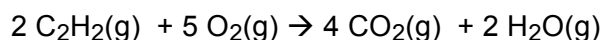
chemical properties of ethyne, combustion reactions, incomplete combustion, organic chemistry, balancing chemical equations, energy and chemical change (thermochemistry)

Instructions

Fill a medium sized test tube with water. Invert the test tube into a beaker of water. Prepare a syringe full of C₂H₂(g). Equip the syringe with a 15 cm piece of tubing. Use water displacement to fill the test tube with ethyne. Completely fill the test tube with C₂H₂(g). Remove the test tube from the beaker of water and immediately ignite with a lit candle. Soot will be produced as the C₂H₂(g) burns. Soot is made up of elemental carbon in the graphite form.

Teaching tips

1. When there is sufficient oxygen for the combustion reaction, ethyne burns cleanly and soot does not appear. The clean combustion reaction is given below:



2. This is the same reaction used by oxy-acetylene (ethyne) torches for welding. When the welder initially lights the torch, the air provides insufficient oxygen for complete combustion and black soot forms. When the welder opens the oxygen valve to mix oxygen and ethyne (acetylene), complete combustion occurs and a very hot blue flame is obtained.

3. The soot likely contains some buckeyball carbon, C₆₀(s).

Questions

1. Did you observe condensed water after igniting the ethyne in the test tube?
2. Soot is made up of elemental carbon in the graphite form. Did you see any soot formed? Balance the reaction in which ethyne, burning in air, produced soot and water vapor.
3. Was this combustion reaction complete or incomplete? Explain why.
4. What products are expected with complete combustion? Would you expect to see soot, C(s), with the complete combustion of ethyne?

EXPERIMENT 3. BANGING BUBBLES!

Equipment

Microscale Gas Chemistry Kit
match or lighter

Chemicals

C₂H₂(g), 60 mL
O₂(g), 60 mL (See Chapter 4 for in-syringe preparation, or make oxygen in a gas bag — See Chapter 5)
3% dish soap solution (3 g dish soap + 97 g H₂O)

Suitability

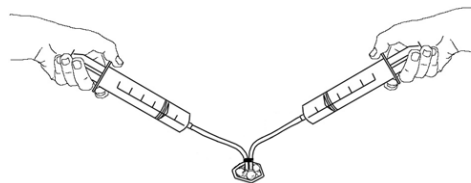
classroom demonstration

Applications, Topics, Purpose

explosive mixtures, combustion reactions, organic chemistry, balancing chemical equations, energy and chemical change (thermochemistry), chemical reactivity of ethyne, rates of chemical reactions (chemical kinetics)

Instructions

Fill a syringe with ethyne and a second syringe with oxygen. Tape the ends of two 15 cm lengths of tubing together as shown in the figure. Connect one of the pieces of tubing to the ethyne-filled syringe and the other to the oxygen-filled syringe. Simultaneously bubble various quantities of O₂(g) and C₂H₂(g) through a 3% dish soap solution forming a mound of bubbles. Remove the tubing from the bubble solution and set it aside. Ignite the bubbles with a piezoelectric lighter, candle or a match. The pop and flame produced will depend on the mixture of C₂H₂ and O₂. Pure ethyne will also produce soot. **Caution!** *The combustion of C₂H₂/O₂ mixtures can be extremely loud. Warn viewers that ear protection may be appropriate.*



Teaching tips

1. Do **NOT** mix ethyne and oxygen in the same syringe. The gases form an explosive mixture and unexpected spontaneous detonation has been known to occur.
2. This reaction can be surprisingly loud. For that reason, it is recommended that it be done as a classroom demonstration. Whether it is done as a demonstration or laboratory activity, warn everyone in the room before detonating the gas mixture.

Questions

1. What mixture of ethyne and oxygen made the loudest “bang”? Did this correspond to the stoichiometric ideal mixture?
2. Although these are explosions, why are they not particularly dangerous (except loud)? Hint: There are at least two reasons.
3. Why is this explosive mixture unstable, i.e. why is it dangerous to store this gas mixture even for a brief period of time before using?

EXPERIMENT 4. ETHYNE/OXYGEN ROCKETS

Equipment

Microscale Gas Chemistry Kit
pipet rockets, See Appendix C
piezoelectric sparker, See Appendix C
several small test tubes

Chemicals

$C_2H_2(g)$, 60 mL
 $O_2(g)$, 60 mL (See Chapter 4 for in-syringe preparation, or make oxygen in a gas bag — See Chapter 5)
limewater

Suitability

high school lab, university lab, and classroom demonstration

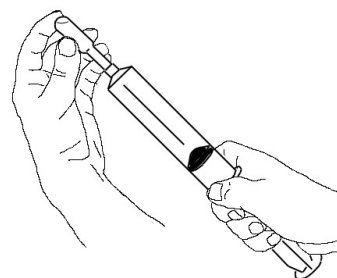
Applications, Topics, Purposes

combustion reactions, kinetics, stoichiometry of reactions, activation energy, explosive mixtures, rocketry, types of chemical reactions, properties of ethyne, energy and chemical change, chemical formulas

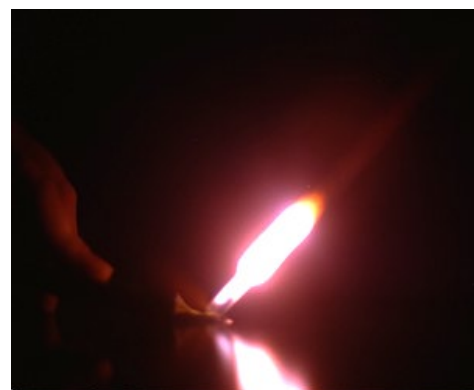
Instructions

Previously, while studying oxygen, you filled and launched rockets using the method you will use here. You may want to review this procedure, which is provided on in Chapter 4, Experiment 4, and again in Appendix C. (A step-by-step color photo sequence on filling and launching rockets is available at our website.) Fill the pipet rockets completely with water and store them open-end down in test tubes filled with water. (Stand the test tubes somewhat upright by placing them in a plastic cup.)

Remove the cap from the O_2 -filled syringe and slip the water-filled pipet rocket over the syringe fitting as shown in the figure. Bubble the $O_2(g)$ into the pipet rocket until it is about half full. Holding the syringe and bulb at a 45° angle works best. Next, complete the water-displacement with $C_2H_2(g)$ until the rocket is nearly filled. Leave some water in the pipet stem. If the water dribbles out, which is fairly common, draw up some more from a cup or beaker.



Position the rocket over the wire of the igniter. Water must remain in the stem because without it, the rocket will not fly very far. If the water dribbles out, draw up some more from a cup or beaker. The ends of the wire leads must be above the water in the gas-filled region of the rocket. Trigger the igniter and the rocket will fly 5 m or more. We have found that ethyne works particularly well and that a small fireball is noticed as the gas mixture ignites.



Repeat the reaction in the dark. Test the contents of the rocket for carbon dioxide with a few drops of limewater.

Teaching tips

1. A step-by-step color photo sequence on filling and launching rockets is available at our website.
2. Try the reaction with the lights out for added effect.
3. IMPORTANT COURTESY: Have students inform everyone in the room before they launch their rockets.
4. Award prizes for the rockets traveling the greatest distances.
5. This experiment illustrates that hydrocarbon and oxygen combustion reactions liberate energy and can perform useful work. The idea of stoichiometric ratios of C_2H_2 to O_2 can also be demonstrated.

Questions

1. How far, in meters, did your rocket fly?
2. What was the reaction that occurred inside the rocket?
3. Why must some water remain in the stem of the rocket?
4. What are the differences between ethyne as a rocket fuel and methane or propane as a fuel? What might account for the differences?
5. Did the contents of the rocket pass the limewater test for carbon dioxide?

EXPERIMENT 5. ETHYNE REACTS WITH AQUEOUS BROMINE

Equipment

Microscale Gas Chemistry Kit
suitable stopper for the 18 x 150 mm test tube (in the gas kit)

Chemicals

C₂H₂(g), 60 mL
aqueous bromine solution (See Appendix D)
sodium bisulfite solution, NaHSO₃(aq), 1 M

Suitability

high school lab, university lab, and classroom demonstration

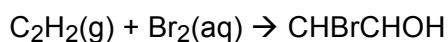
Applications, Topics, Purpose

chemical changes, properties of ethyne, chemical formulas, chemical reactions, chemical bonding, molecular structure, oxidation-reduction reactions, organic chemistry

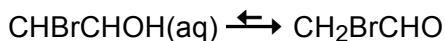
Instructions

Pour 2 - 3 mL of the bromine-water solution into the test tube and stopper. Equip the C₂H₂-filled syringe with the 15 cm length of tubing and transfer about 10 mL of the gas into the test tube, discharging the gas just above the surface of the bromine solution. Stopper and shake to mix gas and liquid. As a control, the reaction between methane (or propane) and Br₂(aq) can be tested. The red color of bromine will disappear in the ethyne-filled test tube but not in the alkane-filled test tube.

Ethyne and aqueous bromine react to form first 2-bromoethenol as follows:



The CHBrCHOH is an alkene-alcohol which prefers the rearranged aldehyde version, however the two probably exist in an equilibrium:



The α -bromoaldehyde is unstable and likely reacts with something. The ultimate products are uncertain. This reaction is quite specific to alkenes and alkynes and is used as a test method to confirm the presence of an unsaturated hydrocarbon.

Excess aqueous bromine solution can be treated by adding 1 M $\text{NaHSO}_3(\text{aq})$ a drop at a time until the color disappears and then safely discarded down the drain.

Teaching tips

1. Shake the test tube vigorously. Use lab film or plastic if stoppers are not available.
2. Destroy all excess bromine water solutions as per the Instructions (last paragraph)

Questions

1. Sketch the structure of CHBrCHOH . What type of carbon-carbon bond is present? Single, double or triple?
2. Sketch the structure of CH_2BrCHO .
3. Both ethyne and bromine are reactive molecules (molecules with high-energy). They quickly react to form the compound CHBrCHOH , which exists in equilibrium with CH_2BrCHO . This compound was described as unstable and soon reacts to form unspecified products that we can assume to be final products and stable. Sketch an energy profile for the reaction that starts with ethyne and bromine and shows all of the steps described. Carefully sketch in reasonable activation energies and changes in energy.
4. Suppose that you had two unmarked gas filled syringes. One contained ethene and the other contained ethane. Propose a simple experiment to determine what gas was in each syringe.

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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 14. EXPERIMENTS WITH ETHYNE

Equipment required

Item	For Demo	For 5 pairs	For 10 pairs
Microscale Gas Chemistry Kit (See Chapter 1)	1	5	10
birthday candle (from kit)	1	2 - 3	4 - 5
match or lighter	1	5	10
large bulb pipets*	1	5	10
piezoelectric sparker	1	5	10
small test tubes	3	15	30
stopper (to fit 18 x 150 mm test tube)	1	5	10

* See Appendix C

Chemicals required

Item	For Demo	For 5 pairs	For 10 pairs
calcium carbide, CaC_2	1 g	5 g	10 g
potassium permanganate	< 0.1 g	< 0.1 g	< 0.1 g
3% v/v dish soap solution*	1 drop	1 mL	2 mL
potassium iodide, KI, powder	1 g	5 g	10 g
6% $\text{H}_2\text{O}_2(\text{aq})^{**}$	20 mL	100 mL	200 mL
sodium bromide, NaBr	1.1 g	a	a
hydrochloric acid, 1 M HCl	11 mL	a	a
sodium hypochlorite ***	8 mL	a	a
sodium bisulfite, $\text{NaHSO}_3(\text{aq})$, 1 M	5 mL	25 mL	50 mL
limewater	2 mL	10 mL	20 mL

* (3 g dish soap + 97 g H_2O)

** 3% $\text{H}_2\text{O}_2(\text{aq})$ will also work, but not quite as well

*** household laundry bleach

a See Appendix D; recipe for "Demo" makes enough for entire group