



Faculty of Science, Engineering and Built Environment

School of Life and Environmental Sciences

SLE155 Chemistry for the Professional Sciences Laboratory Manual 2023

The **Laboratory Manual** for SLE155 Chemistry for the Professional Sciences was prepared from many sources and has been collated and edited by Angela Ziebell, Jacqui Adcock, Madeleine Schultz, Damien Callahan and Kim Quayle.

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Updated for Deakin College by Deepa Ram.

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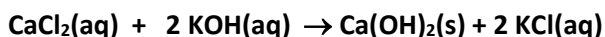
Experiment 1: Stoichiometry, limiting reagents and percentage yield

Introduction

In this experiment, you will perform a synthesis reaction involving two reagents. Using the balanced equation, you will be able to calculate which reagent is limiting and which is excess in your situation. You will use this information to calculate the percentage yield of the product that you obtained, based on your own limiting reagent. Your product, calcium hydroxide (sometimes called calcium dihydroxide) is used in a vast array of industries such as food processing (pickling food, clarifying juice, digestive aid), construction (builder's lime), farming (orchard fungicide), paper production (treating raw wood) and sewage treatment (as a flocculant).



The reaction that you will perform is the precipitation of calcium hydroxide from solutions of calcium chloride and potassium hydroxide. The overall reaction is shown below.



You will weigh out solid samples of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and KOH . Note that while CaCl_2 is the reactant in the equation for formation of $\text{Ca}(\text{OH})_2$, it is the hydrate, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, which is the source of the CaCl_2 . $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ has a formula mass of 147.02 g/mol, and there is one mole of CaCl_2 for each mole of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ used. Because it is the $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ that you weigh you must use the molecular weight of the hydrate for the purpose of determining the number of moles that you weigh. This is needed for the limiting reagent calculation.

Both starting materials are soluble in water, and when they are mixed, an insoluble product forms (precipitates). This is the $\text{Ca}(\text{OH})_2$. Your product will be collected using Hirsch funnel filtration and weighed to determine the actual yield.

Procedure

Follow the procedure below being sure to note down all errors, observations and any issues or challenge you might have. Detailed notes are a scientist's best friend because you never know what observations are important until later.

1. Weigh out by firstly taring the balance with the conical flask on the balance (zeroing), then measuring between 280 - 300 mg $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and 170 – 200 mg KOH into two separate, labelled, conical flasks. Be sure to take your flask off the balance while adding material to protect the balance. Record the mass accurately (include significant figures and units) of each of the reactants used in your worksheet.
2. Add 10 mL deionised (DI) water to each flask and swirl until both solids are completely dissolved. **CAUTION:** if you continue with the experiment before your compounds are fully dissolved, your results will be inaccurate. Why?
3. Carefully pour one solution into the other and mix well. Record your observations.

4. Pre-weigh a piece of filter paper whilst allowing your reaction to complete. Ensure this mixture sits for a minimum of 10 minutes while you organise the filter paper, organise your notes and ask any questions you need answered. Longer is better if you have spare time. This will increase your yield.
5. Collect your solid product using Hirsch funnel filtration according to the video that was watched as part of your prelab exercise. **Note:** do not turn the vacuum on high or you may tear the paper or cause some of your product to go through the filter paper. You will need to ensure all holes are covered and it is lightly wetted.
6. If you see solid in the filtrate (liquid that went through the filter), you will need to filter again by tipping it back into the original flask and pouring it back.
7. Wash the product with a small amount of DI water, then a small amount of acetone to displace the water and help the product dry faster. Then allow the solid to dry on the filter paper with the vacuum turned on for at least 10 minutes. If you notice a clump of wet solid, you can poke it with a glass rod to help it break up and dry. A wet product will give you an inaccurate mass. Why?
8. Weigh your solid product. Record the mass of the filter paper plus solid and calculate the solid only weight (minus the filter paper).
9. Using a pipette, transfer a small amount of filtrate into two separate test tubes. Into one, add approximately 1 mL of 0.1 M CaCl_2 into the other add approximately 1 mL of 0.2 M KOH. Wait for at least 5 minutes to observe any reaction. Use your observations to discuss how much of which reagent you think is in excess in the filtrate.



Experiment 2: pH titrations of weak acids and buffers

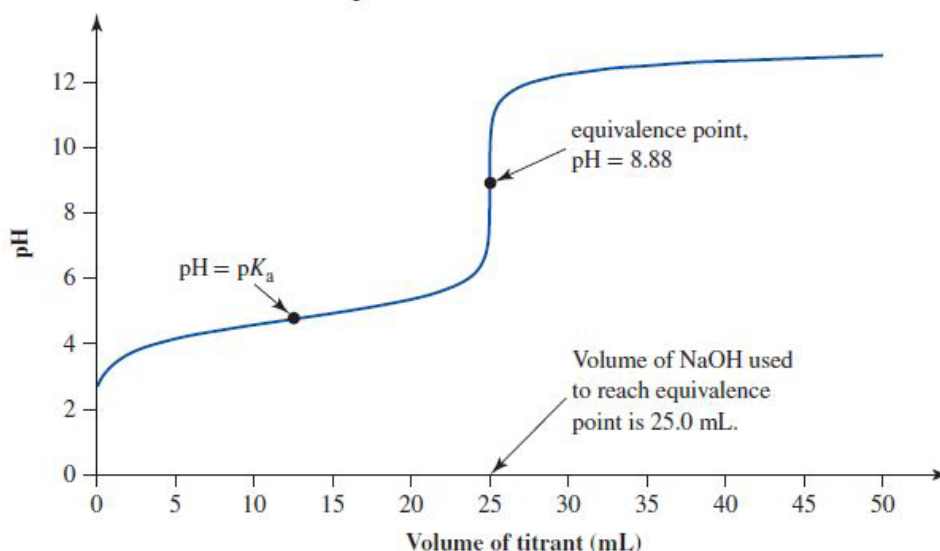
Safety Notes:

- The solutions may cause skin irritation, always wear PPE. If there is any skin contact, wash well with tap water.

In a pH titration, a curve is plotted of pH values against the volume of titrant added (see Figure 11.21 from the textbook below and on page 673). Over most of the titration range, the pH varies gradually, but near the equivalence point the pH changes more abruptly as the concentration of the analyte (H^+) undergoes a rapid variation. The equivalence point may be located by inspection from the inflexion point of the titration curve, i.e. the point which corresponds to the maximum rate of change of pH per unit volume of titrant added, the straight up and down section.

FIGURE 11.21

Titration curve for the titration of a weak acid with a strong base. In this titration, we follow the pH as 25.00 mL of 0.200 M $\text{CH}_3\text{COOH}(\text{aq})$ is titrated with 0.200 M $\text{NaOH}(\text{aq})$.



For a salt derived from a weak acid and a strong base the pH of an aqueous solution is given approximately by:

$$\text{pH} = 1/2\text{p}K_w + 1/2\text{p}K_a + 1/2\log c$$

where

| | | |
|-------|---|---|
| K_w | = | auto ionisation constant for water (10^{-14}) |
| K_a | = | acid dissociation constant for the weak acid |
| c | = | concentration of the salt (M) |

This equation can also be used at the equivalence point of a titration of a weak acid against a strong base.

The term "buffer" is used to describe a solution which does not change pH upon addition of a small amount of acid or base. A common way to prepare a buffer is to combine an acid with a salt of its conjugate base. For weak acids and their conjugate bases at moderate concentrations ($>10^{-5}$ M), the pH of a buffer solution prepared in this way can be calculated using the Henderson-Hasselbalch equation:

$$\text{pH} = \text{p}K_a + \log \frac{[\text{conj. base}]}{[\text{acid}]}$$

In this experiment, you will observe pH changes as a function of added base for a weak acid / strong base combination, and for a buffer that you will prepare. The literature $\text{p}K_a$ value for acetic acid is 4.76.

Procedure

The experiments will be performed in pairs with each student performing a set of three titrations, but each student must complete their own worksheet and all calculations independently. A demonstration of the pH meter in class will be important to watch closely to ensure you know what to do to get reliable readings quickly and efficiently. We know no one likes running out of time in the lab.

A. CH_3COOH vs. NaOH

1. Pipette 25.00 mL of 0.10 M acetic acid into a 150 mL beaker, add a magnetic stirrer. Fill the burette with 0.10 M sodium hydroxide. Note the two decimal places on the 25.00 mL.
2. For this titration, in order to observe the pH change which occurs when titrant is added in the early stages of titrations involving acetic acid, titrant should be added in 1 mL increments for about the first 5 mL to detect whether this section is curved or flat. Larger additions (approx. 5 mL) may then be made until near the equivalence point at the point pH starts to rise more quickly. Your overall additions should be very closely spaced (0.1 mL) near the equivalence point (you can expect the equivalence point to be roughly around pH 7). The titration should be continued until the pH plateaus (when you no longer observe sharp changes in pH as you add more titrant) after the equivalence point.
3. Discard the analyte solution and wash the pH meter with DI water. Rinse the beaker in preparation for the next titration. If required, repeat.

B. Acetate buffer vs. NaOH

1. Using a pipette that has been rinsed (with a few mL of buffer 2-3 times), pipette 25.00 mL of your buffer into a 250 mL beaker. Again, fill the burette with 0.10 M sodium hydroxide.
2. Perform a titration as described in Part A. *Note:* You can expect the equivalence point to be roughly around pH 7.
3. Repeat part B if required.

Concordancy: If your three volumes for titration are different (not within 5-10 percent) how would you work out which one is right? The answer is you can't. When using titration professionally you must be able to reach concordancy which is a fancy word for agreement between your results. Were any of your results within 10% of each other? What about 5%?

Experiment 3: Electrochemistry - corrosion and activity series

Safety Notes:

- Wear gloves always when handling hydrochloric acid.
- Hydrochloric acid can cause burns. If spilt on gloves carefully remove your glove by turning it inside out as you remove it and putting it straight in the chemical waste.
- The metal solutions may cause skin irritation. If there is any skin contact wash well with tap water.

Introduction

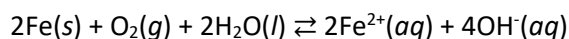
Rust is a hydroxide of iron and the anodic half reaction necessary for the oxidation of iron in an aerated corrodant (such as salt water) is as follows:



The cathode half-reaction necessary for the reduction of oxygen is as follows:



Combining these equations gives:

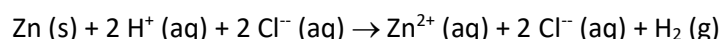


The ferrous hydroxide $[\text{Fe}(\text{OH})_2]$ is then rapidly oxidised by oxygen to ferric hydroxide $[\text{Fe}(\text{OH})_3]$. The net result is that iron reacts with oxygen and water to give rust.

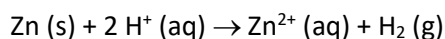
Since Fe^{2+} ions are produced at the anode and OH^{-} ions at the cathode, then anodic and cathodic regions may be detected by addition of potassium ferricyanide and phenolphthalein to the medium surrounding the electrode as follows. At the anodic region, where Fe^{2+} is produced it reacts with the ferricyanide to produce a blue colouration due to the formation of $\text{Fe}^{\text{II}}[\text{Fe}^{\text{III}}(\text{CN})_6]$, also known as Prussian blue. At the cathode, the OH^{-} ion produced reacts with the organic dye phenolphthalein (colourless in acidic or neutral solution) to turn pink.

Iron metal may be completely anodic when electrically coupled with a more noble metal or completely cathodic when electrically coupled with a less noble metal (this is the case for all metals). When coupled with a less noble metal, iron will not corrode. The less noble metal will instead corrode. Coupling steel with less noble metals is the basis of the technique known as anodic protection, i.e. the use of a sacrificial anode where we deliberately add a less noble metal knowing it will corrode protecting the more noble metal. Galvanising on the other hand, is an example of anodic protection. The anode is protected by a coating of more noble metal.

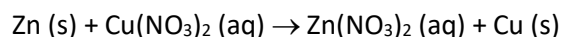
Reactions of metals with acid: Most metals react with acids, for example:



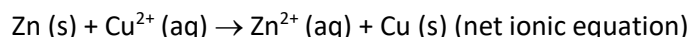
Since the chloride ion is a spectator ion it may be omitted, yielding the net ionic equation (you need this for the worksheet):



Electron transfer among metals: When a more reactive metal is in contact with a solution of a less reactive noble metal, an electron transfer will occur. For example:



Or



The more active metal loses electrons to the more passive metal or reacts more vigorously with water, acids, or oxygen. In some cases, in this laboratory activity, no reaction will occur. This is all controlled by the metal's reduction potential. From your observations in this experiment, you will rank the noble metals according to their relative chemical reactivities.

Procedure

The experiment will be performed in pairs, but each student must complete their own worksheet and all calculations independently.

A. Corrosion

A solution of 3 % sodium chloride, 0.2 % potassium ferricyanide, 0.1 % phenolphthalein and 1.0 % agar (all w/v) has been prepared and heated enough to melt the agar (gently), then poured into a Petri dish for you. Three nails will be pushed into this gel and the sites of any reactions will be identified by colour changes.

Nail 1 A plain iron nail

Nail 2

1. Take a plain iron nail and a copper wire (no coating) or copper foil
2. Wrap the copper wire tightly around **one end** of the steel nail
3. Rinse in acetone and allow to dry

Nail 3

1. Select a galvanised nail.
2. Remove the zinc coating on the lower half of the nail **ONLY** by dipping it in a 6.0 M hydrochloric acid solution for a few minutes.
3. Rinse in DI water, then acetone and allow to dry

Place **warmed** nails into the agar. Over time observe and record the colour change (go on with B & C while you wait).

B. Reactions of metals with acid

1. Add a small piece of the metals: Ca, Cu, Fe, Mg, Sn, Zn and Al to each of seven test tubes then add 1 mL of 6 M HCl.
2. Observe the test tubes and note any changes that occur (such as the evolution of gas, whether it is vigorous or not, and any colour changes). Enter your observations on the worksheet.

C. Reactions of metals in solutions of metal ions

1. Add a piece of calcium metal to each of the seven test tubes containing, respectively, about 1 mL of $\text{Ca(NO}_3)_2$, CuSO_4 , FeSO_4 , $\text{Fe(NO}_3)_3$, $\text{Mg(NO}_3)_2$, SnCl_4 , and $\text{Zn(NO}_3)_2$ solutions (all concentrations 0.2M). Note any reaction that occurs on the surface of the metal or in the solution or whether a gas is evolved. Record your observations.
2. Repeat the process by adding a small amount of copper, iron, magnesium, tin, aluminium and zinc to another 1 mL of each of the metal-cation solutions and record your observations for each experiment. Note it can help to look at the table in the worksheet for organisation.

Experiment 4: Preparation of biodiesel from waste vegetable oil

Background

The high-energy content of fatty foods can make eating large amounts of these foods unhealthy. It is this same high-energy content however, that can make fats a source of fuel for cars. There are a few problems with using vegetable oils directly as fuels. First, they are quite viscous, so they do not flow well, particularly at low temperatures. Second, they have low volatility leading to problems with incomplete combustion. Most fats consist of triesters of long-chained fatty acids with glycerol (glycerine). These triglyceride fats can easily be modified by being broken into smaller esters with lower viscosity and slightly higher volatility. A convenient way to do this is by a process known as transesterification. By reacting the triglyceride with three equivalents of a low molecular weight alcohol (such as methanol) in the presence of either an acid or base catalyst, the glyceryl esters are transformed into three smaller esters (Figure 1). The product of this reaction is called a biofuel because it is made primarily from biomass. This specific fuel is often referred to as biodiesel since it can be used in diesel engines. Even though the viscosity can be decreased, and volatility can be increased by the transesterification reaction, the high cost of the vegetable oil has kept this method from being put into wider commercial use.

One way to lower the cost of producing biodiesel is to use waste cooking oil. The high temperatures involved in deep fat frying, however, can cause the oil to undergo partial hydrolysis, which liberates some of the fatty acids from the triglyceride. The acid catalysed reaction is slower than the base catalysed reaction but has the advantage of being able to convert the free fatty acids into biodiesel via a Fischer esterification. To increase the reaction rate, 1-propanol can be used instead of methanol. The increased reflux temperature of 1-propanol (due to its increased boiling point) greatly increases the reaction rate and the increased chain length on the 1-propanol versus methanol allows for better mixing of the reactants. The methanol product is quite polar because the chain length of methanol is so short.

In preparation for class prepare a flow diagram summarising what you need to do in this laboratory experiment. Writing a flow diagram of what you will do during the lab will help immensely with understanding the lab when you are actually doing it.

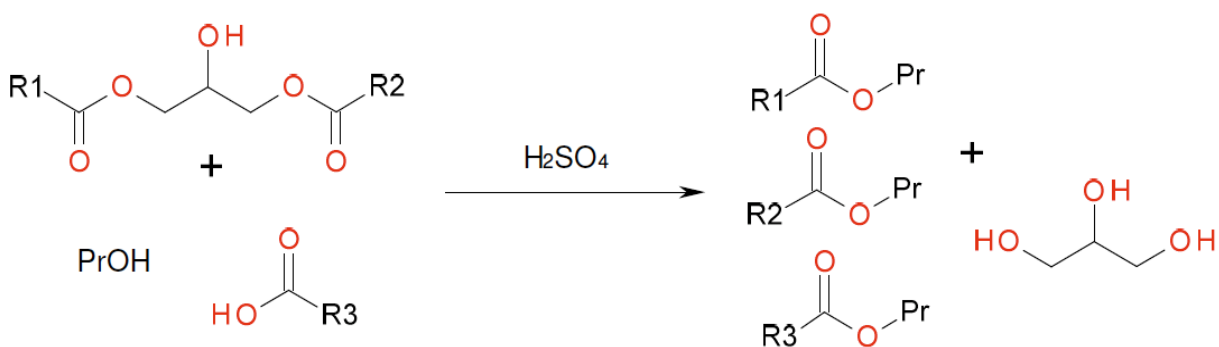


Figure 4.1: Acid catalysed transesterification of vegetable oil

Procedure

Part A: Acid catalysed preparation of biodiesel

1. Put a magnetic stir bar into a 125 mL reaction flask.
2. Combine 1-propanol (8.0 mL) with concentrated sulfuric acid (0.15 mL) in the reaction flask.
3. After mixing, add waste vegetable oil (20 mL).
4. Fit a reflux condenser to the reaction flask. Have a demonstrator check your equipment
5. Heat the mixture on a hot plate or heating mantle to reflux for 1 hour (how do you work out what temperature this will be?).

Separation:

After one hour at reflux, you need to cool the mixture and collect the crude product.

6. Allow the reaction mixture to cool slightly, transfer to a separating funnel and allow the layers to separate (this may take several minutes).
7. Drain the clear brown glycerine layer off the bottom (if it's not clear and is not becoming clearer ask the TA about how clear it needs to be).



Wash the organic layer with NaCl:

8. Add one 15 mL portion of (aqueous) 1M NaCl solution to the organic layer in the separating funnel
9. Stopper the separating funnel and rock/invert very gently, being careful to avoid forming emulsions by mixing vigorously.
10. Vent the funnel at least once (as shown by the TA) and when shaking is complete make sure that the stopcock is closed, then return the separating funnel to its support stand.
11. Drain the aqueous (NaCl) layer off the bottom. This procedure removes any remaining inorganic reactants and impurities.
12. Repeat steps 8-11 two more time to completely wash the organic phase of acid and polar impurities.
13. Check the final washing with pH paper to ensure the pH is neutral (indicates you have succeeded in removing the acids).
14. Drain the ester layer into a flask and dry (remove water) with anhydrous calcium chloride or MgSO_4 .
15. Put a small plug of cotton wool at the bottom of a funnel and use the cotton wool stoppered funnel to gravity filter the product to obtain the biodiesel.

Part B: Analysis of biodiesel quality

Use the following tests to compare the quality of your biodiesel with the vegetable oil.

1. Create two oil-lamps with 2 small vials, cotton string and aluminium foil. Wrap the foil around the top of the vial to support the cotton string. Leave approximately 5 mm of string sticking out the top of the vial and enough of a tail to stay submerged in the oil.
2. Place 1 mL biodiesel into one vial. Be sure to coat the entire string with the liquid.
3. Place 1 mL vegetable oil into the other vial. Be sure to coat the entire string with the liquid.

4. In the designated section of the laboratory away from the flammable solvents, check the ease of lighting each lamp, quality of flame, and time required for the flame to extinguish itself. Does one wick ignite easier than the other? Which liquid wicks better? Which burns stronger? Can you relight the wick once it is extinguished?

Viscosity:

1. Fill one Pasteur pipette with vegetable oil and another with biodiesel and allow them to drain (without the bulb) simultaneously.
2. Record the time it takes each pipette to empty. Which is more viscous, the vegetable oil or the biodiesel? How does viscosity relate to wicking?

Experiment 5: Modelling Chirality (all instructions on worksheet)

The aim of this exercise is to investigate the 3-dimensional shape of organic molecules and to help you visualise the connection between the real 3-dimensional shape and the flat 2-dimensional representations in books and on whiteboards.

Thinking in three dimensions is one of the most important skills in chemistry. Most organic molecules (especially biological molecules) function through, and because of, their particular three-dimensional shapes. You must be able to translate flat pictures of molecules into three-dimensional models in your mind in order to do well in either organic chemistry or biochemistry.

This exercise will allow you to become familiar with the 3-dimensional (3-D) structures of some hydrocarbons. When evaluating the 3-D aspect of your models you should be thinking of **geometry** and the associated **bond angles**. From the lectures and your textbook readings you should know the terms associated for a carbon atom that has four groups covalently bonded to it such that the attached atoms are equally spaced and have equal bond angles. Additionally, this exercise will expose you to the topics of **isomers**, **structural formula**, **condensed structural formula**, and **skeletal formulas**. You will be using the model kits provided. The important pieces are:

- 1) The tetrahedral pieces (black, red and blue) which are the standard (or CPK) colours for carbon, oxygen and nitrogen respectively, and
- 2) The sticks to represent bonds to connect the carbon atoms to one another and to connect the carbon atoms to other atoms:
 - the short thicker ones represent single bonds and
 - the longer skinny ones are used for multiple bonds.
- 3) Typically, the small white or grey balls with one hole represent the hydrogen atoms.

As you construct each molecule discuss it with your colleagues and make sure that you understand all aspects of it before moving on to construct the next model. If there are doubts about a particular model, be sure to consult with your demonstrator.

Some definitions

Isomer

One of several species (or [molecular entities](#)) that have the same [molecular formula](#), but different physical and/or chemical properties.

Structural or constitutional isomer

Isomers having the same [molecular formula](#) but different [physical](#) and [chemical properties](#) because the connectivity of their component [atoms](#) is different.

Stereoisomers

Isomers in which the atoms are connected in the same way, but are different in the way the atoms are arranged in space.

Geometric (cis/trans) isomers

Stereoisomers, which have restricted rotation somewhere in the molecule so that the *cis* isomer has similar atoms (or groups) on the same side of the restricted-rotation region, while the *trans* isomer has similar atoms (or groups) on opposite sides of the restricted-rotation region.

Enantiomers

Stereoisomers, which have a mirror-image relationship. (Greek *enantio*, meaning “opposite”.) All the stereocentres in one isomer must have the opposite configuration to all the stereocentres in the other isomer.

Diastereoisomers

Non-geometric stereoisomers, which do not have a mirror-image relationship. Some of the stereocentres in one isomer have the opposite configuration to the corresponding stereocentres in the other isomer, but some of the corresponding stereocentres have the same configuration in both isomers.

Stereocentre or stereogenic centre

A carbon (or similar) atom with four different substituents (atoms, functional groups, etc.).

Chiral

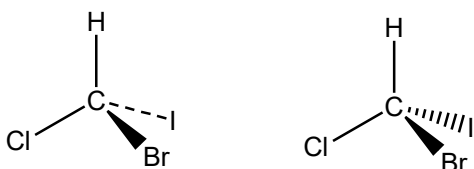
A chiral molecule is one that cannot be superimposed on its mirror image.

Achiral

An achiral molecule is one that can be superimposed on its mirror image.

Wedge and dashed wedge representations

The following *wedge* and *dashed wedge* pictures represent a substituted methane:



The convention is that all solid-line bonds are in the plane of the page; *solid wedge* bonds point towards the viewer or reader; *dashed wedge* bonds point away from the viewer or reader. Hence the above representations show C, H and Cl atoms in the plane of the page, the I atom is behind the page, while the Br atom is in front of the page.