Discipline does not mean suppression
or control, nor is it adjustment
to a pattern or ideology. It means
a mind that sees 'what is' and learns
from 'what was'. -- Krishnamurti

The Synthesis and Characterization of zinc iodide

It may be a little difficult to believe today, but in the late 1700's and early on into the 19th century the raging controversy among chemists was the idea that matter was particulate and had fixed chemical properties, i.e., that it was composed of what we today call *atoms*. The evidence for the earliest atomic hypotheses is fairly simple:

1. There are *elements* and *compounds*; unlike mixtures, compounds do not retain the properties of the elements in them
2. A given compound always contains the same proportion of its elements by mass (Proust)
3. When two elements form several different compounds, their relative masses are ratios of small whole numbers (Dalton)
4. Mass is conserved in chemical processes (Lavoisier) such as the formation of compounds from elements

Because we take the concept of atoms for granted today, it is sometimes a stretch to imagine that from this rather simple experimental evidence people like John Dalton were able to postulate the existence of *atoms* (1808) and use the word in much the same way that we do today. Therefore in this experiment you will see if you can reproduce some of this evidence by making zinc iodide from the elements zinc (Zn) and iodine (I), investigating the proportions in which they combine (to determine the chemical formula), comparing the properties of the compound with those of the original elements, and decomposing the compound to (hopefully) obtain the original elements.

The composition of a compound is often expressed in percentages by mass. The entry at the end of this experiment for zinc iodide has this information deleted but it is standard data which can be found in any good handbook. A simple example will show how the values are calculated:

Adapted from: Synthesis and Decomposition of Zinc Iodide: Model Reactions for Investigating Chemical Change in the Introductory Laboratory, Stephen DeMeo, *J. Chem Educ.*, 1995, vol. 72, No. 9, p.836

The formula mass of water, H_2O , is [2(1.0) + 16.0] or 18.0

Therefore the %, by mass of water that is **hydrogen** is:

$$\frac{2(1.0)}{18.0} \times 100 = 11\%$$

In a similar manner the % that is **oxygen** can be obtained:

$$\frac{16.0}{18.0} \times 100 = 89\%$$

Note that these are merely simple fractions which represent *the mass of a particular element in the formula divided by the total formula mass*. Notice also that in the case of hydrogen, the subscript $_2$ is used in calculating the % H since there are two hydrogens in the formula for water.

No matter how simple or complex the formula for a substance may be, its composition may be expressed in this manner. Proust's statement (number 2 on the facing page) simply says that the numbers calculated for a compound are always the same for that compound, regardless of how it is prepared or from where it is obtained. This is equivalent to saying that water is always H_2O, never H_4O, HO_2, or some other arbitrary combination.

Fundamental atomic theory also tells us that atoms cannot be divided into smaller parts without losing their chemical identities. During chemical processes, atoms remain intact. Thus atoms which combine to form molecules must do so in whole-number ratios (Dalton). The smallest whole-number ratio of atoms in a molecule (compound) is known as the *empirical formula*. If discrete molecular units exist which have this ratio, then the formula is also the *molecular formula*. For example, the empirical and molecular formulas for water are both H_2O.

In contrast, some compounds exist only in a multiple of their empirical formula. Hydrogen peroxide is an example. The actual molecule contains two atoms of hydrogen and two atoms of oxygen. Thus the molecular formula is H_2O_2. The empirical formula is simply HO. In general once the empirical formula of a substance has been established in the laboratory, other tests are required to determine if there is a different molecular formula.

To find the empirical formula, a ratio of numbers of atoms (or *moles*) is needed. What is measured in the lab is typically *mass*. A simple example illustrates the process of conversion:

1.50 g of Mg metal is placed in a crucible and heated until it ignites. After cooling, the sample is found to have a mass of 2.49 g.

Assuming that the product contains only Mg and O, the mass of O would be:

$$2.49 \text{ g} - 1.50 \text{ g} = 0.99 \text{ g}$$

Moles of each element are then calculated:

$$\text{moles Mg} = \frac{1.50 \text{ g}}{24.3 \text{ g/mol}} = 0.0617 \text{ mol}$$

$$\text{moles O} = \frac{0.99 \text{ g}}{16.0 \text{ g/mol}} = 0.0619 \text{ mol}$$

Within experimental error this is a 1:1 ratio so the empirical formula is **MgO**

In order to know if this is the actual molecular formula we would need information such as the measured molar mass--but that's another experiment!

Sometimes it is not so obvious from a simple calculation just what the integer ratio should be. Consider the next example:

7.3 g of finely divided Al is heated in oxygen. The final mass after complete reaction is 13.8 g. What is the empirical formula of aluminum oxide?

The mass of oxygen would be: 13.8 g - 7.3 g = 6.5 g O

The moles of each element then are: $7.3 \text{ g Al} \times \frac{1 \text{ mole}}{27.0 \text{ g}} = 0.27 \text{ mol Al}$

$$6.5 \text{ g O} \times \frac{1 \text{ mole}}{16.0 \text{ g}} = 0.41 \text{ mol O}$$

It is not exactly clear what the ratio is here but you can get a better idea by dividing the smallest number of moles *into* each value:

$$\frac{0.27}{0.27} = 1.0 \qquad \frac{0.41}{0.27} = 1.5$$

The corresponding integer ratio is thus 2:3 and so the empirical formula is Al_2O_3

What about zinc and iodine? By doing a little detective work in your book you can probably figure out what the formula for the compound of these two elements should be. Then you could calculate the % composition. But what if you didn't know the formula? The challenge in this experiment is to put yourself in the position of an early chemist: you are still trying to figure out what things are made of (and that's not far from the truth!). One other important aid in this endeavor is your own senses. Compounds and their elements seldom have the same physical properties or appearance (certainly not the same chemical properties). Your careful observations about physical appearance, water solubility, electrical conductivity, and so on can be very useful. Many compounds like zinc iodide are held together by electrical forces and so it is sometimes possible to break them up into their elements again using electricity. At the end of the experiment you can also try this to see if you recognize the original starting materials, zinc and iodine.

Preparing to experiment

You will be provided with the following materials:

1. granular zinc (use about 0.5 g)
2. iodine [at balances] (use about 0.5 g)
3. slightly acidic water (use about 3 mL)
4. two 18x150 test tubes
5. test tube clamp
6. 24-well plate
7. conductivity tester
8. 9-volt battery and wire clip

Design an experiment to make zinc iodide by reacting zinc and iodine in acidic water, recovering and measuring the amount of compound formed and the leftover zinc remaining. Test the compound for water solubility and electrical conductivity in solution.

Technique

Zinc can react with iodine pretty vigorously--the combination can be dangerous. So a little respect is due to these elements, especially iodine. *Be sure to read the information at the end of this handout on the substances in this experiment*. Because the two elements are solids at room temperature, reaction between them is very slow unless they are ground together vigorously. **THIS IS VERY DANGEROUS!** So we won't do it.

1. **Quantitative transfers** are very difficult to make. Stuff gets left behind. In an experiment of this kind, with small masses involved, loss of even a tiny amount can lead to significant experimental error. The best way to avoid this problem is not to transfer unless necessary.

Measure the zinc directly into a pre-massed test tube. You can stand the test tube up in a beaker which has been "tared-out" on the balance. It takes a little more time to place solid into the narrow test tube, but it's all in there when you're done. To add iodine, just tare the whole mess and keep adding----but be careful: if you add too much it all has to be discarded since the materials are contaminated with each other.

2. **Recovery from solution** requires different techniques depending on whether the material is soluble or not. Fortunately iodine is slightly soluble in water and bringing at least one element into solution speeds up this reaction significantly, but <u>constant swirling</u> is needed to get the job done in the class time. However, zinc will slowly react with water to form zinc hydroxide. This will interfere with your experiment, so rather than use the standard distilled water from your water bottles, *be sure* to use the slightly acidic water provided for this experiment. It will prevent the formation of zinc hydroxide during the reaction between zinc and iodine.

At the end of the reaction, zinc is left over at the bottom of the test tube but the product is invisible in the solution. The solution can be *decanted* into another clean, dry pre-massed test tube, i.e., the liquid on top (*supernatant*) is carefully poured off, either into another container (in this lab, another clean, dry pre-massed test tube) or the sink. Generally, to ensure that all of the substance in solution is removed/recovered, several small rinses of the solid and walls of the original container are also made. These are combined with the original decanted solution. In this experiment, any rinsings will add to the total volume of solution which must eventually be boiled away. It is therefore wise to keep the rinsings small, perhaps 3 rinses of 1 mL each---be sure to use the acidic water.

Solids in test tubes can be troublesome to dry other than slowly in an oven, but it is possible to heat the tube gently at first, then more strongly, and drive off any liquid present, holding the test tube in a spring clamp. Keeping the test tube moving through the flame will help prevent little steam explosions that can eject the contents. Also, the clamp itself will become too hot to hold if it is held in position over the flame constantly. Once the initial heating is done, deliberate heating, moving from the bottom of the test tube to the mouth--driving out any moisture--should be done. Any water remaining will make the final massing inaccurate.

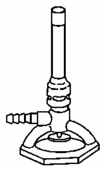
Liquids in test tubes boil very rapidly and have a tendency to "foam" up and --occasionally--out. To minimize this, hold the test tube at an angle above the flame and keep it moving all the time. Be sure not to point the mouth of the test tube at anyone. Because solutions sometimes bump and spurt out when being heated (especially as they become more concentrated), a "boiling stone" is often added to aid in the formation of smaller bubbles during boiling. These stones do add mass, however, and we will not use them in this experiment. Zinc iodide absorbs water rapidly from the air and should be heated until it is slightly yellow. Overheating will decompose the compound, liberating iodine gas and ruining your results.

It may be that your class will do the experiment on two consecutive days. In that case much or all of the drying may be done in the oven overnight.

3. **Placing hot objects on the balance is a no-no**. The heat they give off creates air currents which often cause the sensitive balance pans to move up and down. Also there is a possibility that *very* hot objects will damage the balance. The general rule is that you must be able to pick up the object *with your hand* and <u>walk</u> to the balance with it. If you can't, it's still too hot.

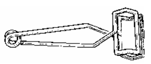
ALWAYS USE THE SAME BALANCE FOR AN ENTIRE EXPERIMENT.

Equipment



Bunsen burner

test tube



test tube clamp

The chemicals

Iodine can be obtained from a variety of natural sources including some brines (concentrated salt solutions) associated with oil wells and seaweed. In rocky minerals it is present only to the extent of 3×10^{-5} %, in seawater, 5×10^{-8} %. Once the compounds of iodine have been isolated they can be treated with chlorine which displaces the elemental iodine. At room temperature iodine is a shiny, gray-black, non-metallic solid. In the gas phase and in some solvents, it is violet. In water and alcohols (like ethanol) it has a reddish-brown color. *Tincture of iodine* is a solution of iodine in alcohol and is used as an antiseptic. *Iodized* table salt contains potassium iodide (KI) as a nutritional supplement to help prevent iodine deficiency diseases such as goiter.

In pure form and in solution iodine is very reactive and can cause staining on skin and clothing. Its vapors are irritating to the eyes, nose and throat. Iodine shares an unusual property with another common substance, carbon dioxide: it does not pass through a liquid phase at ordinary pressures but instead goes directly from solid to gas upon heating. This process is known as *sublimation*.

Zinc is generally obtained from ores of zinc containing sulfur. Its abundance in the earth's crust is about 0.02%. Zinc is a fairly reactive metal which combines readily with oxygen, sulfur and the halogens. Pure zinc, when exposed to air gradually becomes coated with white zinc carbonate ($ZnCO_3$). Most zinc compounds are colorless in solution (or white as solids). Zinc is readily attacked by dilute acids, releasing hydrogen as it dissolves. It is used in corrosion protection (*galvanizing*) and its compounds are employed as paint pigments and disinfectants.

Zinc iodide is a white (hydrated), or pale yellow (dry), odorless powder when pure. It becomes brown on exposure to air and light, slowly releasing iodine. One gram dissolves in 0.3 mL of water. It is sometimes used as a topical antiseptic or astringent.

Analysis

1. Use your data to determine how many grams of zinc reacted and how many grams were left over.

2. Assuming that all of the iodine reacted, what is the percent composition (by mass) of each element in the compound? (be sure to use your *data* for this, not some theoretical calculations) [*hint*: this determination should be based on the zinc reacted--from #1-- the iodine reacted and the recovered mass of zinc iodide]

3. Based on your answer to question 2, what is the empirical formula of zinc iodide?

4. Use cation and anion charges to determine the formula for a compound between zinc and iodine. Does the theoretical % composition calculation [show this!] from the <u>expected</u> formula match your result in question 2? If not, can you suggest where errors might have been made? (be specific--for example, if your mass percent of zinc is too large, what does this suggest? or if your mass percent of iodine is too large, what measuring error might you have made?)

5. Compare the mass of zinc iodide recovered with the expected total mass of product based on the masses of the elements which reacted. Should these masses be the same or different? Explain. In addition to those already discussed in question 4, what errors might have contributed to any inequality between the *expected* and *experimental* masses of zinc iodide?

6. What evidence did you see that both zinc and iodine were present in the final recovered solid? [*hint*: what happened when electric current was run through a solution of the product? Be specific for each of the two wires.]