Experiment No. 18

Gravimetric Determination of Lead (as Lead Sulfate)

On two occasions during this experiment you will be heating your crucible to constant weight. The first time empty and the second time after burning off the filter paper with your lead sulfate sample. The process is to heat the crucible with a very hot flame, cool it in the desiccator, and weigh it. Then repeat until 2 consecutive weighings are “constant”. Your TA will specify how close the weighings should be to be considered “constant”.

Use the black-based ringstand; set the burner on it and adjust the ring to be about 1.0 – 1.5 inches above the burner. Place the clay triangle on the ring and then put the crucible in it. Remove the burner, light it, adjust the flame and put the burner back under the crucible. Your TA will show you how to adjust the flame. It should appear as 2 shades of blue. Inside the outer blue should be a luminous blue shaped like an inverted cone. The hottest part of the flame is the tip of that inverted cone and should be in contact with the bottom of the crucible. If you look down into the crucible, it should be an orangish hue. The data page has space for 3 heatings, but if you are doing it correctly only 2 may be needed. Do not do a third unless necessary. You will need to keep half an eye on this process while doing the reaction part of the experiment.

First you need to determine the mass of your product from last week. Place a 150- (or 100-) mL beaker on the balance and zero it. Then transfer all of your product to the beaker and record the weight. If you slip a spatula between the filter paper and the edge of the lead iodide, you will find that most of your product will peel off as one big flake. Transfer any remaining sample by lifting it off the paper, do not scrape it off or you will contaminate with filter paper. You will likely have more product than the 200-220 mg amount to use for the reaction today. If so remove enough from the beaker to get down to the desired mass. If you need to remove some of the big flake, remove the beaker from the balance. Get 2 spatulas. Use one to hold down the flake and use the other to slice off pieces until you reduced to the desired mass.

There will be a jar on the desk where you can put any excess lead iodide. If you did not have enough, then take some from it. Put the spatula(s) in the “dirty” beaker. There will also be a bottle for the filter paper.

Take the beaker of lead iodide to your desk and add the stir bar. Instead of using 5 mL of concentrated nitric acid (HNO₃) and 5 mL of water, you will get about 10 mL of 50% nitric acid. Add it to the beaker under your hood, because when it contacts the lead iodide, it will darken and fumes may start to form. There will be 2 fumes: red-brown nitrogen dioxide and purple iodine. Both are poisonous. Cover with the watch glass and heat gently on the hot plate until there are no more colored fumes. You might have to add more acid.

While the reaction is proceeding, remember you are in the process of heating the crucible to constant weight. You can also get a 100-mL volumetric flask and rinse it with distilled water. DO NOT put soap in volumetric flasks as it it difficult to rinse it out. Soap can be a serious contaminant. It would very likely form a gummy deposit with lead ions.

When no more colored fumes are being generated, cool the mixture to room temperature and add ~20 mL of water. It should be a colorless solution. Transfer to the 100-mL volumetric flask, rinse the beaker twice with 5 mL of distilled water and add to the flask. Dilute to the line with distilled water using a Pasteur pipet for the last few drops so that you do not overshoot. Mix the solution by slowly inverting the flask so that the air bubble will travel through the solution from the stem of the flask to the base and then uprighting so that the bubble can travel back to the stem. Repeat 10 – 20 times. Just shaking the flask is not effective.

Use a 25-mL volumetric pipet and a pink pump to pipet two(2) portions of the solution into a 150-mL beaker. Set the remaining solution in the volumetric flask aside, in case something goes wrong with the next part of the experiment, you will have this solution to try again. Get 10 mL of concentrated sulfuric acid in a 10-mL graduated cylinder. While mixing with your stir rod carefully pour it into the beaker of solution. The rule about always pouring concentrated acid into water and not the reverse, most especially applies to sulfuric acid. If quickly poured into water without stirring it will get so hot that it will boil and splatter. Rinse the graduated cylinder twice
with 10 mL of water and add to the beaker. Wash your hands. If you get sulfuric acid on yourself, you may feel a
burning sensation. But sometimes you might not feel anything and not know you came in contact with it until you
notice a blister forming. Warm the solution on the hot plate as you prepare the filtration apparatus like last week.
Cool to room temperature. You may speed it up by using an ice bath if you wish.

Pour the liquid through the filter paper. Transfer the small amount of white precipitate by using the Pasteur
pipet to wash it with very dilute sulfuric acid to the filter paper. This diluted sulfuric acid will be under the window.
IT IS VERY IMPORTANT THAT YOU NOT USE CONCENTRATED SULFURIC ACID HERE. IT WILL
ATTACK THE FILTER PAPER AND TURN IT INTO A GELATINOUS MESS. This could be why you saved the
other half of the solution in the volumetric flask. Draw air through the filter paper for a few minutes. Hopefully you
have your crucible at constant weight by now. Carefully fold the filter paper and transfer it to the crucible. Rinse
your hands.

Heat the crucible and filter paper gently at first to dry the paper and then more strongly. The paper will
char and turn black. Then glow red as it combusts. Finally the black should burn away leaving a small amount of
white solid. This should be under the hood as the odor is not pleasant. You are now in the constant mass mode again.
Heat, cool, weigh; heat cool, weigh It will probably take more than two cycles. One thing to watch for: when the
paper burns it often leaves behind a tar like deposit. Each time you heat the crucible a little more of it burns off and
changes the weight. If you have such a deposit, concentrate on burning it off. Change the position of the crucible,
hold the burner to direct the flame at the deposit.

During this process you can dispose of the waste in the jug. Return the stir bar. Clean the glassware. Once
you reach constant weight, gently scrape any loose product into the waste jar and put the crucible in the tray by the
ovens like you did with the beakers 2 weeks ago, Do not wash the crucibles.