The Properties Unknown Substances, An Interview with Sherlock Holmes, Consulting Detective

Introduction:

Only on rare visits to the London rooms of Sherlock Holmes at 221B Baker Street did I find my old friend engaged in laboratory pursuits. This was one of those occasions. It was a dreary, wet day in the winter of the year when I was admitted by the housekeeper. I found Holmes in his laboratory contemplating several small vials containing white solids and colorless liquids. Holmes greeted me with a smile, nodded toward the small sealed bottles, and said enthusiastically, "Watson, here is the most challenging problem that a chemist can face. To be presented with a small, sealed, unlabeled bottle containing a colorless substance and to be asked, what is it?" I face such a puzzle now, Watson. These mysterious vials were delivered by courier this morning. The accompanying letter requested me to ascertain the identities of these substances; the letter bore the seal and signature of an heir to the throne of a small European monarchy. The resolution of this mystery is a matter of utmost gravity. I have not been faced with such a task since the *Adventure of the Moravian Mountebank!"*

"I don't remember that one. Holmes," I said in a confused tone.

"It was a quickie, Watson, I guess I forgot to write it up."

"Oh."

"You see, Watson," he continued, "if you have absolutely no clue as to the identity of a substance, you have a real problem. It could be a mixture of many different things, it could be explosive or extremely toxic, it could be radioactive and lethal, it could be a crystallized sample of a virus like *Ebola* and you could initiate a world population threatening plague just by opening the bottle."

"Fascinating," I said hoping he would not open any of the vials in my presence, "how will you proceed with the investigation?"

"Watson, the characterization of an unknown is a process involving logic, careful observation, and deduction. One begins by examining general properties, the presence or absence of which can eliminate whole classes of substances. One carries out such preliminary tests with very small amounts of material. You only need to use enough to see what happens. As in this case, the sample may be all there is in the world! If you use it up, you can't get any more. A few of the basic starting questions are: How does the substance respond to heat? Is it a pure anhydrous substance or does it contain water of hydration? Does it dissolve in water? Does it dissolve in any other solvent? If it dissolves in water, how does the solution behave?

"Let me elaborate on some of these points," Holmes continued, "putting a little bit of solid on the tip of a spatula and holding it near (not in) a burner flame permits examination of the substance's behavior toward heat. One observes what happens. Does it melt? Does it burn? Does it change color or char? Does anything happen? If nothing happens, the spatula can be moved closer to the flame so that the sample is subjected to a higher temperature. Does anything happen then? If not, the spatula can be moved even closer to the flame. This can be repeated until either something happens or one can conclude that the substance is unaffected by heat. The tip of the spatula with the sample can be put into the burner flame. Does the burner flame become colored? All such observations and conclusions should be carefully recorded in a lab notebook."

"If a colored substance loses some of its color when held near the flame, it may be a hydrate. Water of hydration may cause the color of the compound to be darker or more intense."

"Are all hydrates colored, Holmes?"

"No, my dear Watson, some may be white."

"Then how can one be sure if the compound is a hydrate?"

"If you put a small amount of the solid in a small test tube and gently heat it in a burner flame, the water of hydration will form droplets on the cooler, upper part of the test tube."

"What happens to the residue, Holmes, does it melt?"

"Not always, Watson. Sometimes the residue may have a high melting point and no further change may be observed. However, there are a few organic compounds that will decompose on heating and will form water."

"This is confusing, Holmes. How does one recognise this?"

"The residue, Watson, usually turns brown or black."

"I see! But, some compounds do melt. Isn't that so Holmes?"

"Yes, Watson. If the substance melted a short time after being held near the burner flame, it may be possible to measure the precise temperature at which melting occurs. This temperature is called the ***melting point*** of the solid and is usually a characteristic property which can be an important piece of evidence for the later complete identification of the substance."

"The substance may have caused the burner flame to change color for a moment. Some elements give characteristic colors to flames under the conditions of this test. One needs to be careful with conclusions here, however, because the metal spatula itself can sometimes cause some flame coloration. Amateurs are especially vulnerable to this pitfall. Among the most common elements coloring flames is sodium (Na) which imparts an intense yellow color to a flame. This test is extremely sensitive. Minute traces of salt (NaCl) from a finger print can produce a strong yellow flame color. It is especially important to be cautious with such a result; it might be a false test caused by traces of salt or it might mask the color of another element that is really present in the unknown. As I recall, the police were misled by this false evidence in the *Adventure of the Bavarian Budgerigar."*

“How do you avoid these false tests?” I asked.

“Elementary, Mr Dear Watson!” Holmes explained. “Simply heat the tip of the spatula, or a wire test loop, whatever you may be using, in the burner flame, them dip it into some concentrated hydrochloric acid in a small beaker. That will remove the impurities.”

“Is it that easy?” I asked, amazed that some of the police investigators would not have known this.

“You might have to repeat this process several times until all the impurities are removed.” Said Holmes. “When it is clean, the metal will not impart any color to the burner flame..”

"Another quickie?" I queried.

"Yes."

"What about the question of dissolving in water or other liquids?" I asked after examining the list.

"Look at the other side of the chart," Holmes said showing mild annoyance at my lack of observational skill. "There you will find a set of elementary directions for ascertaining the properties of solids. I once prepared this for a Forensic Studies seminar I gave at Scotland Yard."

The reverse of the chart held the following entries:

Observations: Note the texture, shape, size, smell, etc. of your solid. This information alone can provide an abundance of information.

Flame Tests: Sometimes it is difficult to detect if there is a color change in a flame. Compounds are provided that exhibit a flame color change. You need to try these so you have something to compare your sample with and you may need the information next week.

Burning: Organic compounds tend to burn, often with a yellow sooty flame.

Hydrates: Some compounds contain water mixed in their crystal structure. If you suspect a hydrate, try heating a small amount of solid in a test tube.

Melting Point: If your solid melts in the flame, be sure and determine a melting point temperature on your solid.

Does the Substance Dissolve in Anything?

Plain Water: Place a small amount of solid on the tip of your spatula and drop the solid into a small test tube containing two mL of distilled water. Agitate the solid and water by gently tapping the test tube with your fingertip or place a piece of parafilm over the top of the test tube and gently *shake ... don't cover the end of the test tube with your thumb and shake!* This is poor technique. You can contaminate the sample with salt from your skin, and, in the real world, you don't know whether the unknown might be absorbed through the skin (lots of things are). Did the substance dissolve? Did anything happen? Record your observations in your notebook.

Dilute Acid: Try the same procedure with two mL of 0.1 M hydrochloric acid in place of the water.

Dilute Base: Try the same procedure with two mL of 0.1 M sodium hydroxide in place of the water.

Alcohol: Repeat the experiment with your solid and two mL of isopropyl alcohol.

Acetone: Repeat the experiment with your solid and two mL of acetone.

Behavior of the Water Solution:
If your unknown dissolved in plain water, it is worthwhile to examine some fundamental properties of the solution such as pH and conductivity..

pH Test: Some substances characteristically make water acidic when they dissolve, others make water basic or alkaline, and some have no effect at all. The so-called pH of a solution is simply an indication of how acidic or basic the solution happens to be. A pH less than 7 indicates an acid solution; the smaller the pH number, the more acidic the solution. Conversely, a pH greater than 7 implies the solution is basic or alkaline; the higher the number, the more basic the solution. Dissolve a small amount of your unknown in water and place a drop of the solution on a piece of universal pH paper. The paper is impregnated with dyes that turn color in response to different pH levels. You will have a color key for interpretation of the result available in the lab.

Is the Substance an Electrolyte? This involves a very simple test to determine whether the water solution conducts electric current well enough to make a small flashlight bulb light up. Normally, pure water does not conduct electric current well enough for this to happen. When a dissolved substance increases the ability of water to conduct electric current, that substance is called an *electrolyte.* Dissolved substances that do not change the current conducting ability of water are known as *non-electrolytes.*

Electrolytes are substances that dissolve in water to form positive (cations) and negative (anions) ions. Thus, if a substance is an electrolyte, a water solution of that substance will conduct an electric current. The most common electrolytes are ionic compounds (usually a compound of a metal with a non-metal), although some molecular compounds like strong acids also produce positive and negative ions in water and give a positive test for an electrolyte.

Non-electrolytes are usually molecular compounds (non-metal - non-metal compounds). They do not form ions in water solution and will not give a positive test for electrolytes.

CHARACTERIZATION OF COMPOUNDS

The Problem: For this project each student, or student group, will receive two colorless or white substances in screw cap vials with coded labels. Your job is to characterize, as well as possible, the two unknowns given to you. You must confirm your observations on each unknown with the other student(s) in the section, some of whom may be in possession of the same substance.

The overall goal for the whole lab section is to prepare a ***Table of Measurable Properties*** for all the coded unknowns. Each entry, measurement, or observation on a given substance in the table should be confirmed and agreed upon by all students having that unknown. In other words, if two students measure the melting point of a given substance and find 87°C, but if a third gets 95°C for the same substance, the measurement should be repeated until everybody agrees on the result.

The *Table of Measurable Properties* will be constructed on the chalk board or white board and will be used by the whole lab section. Each person will be responsible for putting their information on the data table.

At the conclusion of the experiment, you are to present you data to your instructor, who will give you a list containing the names of the compounds you characterized. You must, then, look up each compound in a chemistry handbook and compare your results with the literature values.

The tools you have to work with are:

- Two unknown substances, the unknowns are in vials labeled A through N
- Spatula or scoopula
- Bunsen burner
- Melting point apparatus with capillary tubes (See *The General Chemistry Laboratory Survival Manual* for instructions.)
- Deionized water

Hydrochloric acid, 0.1 M
Hydrochloric acid, concentrated
Sodium hydroxide, 0.1 M
Isopropyl alcohol, 99%
Acetone
pH paper, wide range
Conductivity tester
Flame test standards
Flame test loop
Beaker, 50 or 100 mL
Test tubes, 13 x 75 mm
Test tubes, 18 x 100 or 150 mm
Test tube holder

Most of the apparatus is in your laboratory drawer. Additional items will be supplied in the lab.

Safety Precautions:

Wear safety goggles in the laboratory at all times.

Isopropyl alcohol is flammable, keep it away from any flames. Continued skin contact with isopropyl alcohol can cause drying and redness of the skin.

Acetone is flammable, keep it away from any flames. Acetone is commonly called nail polish remover. Continued skin contact with acetone can cause drying and redness of the skin.

0.1 M hydrochloric acid can cause minor irritation of the skin. In the event of skin contact, wash the affected areas with water.

Concentrated hydrochloric acid can cause burns to the skin. Its fumes are irritating. Work in a fume hood. In the event of skin contact, wash the affected areas well with water. If redness or burns occur, seek professional medical assistance.

0.1 M sodium hydroxide acid can cause minor irritation of the skin. In the event of skin contact, wash the affected areas with water.

When determining flame tests and melting of materials using a spatula, the spatula will get hot. Allow the spatula to cool before handling.

Disposal

All materials must be disposed of according to laboratory regulations. Please place all wastes in the proper containers provided in the laboratory.

Any vials containing unused unknown compounds should be returned to the stockroom. Please do not discard these materials.

Report

You are required to prepare a **report**, which will be **due next class.** In the report, list the tests that you performed on your unknown along with the results. You will be supplied with a list containing the identity of the unknown compounds you tested. You must go to a Chemistry Handbook and compare your results with the published properties of the compounds. You should tell whether your results are in good agreement with the published values or if they disagree. You should also classify the compounds as organic or inorganic, and may be able to state the identity of some elements present, based on your laboratory tests.