Identification of an Unknown – Two-Component Unknown – Procedure

Purpose:

You are the first to arrive at *another* crime scene. There is a mysterious white powder by the base of the right foot of the body. What is it? Is it a pure substance or a mixture? In the second week of this experiment you will determine the composition of an unknown, two-component mixture that was collected at the scene of the crime. The possible substances are again given in Table 1 at the end of this procedure. Did the victim choke on table sugar laced with antiperspirant, or was the death caused by baking powder mixed in chalk?

Procedure:

Today your unknown will be a two-component mixture. One component is *soluble* in water, while the second component is *insoluble* in water. Any substance which has a listed solubility of *greater than* 0.5 g/100 mL of water can generally be considered soluble. Likewise, any substance which has a listed solubility of *less than* 0.5 g/100 mL of water can be considered insoluble. It is critical that you understand this point, and how to read the solubilities of the possible unknowns listed in Table 1. Can the insoluble component of your unknown be NaCl? Clearly not, since you can dissolve 209 grams of NaCl in 100 mL of H_2O. Use common sense!

Each person will once again have their own unknown. You are encouraged to work as a team with your lab partner, but *it is solely your responsibility to correctly identify both components of your unknown*. For the last time, your two-component unknown contains

one H_2O soluble component

and

one H_2O insoluble component

and

you must identify both components!

Obtain an unknown and record the sample ID code in your notebook. Without an ID, you cannot get credit for this experiment. You can apply the tests described in the Week 1 Procedure in any order that you wish, but some tests will logically follow others. Again, it is absolutely critical that you not only perform these tests on your unknown, but on the provided known standards as well. Use a flowchart to keep track of the compounds you have eliminated with your tests and those that remain as possibilities.

Separation of the Soluble and Insoluble Components

Given that the mixture unknown contains two compounds that differ greatly in their solubility in water, the first step in your analysis is to separate the two compounds. Take approximately one-third of your unknown sample and put it into a medium-sized, scrupulously cleaned, plastic test tube. Plastic tubes (if available) are preferable to glass tubes because the plastic tubes will not shatter in the centrifuge. The tube must be clean and rinsed with deionized water to minimize potential contamination. Add deionized water and mix thoroughly using a stirring rod.

Centrifuge this mixture in the plastic tube. Make sure to balance the centrifuge by placing an equally weighted tube (filled with just water), or another student's sample tube, opposite from yours. Failure to counterbalance samples in the centrifuge can result in a catastrophic failure! Allow the centrifuge to come to a complete stop before opening the cover and retrieving your sample. It is bad technique to use your fingers as a "brake" to slow the centrifuge. First of all, you risk losing a finger and perhaps further injury, but also this will disrupt the settling of the insoluble component within the tube.

Retrieve your sample from the centrifuge and decant the top, clear solution (the supernatant) into a clean, labeled beaker. The supernatant is an aqueous solution of the soluble component of your unknown. Try not to pour off any of the solids left in the bottom of the tube. Wash the remaining solid by adding deionized water and again mix thoroughly and centrifuge. Decant the supernatant and discard it. Repeat this washing step *at least* one more time. The remaining solid should be a pure sample of the insoluble component of your unknown. The repeated washing steps are really important because if your solid sample is contaminated with the solution containing the soluble unknown, you will get very misleading results in the chemical tests.

At this point, you have physically separated the two components of your unknown. Now you must perform tests on each component to unambiguously identify each component. The tests are the same as those done in Week 1, and are given in the Week 1 procedure. If during the course of the chemical tests, you need more of a particular component of your unknown (soluble or insoluble), you must perform the physical separation again.

Waste Disposal: Dispose of all waste (solid and liquid) in the appropriate waste container.

Conclusion:

Identify both substances in your unknown. Justify your identifications by referencing all of your results and observations. Your grade depends on:

- the correct identification of both components of your mystery mixture.
- the strength of your arguments supporting your identifications.

The more clearly (and neatly) you present your concluding arguments, the easier it will be to convince a jury (and your instructor) of the composition of your unknown sample. The best way to support your conclusion is to refer to a neatly drawn flowchart of your results.

Table 1. Unknown Candidates

IUPAC name	common name/use or trade name	formula	melting point (°C)	solubility (g/100 mL H_2O)
aluminum chloride hexahydrate	antiperspirant	$AlCl_3 \cdot 6H_2O$	100 (d)[a]	45
aluminum hydroxide	Amphogel	$Al(OH)_3$	d[a]	0.0001
calcium carbonate	chalk	$CaCO_3$	825 (d)[a]	0.0015
calcium chloride dihydrate	ice melter	$CaCl_2$	772	60
calcium hydroxide monohydrate	water softener	$Ca(OH)_2 \cdot H_2O$	580	0.185
calcium sulfate dihydrate	Gypsum	$CaSO_4 \cdot 2H_2O$	$-\frac{1}{2}H_2O$, 163[b]	0.3
lithium carbonate	anti manic-depression drug	Li_2CO_3	618	1.5
magnesium carbonate	Di-Gel	$MgCO_3$	350 (d)[a]	0.01
magnesium hydroxide	Milk of Magnesia	$Mg(OH)_2$	$-H_2O$, 350[b]	0.00009
magnesium sulfate heptahydrate	Epsom salt	$MgSO_4 \cdot 7H_2O$	$-6H_2O$, 150[b]	71
potassium aluminum sulfate dodecahydrate	alum	$KAl(SO_4)_2 \cdot 12H_2O$	92	11.4
potassium carbonate	potash	K_2CO_3	891	112
potassium chloride	salt substitute	KCl	776	35
potassium hydrogen carbonate	antacid	$KHCO_3$	100-200 (d)[a]	22
potassium sulfate	fertilizer	K_2SO_4	> 400	12
sodium hydrogen carbonate	baking soda	$NaHCO_3$	$-CO_2$, 270[c]	7
sodium carbonate decahydrate	washing soda	$Na_2CO_3 \cdot 10H_2O$	$-H_2O$, 32[b]	17
sodium chloride	table salt	NaCl	801	209
sodium sulfate decahydrate	Glauber's salt	$Na_2SO_4 \cdot 10H_2O$	884	5
sucrose	table sugar	$C_{12}H_{22}O_{11}$	186 (d)[a]	180

[a] The letter "d" indicates that the substance decomposes before it melts, often at a certain temperature.

[b] "$-H_2O$" indicates that the substance loses its waters of hydration at a certain temperature.

[c] "$-CO_2$" indicates that the substance decomposes, producing CO_2(g) at a certain temperature.