Spring 2012

A Cooperative Approach to the N-Solution Problem

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Approved April 12, 2012

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A Thesis

Presented to

The Academic Faculty

by

Sean B. Murray

An Honors Thesis

As part of

The Honor Scholars Program

And the Carroll College

Chemistry Department

Carroll College

April of 2012

Acknowledgements:

First of all, I would like to extend my thanks to Dr. Colin Thomas for his inspiration and guidance as my thesis director. I would also like to thank the Carroll College chemistry department for the use of both facilities and chemicals. The professors and students who participated in the trials deserve a big thank you as well. Their suggestions and troubleshooting comments were vital to the development of this procedure. Special thanks for Dr. Caroline Pharr and Dr. Thomas for allowing this procedure to be tested in their general chemistry labs.

Abstract:

Collaboration is an ever-present element in scientific research today. Even within laboratories and research teams, colleagues need to analyze, interpret, and disseminate research outcomes individually. Despite this need, the interdependence of scientists is seldom emphasized. We have added collaborative skill to the learning outcomes of a qualitative analysis laboratory exercise. Student collaboration, interdependence and enjoyment were measured using an anonymous survey and showed a marked increase over the control group in all areas. Presented here is the design with results from the lab protocol testing phase.

Introduction:

Learning outcomes commonly refer to developing students' ability to communicate, both orally and written, using discipline specific language and norms. A simple and practical way to develop these skills is to structure laboratory exercises to not only emphasize cooperation, but require it. Existing methods do not focus on improving students' ability to work interactively, that is, to determine and report data and make use of others' data in the interpretation of their own results. The laboratory setting provides a structured, yet interactive setting unrivaled for the teaching of valuable skills. A lab exercise commonly called the n-solution problem, or qualitative analysis, was altered to focus on valuable learning outcomes. Students will encounter three main challenges in the course of the procedure: 1- data interpretation, laboratory observation, and applied critical thinking, 2-interpersonal communication, and 3-spontaneous student self-organization. In this work we present a lab procedure that achieves all of these important aims.

Background:

The n-solution problem, a well-known qualitative laboratory exercise has been used in the chemistry education since 1940[1]. The original procedure required students to identify a set of unknowns using precipitation reactions, color changes, pH tests, scent, and known test reagents. For example, one variation requires students to mix their unknown solutions with only other unknowns[2], while another increases the number of unknowns, which lengthens the exercise to span several lab periods.

Common to these lab procedures is the ability of complete determination by a single experimenter. This focus on individuals is the area existing procedures could be modified to promote a wider skill set. While problem solving skills are a part of a general chemistry laboratory course, expanding the learning outcomes to include communication, collaboration, and interdependence greatly increases the students' abilities and better represents modern research environments. The procedure proposed in this work was specifically designed to accomplish these goals by allowing individual groups access to specific information.

Experimental:

To accomplish this cooperation, each group was not given all of the unknowns. The unknowns were separated into six unique combinations specifically designed to limit the information students are able to obtain on their own. This procedure used known test reagents, similar to published examples[1], but kept the number of unknowns to five, a realistic number for a three hour lab period. Of these five unknowns, only three were given to each group as well as one known test reagent, with which students could deduce the identity of some of their unknown cations and anions. The students started by testing their three unknowns with their test reagent, and then tested their three unknowns with each other. Using their observations, a solubility table, and the information provided by other groups' results they are able identify all five of the unknowns' cations and anions, a total of ten unknowns. To prohibit one student from determining all unknowns and distributing results, the solutions were not allowed to move from group to group. Each student was responsible for reporting all identities in the five unknown solutions. No one student can determine all of the unknown identities from what was given.

All the original benefits which made this exercise appealing are still there; the qualitative chemistry, the practice of safety procedure with unknown solutions, and the problem solving, but the alterations force the students to share the results of their experiments, which only then allow other groups to assign their unknowns. We want students to question each other, to disagree, to resort to redoing tests to clear up confusion.

The procedure was tested twice with a small group of fourth year chemistry majors and twice with general chemistry labs to determine whether the successful identification of all unknowns was possible, whether the cooperative goal was realistic, and whether the interaction between students would be self-generated.

The students in the large scale test were randomly separated into two groups. One performed our new procedure (appendix I) and the other did a control procedure (appendix II). These labs were separated and administered an anonymous survey to assess the extent of each students' reliance on others, willingness to participate, and overall enjoyment.

Results and Discussion:

The initial design tested in the first small scale trial did not allow for an unambiguous identification of ions. Because of this, a sixth combination was added to the procedure. The confusion arose from the anions of unknown #1 ($AgNO_3$) and #3 ($BaCl_2$). Without more information the reaction of unknown #1 with unknown #3, the white precipitate could be assigned as either AgCl or $BaSO_4$. The addition of $Ba(NO_3)_2$ as a test reagent in conjunction with unknowns #1, #3, and #5 allowed students to eliminate sulfate as #1's anion, thereby identifying the white precipitate of #1 and #3 as AgCl. A second test group performed the exercise with this revision and achieved a complete identification of all unknown solutions.

Throughout the large scale tests, there was a problem with unknown #2 (lead nitrate). Due to the lead chloride compound being somewhat soluble, reactions between lead nitrate and both barium chloride and sodium chloride solutions were not reliably yielding the precipitate $PbCl_{2(s)}$. To force the precipitate to form the chloride ion concentration was increased by adding sodium chloride to both unknown #3 and the sodium chloride test reagent solution.

Students in both the test and control group participated in an anonymous survey. They were asked whether they enjoyed the lab, how much they'd learned about using precipitation reactions to determine an unknown's identity, how much they learned about collaborating with others for a scientific goal, whether they were heavily reliant upon other groups, whether they were heavily relied on, and whether they were hindered by other groups' incorrect unknown identifications. The responses ranged from one to five; a five indicating a completely affirmative response and a one indicating a complete negative response. Table one contains the survey responses of both large scale tests, A and B.

Table 1: Anonymous survey results for experimental and control groups

	Experimental A	Control A	Experimental B	Control B
Lab was fun/enjoyable	3.7	2.6	4.5	3.2
I learned much about using precipitation reactions to identify unknowns.	3.9	3.7	4.4	4
I learned much about collaborating with others to accomplish scientific aims	4.7	2.7	4.9	3.2
I was heavily reliant upon other students correct assignment of unknowns	4	2.6	4.5	2.1
I was heavily relied upon to help others assign their unknowns	3.5	1.8	3.6	1.9
I was hindered by student's incorrect assignment of unknowns	2.1	2	3.3	1.6

The students in both of the large scale tests successfully determined the identity of all ions in solution. Students first tested their solutions and made observations, and then started wandering around to other groups, sharing information. When they realized this method was relatively unproductive, they spontaneously gathered the class into a single group. This massive group was at first chaotic, but in both large scale trials there were a few students who took initiative and attempted to direct the discussion. Encouragingly the survey results from students who participated in the new procedure's trials indicated a much higher level of cooperation than the control group. Group A results show a 4.7 for the collaboration question compared to a 2.7 on the control. Group B showed similar results (4.9:3.2). These differences are statistically significant at $p=3.1 \times 10^{-8}$. Students felt they contributed to the overall group effort, responding rather positively to the 'others relied heavily upon me' (3.5 and 3.6) survey question. These positive responses to the reliance questions indicate a higher level of individual and group accountability. It wasn't an accountability to a professor either, but to other students.

Students believed they learned as much or more about using precipitation reactions with the collaborative design than the control (3.9:3.7 for group A and 4.4:4.0 for group B). This data was statistically indistinguishable. This indicates the original qualitative analysis learning outcome was preserved. Both procedures seemed to effectively convey the content, with rather positive responses, however, students who were in the control group behaved markedly different from those who were not. They tended to stay at their stations; not moving around or asking questions. It was a much more individualistic approach as the survey shows, with a 2.7 and 3.2 response on the collaboration question for control group A and B respectively. They also tended to enjoy the control procedure less. The averages show a rather indifferent response (2.6 and 3.2) to control group, even slightly negative in group A, to the 'this lab was enjoyable' question.

Conclusion:

The groups in both large scale tests successfully identified all unknowns, indicating the procedure design was sound. Spontaneous, student-led groups formed and were focused for nearly an hour, leading heated discussions in all four test groups. These groups were extremely productive in both the small scale and large scale tests. The survey results show a larger degree of cooperation, personal accountability, and enjoyment when compared to the control group.

The proposed procedure clearly accomplished the learning outcomes it was designed for: communication, student self-organization, data interpretation, observational skills, and applied critical thinking. The qualitative analysis learning outcome of the control procedure was preserved as well. The procedure was met with widespread approval from all parties involved, both students and professors. It was both enjoyable and instructive and would an excellent addition to undergraduate chemistry courses.

Reference

1.MacWood, G. E.; Lassettre, E. N.; Breen, G., A laboratory experiment in general chemistry. *Journal of Chemical Education* **1940,** *17* (11), 520.

2.Zuehlke, R. W., The case of the unlabeled bottles: Descriptive chemistry in the introductory laboratory course. *Journal of Chemical Education* **1966,** *43* (11), 601.

3.Ryan, D. P.; Strothkamp, R. E., How to make the N-bottle problem more rigorous. *Journal of Chemical Education* **1993,** *70* (10), 850.

Appendix I

Qualitative Determination of Unknown Solutions

Purpose:

The goal in this lab is to determine the anions' and cations' of the unknowns by use of descriptive chemistry. Using your three unknowns and one given solution, you must glean what information you can on your own by reacting them with each other, and then confer with your associates to identify the rest. All the solutions must be identified, not just your own. Solutions cannot move from group to group, they must stay with the group they were originally assigned to.

Materials:

Three unknown solutions (Six possible combinations)
One given solution (Five possible) ($NaOH$, $NaCl$, $BaCl_2$, $Ba(NO_3)_2$ and Na_2CO_3)
3x4 spot plate
Plastic droppers

Procedure:

The reactions of the solution should be done in the spot plate, by placing a few drops of unknown from a test tube into a well using a dropper. React these drops with your known solution. Be careful not to cross contaminate reactions in adjacent wells and clean your plate thoroughly to avoid contamination. Do not use the same dropper for different solutions, have one for each.

These precipitation reactions will be easy to identify; keep in mind that a positive reaction will be a **precipitation** not merely a color change. You'll see a solid form and the liquid will become cloudy (as opposed to clear). Use the initial observation as opposed to one taken minutes later, as some reactions will discolor over time. Any reaction should be noted and used in conjunction with the solubility table to identify the cations and anions of that particular unknown.

Notice that the given solubility table has far more information than you will need. Part of your task will be to sort through the table and pick out what you will need to use. Look at the possible cations and anions to rule out any information that doesn't apply. Keep in mind that the smaller the K_{sp}, the more insoluble it is. It is important that you make sure the solutions are mixed well before you test them. A poorly mixed solution can lead to a false negative test.

Once you have reacted all possible combinations of unknowns and given solution, (should be six different possible combinations, including unknown-unknown) compare your results with those of others. You will not be able to fully identify your unknowns on our own. The five unknowns will be the same for all, and identified by number. Compare how each unknown reacts with others' solutions. It may help to categorize your information into, for instance, what you know, what you suspect, and what you know it can't be. Remember that a negative test is just as important as a positive, indicating what cannot be in an unknown. If there are disagreements among groups, it may help to do the test in question again. Be careful handling the unknown solutions as you don't know if they are harmful or not. Keep track of which unknowns reacted with which. Once all of the unknowns are successfully identified (as a class), lab is over. Groups are not allowed to leave even after identifying their ions, help others figure theirs out.

Possible Unknown Cations and Anions

Ag^+	
Pb^{2+}	NO_3^-
Ba^{2+}	Cl^-
Cu^{2+}	SO_4^{2-}
Na^+	

Groups

1. Unknowns #1, #2, and #3 and a known 0.1M NaCl
2. Unknowns #2, #3, and #4 and a known 0.1M Na_2CO_3
3. Unknowns #1, #3, and #5 and a known 0.1M $BaCl_2$
4. Unknowns #1, #2, and #4 and a known 3.0M KOH
5. Unknowns #3, #4, and #5 and a known 3.0M KOH
6. Unknowns #1, #3, and #5 and a known 0.1M $Ba(NO_3)_2$

		K_{sp}
Br^-		
Ag^+	pale yellow, curdy	5.0×10^{-13}
Hg^{2+}	white	8.0×10^{-20}
Pb^{2+}	white	4.0×10^{-5}
Cl^-		
Ag^+	white	1.8×10^{-10}
Pb^{2+}	white	1.7×10^{-5}
I^-		
Ag^+	pale yellow	8.3×10^{-17}
Hg^{2+}	bright orange-red	3.2×10^{-29}
Pb^{2+}	bright yellow	7.1×10^{-9}
Cu^{2+}	light brown	
S^{2-}		
Ag^+	black	6×10^{-51}
Cu^{2+}	black	6×10^{-37}
Fe^{3+}	black	
Hg^{2+}	black	2×10^{-53}
Ni^{2+}	black	3×10^{-20}
Pb^{2+}	black	3×10^{-28}
Zn^{2+}	white	2×10^{-25}

		K_{sp}
OH^-		
Ag^+	gray-brown suspension	2×10^{-8}
Cu^{2+}	pale blue	4.8×10^{-20}
Fe^{3+}	red, rust	4×10^{-38}
Hg^{2+}	yellow with excess OH^-	
Ni^{2+}	pale green, gelantinous	6.0×10^{-16}
Pb^{2+}	white	1.2×10^{-15}
Zn^{2+}	white	3.0×10^{-16}
CO_3^{2-}		
Ag^+	white	8.1×10^{-12}
Ba^{2+}	white	5.0×10^{-9}
Ca^{2+}	white	4.5×10^{-9}
Cu^{2+}	light blue	2.3×10^{-10}
Fe^{3+}	orange brown	
Hg^{2+}	dark red-orange	
Ni^{2+}	pale green	1.3×10^{-7}
Pb^{2+}	white	7.4×10^{-14}
Zn^{2+}	white	1.0×10^{-10}
PO_4^{3-}		
Ag^+	light yellow	2.6×10^{-18}
Ba^{2+}	white	
Ca^{2+}	white	2.0×10^{-29}
Hg^{2+}	white	
Pb^{2+}	white	1.5×10^{-32}
Zn^{2+}	white	9.0×10^{-33}
SO_4^{2-}		
Ba^{2+}	white	1.1×10^{-10}
Pb^{2+}	white	6.3×10^{-7}

Correct unknown assignments for the qualitative analysis lab.

Unknown #1-$AgNO_3$

Unknown #2-$Pb(NO_3)_2$

Unknown #3-$BaCl_2$

Unknown #4-$CuNO_3$

Unknown #5-$BaSO_4$

Appendix II

The n-solution Problem

Purpose:

Perform a qualitative analysis using the concepts of equilibrium, solubility products and spectator ions.

CAUTION: Treat all solutions as dangerous. Some are corrosive, some are health hazards and you don't know which is which.

Procedure:

The premise of this problem is that you have ten bottles without labels. You know which ions are present, but not which compounds. You must identify what is in each bottle by using only pH paper and the reactions of the ten solutions with each other.

This experiment is similar to an earlier one that you did with 5 unknown solutions. This time take about 5 ml of each solution in ten small test tubes. Use droppers to use a few drops of solution for each test. When testing pH, use a stirring rod, dropper or cork to put a drop on the pH paper. Do not dip the pH paper into your solution. It could contaminate the solution.

Remember to check for gases and odors. Ammonia has a familiar smell. hydrogen sulfide, H_2S, smells like rotten eggs. A sulfide will give off $H_2S(g)$ when mixed with an acid. A carbonate will give off $CO_2(g)$ when mixed with an acid.

You may find it useful to start with a 10 x 9 grid plus a row for pH. Fill in what happens when two solutions are mixed. One approach is to test every solution with every other solution, and then sit down and think about it. Another approach is to think about each test and try to perform the minimum number of tests to identify each solution.

Some ions are present twice. There is only one sulfate. The unknowns were chosen so that no combination of two solutions will give *two* precipitates. There will either be one precipitate or none.

Keep your spot plate clean and rinse your droppers with a wash bottle when switching between solutions. Weak tests usually mean contamination, so consider a weak test as a negative test.

Cations	Anions	Other
Ag^+	Cl^-	H_2O
Ba^{2+}	I^-	NH_3
Cu^{2+}	NO_3^-	
H^+	OH^-	
Hg^{2+}	S^{2-}	
Na^+	SO_4^{2-}	