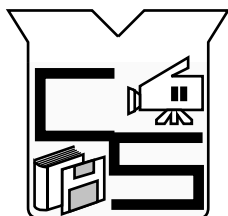
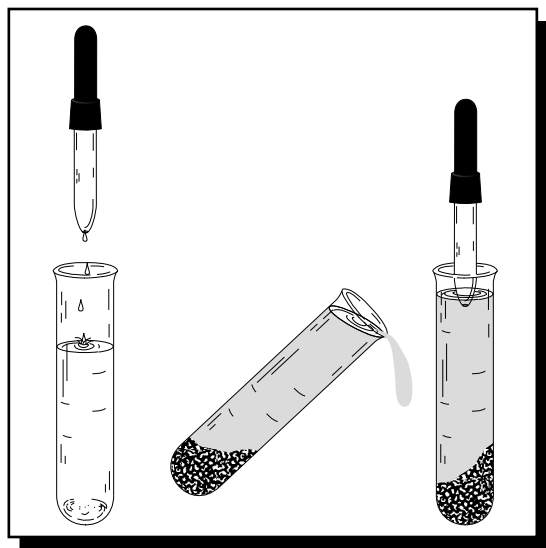


A SourceBook Module

Version 1.0 1994

*Funded in part under
National Science Foundation
Grant No. TPE 88-50632*

*ChemSource Project Principal Investigator:
Mary Virginia Orna, OSU
Department of Chemistry
College of New Rochelle
New Rochelle, NY 10805
Phone: (914) 654-5302
FAX: (914) 654-5387*

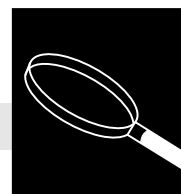


ChemSource

*Instructional Resources for Preservice and
Inservice Chemistry Teachers*

**INORGANIC QUALITATIVE
ANALYSIS**

Topic Overview



CONTENT IN A NUTSHELL

From the crime lab to the water treatment plant, from environmental testing to agricultural soil testing to environmental quality determination, society asks chemistry to analyze materials and determine what and how much is contained in them.

Analysis is one of the central branches of chemistry. It is conveniently divided into qualitative analysis, which concentrates on what materials are present in a sample, and quantitative analysis, which is concerned with how much of a substance is present in a sample. In practice, qualitative and quantitative methods go hand-in-hand.

For many reasons, inorganic qualitative analysis laboratory has almost disappeared from the curriculum. In the first place, the modern practice of chemical analysis is being done with instrumentation, and more traditional “wet” chemistry methods might be considered old fashioned. Secondly, traditional qualitative analysis schemes of the metal ions were carried out using H_2S gas, which is noxious and toxic. Even with the substitution of thioacetamide as a source of sulfide, the odor of the qualitative analysis laboratory gave chemistry buildings their characteristic “stink.” Finally, qualitative analysis schemes were so complicated that the scale of the laboratory required it to be taught over a period of one to two semesters. Since it became increasingly difficult to coordinate this laboratory with a concept-centered lecture course, it became necessary to teach qualitative analysis as a stand-alone course. As the undergraduate chemistry curriculum has become more loaded, it becomes less practical to devote so much time to this subject.

Nevertheless, qualitative analysis has many defenders who point out its value and argue for its place in the chemistry curriculum. First of all, qualitative analysis schemes are an excellent source of descriptive chemistry information. As beginning chemistry courses are becoming more principle oriented, many chemists are concerned that chemistry students don't have a basic repertoire of reaction chemistry that illustrates chemical concepts. Secondly, the development and use of qualitative analysis schemes require the kind of logical reasoning that is sometimes used as a justification for science courses. Finally, qualitative analysis schemes allow the development or review of such topics as solubility product equilibria, kinetics, acid/base chemistry, and coordination complexes. For these reasons, inorganic qualitative analysis has undergone a resurgence in recent years.

In order to answer the safety criticisms leveled at the use of qualitative analysis, alternative analysis schemes have been developed using benzoate, hydroxide, and phthalate anions. There has also been an effort to “scale-down” the amounts of noxious chemicals using semimicro, and in recent years, small-scale techniques. If the users of *SourceBook* want to carry out an extended qualitative analysis instruction using a traditional or more modern metal ion scheme, references can be found at the end of this module.

This module takes the view that qualitative analysis can play a useful role in the high school chemistry curriculum if it meets several criteria. (1) Qualitative analysis should be limited in scope so that it can be taught as a module or part of other modules. (2) Laboratory activities should be done with small amounts of chemicals and involve chemicals that can be handled safely. (3) The emphasis of a qualitative analysis scheme should be on the development of the scheme and its logic rather than exclusively on the use of a scheme developed by others.

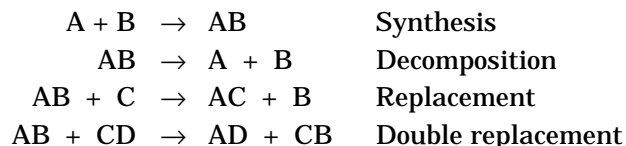
As envisioned here, this module could be used as a review of chemical ideas developed during a high school chemistry course. As such it should be used at the end of a one-year course in chemistry, or during a second-year course. Many of the individual activities could also be used to supplement other modules. For example, some of the reactions in the qualitative analysis laboratory could be used to illustrate solubility and acid/base equilibrium. This module could also be used as an opportunity for students to develop and practice the logical processes used in chemistry.

PLACE IN THE CURRICULUM

1. Chemical substances can be identified (specified) from their chemical and physical characteristics and interactions.
2. A scheme or sequence of procedures can be used to identify the presence or absence of a chemical substance in combination with others.

CENTRAL CONCEPTS

1. Acids and bases are chemical species that exhibit distinctive sets of observable properties.
2. Electrons occupy specific energy levels in an atom. These energy levels are quantized and are responsible for emission spectra.
3. Reaction rates are influenced by factors such as the nature of the reactants, presence of a suitable catalyst, temperature, concentration of the reactants, and available surface area of solid or liquid reactants, or pressure for gases.
4. Many chemical reactions can be classified, at the simplest level, according to general kinds of change involved:



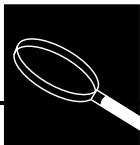
Many chemical reactions involve the transfer of electrons between reacting chemical species.

5. When a solute is only slightly soluble, the extent of solubility can be quantitatively expressed as the solubility product constant.
6. The solubility of a substance can be altered by common ion effect, pH, temperature change, formation of a complex, *etc.*
7. Accumulated data allow one to predict the degree of solubility of many substances, *i.e.*, solubility rules.

RELATED CONCEPTS

1. Name simple ionic compounds.
2. Use flame tests to identify active metals.
3. Use indicators to determine the pH of solutions.
4. Write balanced chemical equations for chemical reactions.
5. Hypothesize chemical reactions from reaction types.
6. Use a centrifuge to separate a precipitate from a solution.
7. Use basic separation techniques.
8. Utilize solubility rules.
9. Utilize basic chemical tests to identify common gases and other chemicals.

RELATED SKILLS



**PERFORMANCE
OBJECTIVES**

After completing their study of qualitative analysis, students should be able to:

1. Analyze a sample for the presence of a specific ion, given testing procedures.
2. Develop an analysis scheme for a set of ionic substances.
3. Use comparative reaction information to identify a precipitate.



Concept/Skills Development

Activity 1: Qualitative Analysis Laboratory I: Developing a Qualitative Analysis Scheme for the Cations Zn^{2+} , Ca^{2+} , Cu^{2+} , Al^{3+} , Co^{2+}

LABORATORY ACTIVITY: STUDENT VERSION

Introduction

Solutions often contain unknown ions. Fortunately, patterns of chemical reactivity and solubility allow for the isolation and identification of ions in a solution. This identification activity is called qualitative analysis. In this activity you will devise your own identification scheme for five cations.



Purpose

To study some of the properties and patterns of reactivity that allow for the separation and identification of certain ions in a solution.

Safety

1. Wear protective goggles throughout the laboratory activity.
2. Be careful to avoid contaminating the test solutions and stock solutions.

Procedure

Obtain approximately 10 mL of 0.1 M solutions Zn^{2+} , Ca^{2+} , Cu^{2+} , Al^{3+} and Co^{2+} in separate clean, dropper bottles. (Each of these solutions was prepared from the nitrate salt of the cation.) You are to subject each of these solutions to the tests outlined below and record your observations in the table provided.

Tests

1. Put 10 drops test solution (Zn^{2+}) into a 75-mm test-tube. Using pH paper, test the pH. If needed, add 3 M NH_3 or 3 M HNO_3 dropwise until the pH is neutral, *i.e.*, between 6 and 8. Now add 7 drops 0.3 M Na_3PO_4 .
2. To 10 drops fresh test solution (Zn^{2+}) add 3 M HNO_3 until the solution is strongly acidic, *i.e.*, $pH < 3$. Add 7 drops 0.2 M $K_3Fe(CN)_6$, potassium ferricyanide.
3. To 10 drops fresh test solution (Zn^{2+}) add 6 M $NaOH$ until the solution is just basic, *i.e.*, $pH > 8$. Add 10 drops 6 M $NaOH$. If a persistent precipitate forms, mix the sample and centrifuge. Decant the solution and save any precipitate formed.
4. Wash the precipitate twice with about 5 mL of distilled water. Add 10 drops 6 M NH_3 and mix thoroughly, then centrifuge for 1 min. Observe.
5. To 10 drops of fresh test solution (Zn^{2+}) add 6 M $NaOH$ until the solution is just basic. Add 10 drops $NaOH$. Mix, then centrifuge if a precipitate develops. Decant. If there is a precipitate, wash it twice with distilled water and centrifuge. Add 15 drops 3 M HNO_3 to the precipitate and mix.

Write your observations (precipitates, colors) in the table that follows.

6. Repeat Steps 1-5 for the remaining 4 cations.
7. Thoroughly wash your hands before leaving the laboratory.

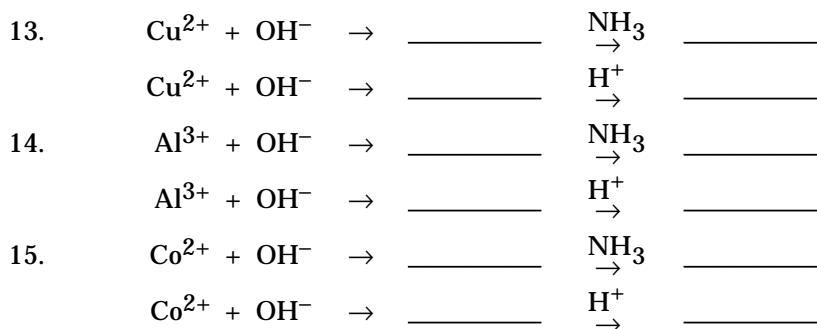


Reagent	Cation				
	Zn ²⁺	Ca ²⁺	Cu ²⁺	Al ³⁺	Co ²⁺
Test 1 PO ₄ ³⁻					
Test 2 Fe(CN) ₆ ³⁻					
Test 3 OH ⁻					
Test 4 NH ₃					
Test 5 H ⁺					

Data Analysis and Concept Development

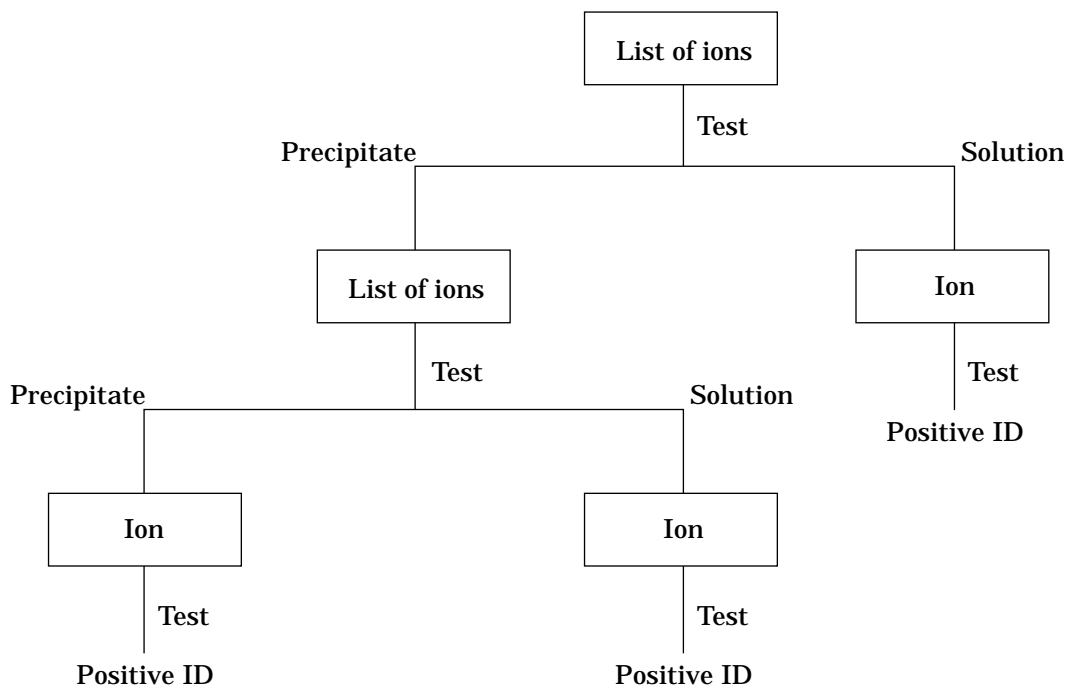
From your data complete the equations listed below. You will be speculating, but your speculation will be accurate in 90% of the cases if you use the following guidelines. (1) Assume that most reactions are a simple combination of cations and anions. (2) If the product is dissolved, it is ionic. (3) If the product is solid, it is neutral. If you observed no change when the particular reagents were mixed, write N.R. for “no reaction.”

- Zn²⁺ + PO₄³⁻ → _____
- Ca²⁺ + PO₄³⁻ → _____
- Cu²⁺ + PO₄³⁻ → _____
- Al³⁺ + PO₄³⁻ → _____
- Co²⁺ + PO₄³⁻ → _____
- Zn²⁺ + Fe(CN)₆³⁻ → _____
- Ca²⁺ + Fe(CN)₆³⁻ → _____
- Cu²⁺ + Fe(CN)₆³⁻ → _____
- Al³⁺ + Fe(CN)₆³⁻ → _____
- Co²⁺ + Fe(CN)₆³⁻ → _____
- Zn²⁺ + OH⁻ → _____ $\xrightarrow{\text{NH}_3}$ _____
 Zn²⁺ + OH⁻ → _____ $\xrightarrow{\text{H}^+}$ _____
- Ca²⁺ + OH⁻ → _____ $\xrightarrow{\text{NH}_3}$ _____
 Ca²⁺ + OH⁻ → _____ $\xrightarrow{\text{H}^+}$ _____



Implications and Applications

1. Assume you were given a solution that was said to contain Ca^{2+} cations only. Describe how you would confirm or deny the presence of this cation in the solution. Explain your reasoning.
2. Assume you were given a solution that was said to contain Zn^{2+} and/or Al^{3+} cations only. Describe how you would determine if neither, only one or both of these ions were present in solution. Explain your reasoning.
3. Assume you were given a solution that was said to contain Cu^{2+} and/or Co^{2+} cations. Describe how you would determine if neither, only one or both of these ions were present in solution.
4. Assume you were given a solution that was said to contain Zn^{2+} , Ca^{2+} , Cu^{2+} , Al^{3+} , and/or Co^{2+} cations only. Describe how you would confirm or deny the presence of each of these ions in the solution. Explain your reasoning. (Hint: You are encouraged to design an analysis scheme—a flow chart—such as the one shown in the following example.)





**LABORATORY
ACTIVITY:
TEACHER
NOTES**

***Activity 1: Qualitative Analysis Laboratory I:
Developing a Qualitative Analysis Scheme
for the Cations Zn^{2+} , Ca^{2+} , Cu^{2+} , Al^{3+} , Co^{2+}***

NOTE: A more guided alternative to the suggested laboratory can be found in Dreyfus Microscale Lab: Qualitative Analysis #36 (Chen & Ingels, 1987).

Major Chemical Concept

Cations in solution can be separated and identified by characteristic chemical changes. These changes can be observed by precipitate formation, color change, or formation of gases.

Level

General and Honors

Expected Student Background

1. Experience with observational evidence for chemical change.
2. Experience in chemical formula writing and knowledge of common ion charges.
3. Ability to balance chemical equations.
4. Some knowledge of solubility patterns for selected compounds. (A list of “solubility rules” can be found in most basic chemistry books.)

Time

The first set of tests will require two or three 50-min laboratory periods. The *Data Analysis and Concept Development* section can be assigned as homework. *Implications and Applications* will require one or two periods.

Safety

Read the *Safety Considerations* in the *Student Version*.

1. Students should be cautioned that the solutions are toxic. Some are caustic and should be handled with care.
2. Students should wash their hands prior to leaving the laboratory.
3. They should also wash their hands if their skin feels “slippery.” This sensation indicates they have come in contact with NaOH.
4. Safety goggles should be worn while performing these activities.

Materials (For 24 students working in pairs)

Nonconsumables

- 12 Test-tube racks
- 120 Test-tubes, 75-mm
- 72 Medicine droppers
- 60 Stirring rods
- 2 Vials universal pH paper
- 4 Centrifuges
- 5 Dropper bottles per group

Consumables

- 3 M Ammonia, NH_3 , 150 mL [30 mL 15 M (conc.) NH_3 per 150 mL solution]
3 M Nitric acid, HNO_3 , 250 mL [47 mL 16 M (conc.) HNO_3 per 250 mL solution]
0.2 M Potassium ferricyanide, $\text{K}_3\text{Fe}(\text{CN})_6$, 150 mL (9.9 g $\text{K}_3\text{Fe}(\text{CN})_6$ per 150 mL solution) *NOTE:* $\text{K}_3\text{Fe}(\text{CN})_6$ is called potassium ferricyanide—not ferrocyanide. The correct name for this compound is potassium hexacyanoferrate(III).
0.3 M Trisodium phosphate, Na_3PO_4 , 150 mL (17.1 g $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$) per 150 mL solution)
6 M Sodium hydroxide, NaOH , 150 mL (36 g NaOH per 150 mL solution)
6 M Ammonia, NH_3 , 150 mL (60 mL 15 M (conc.) concentrated NH_3 per 150 mL solution)
0.1 M Zinc nitrate, $\text{Zn}(\text{NO}_3)_2$, 150 mL (4.5 g $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ per 150 mL solution)
0.1 M Calcium nitrate, $\text{Ca}(\text{NO}_3)_2$, 150 mL (3.5 g $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ per 150 mL solution)
0.1 M Copper(II) nitrate, $\text{Cu}(\text{NO}_3)_2$, 150 mL (3.6 g $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ per 150 mL solution)
0.1 M Aluminum nitrate, $\text{Al}(\text{NO}_3)_3$, 150 mL (5.6 g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ per 150 mL 0.01 M HNO_3 solution) [0.01 M HNO_3 (0.5 mL 3 M HNO_3 per 150 mL solution).]
0.1 M Cobalt(II) nitrate, $\text{Co}(\text{NO}_3)_2$, 150 mL (4.4 g $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ per 150 mL solution)
pH Paper Range 0-13

Advance Preparation

1. Sets of solutions of each reagent should be prepared and placed in labeled dropper bottles in convenient locations in the laboratory. Students should be advised to return the dropper to its bottle immediately after using it. This will avoid contamination of the reagents.
2. The reading on semi-micro laboratory techniques (included in the *Appendix*) should be reproduced and given to students to read prior to the first laboratory period. The procedure assumes that students understand these techniques.

Pre-Laboratory Discussion

1. Review and answer student questions on the semi-micro techniques reading. Students should use the reading for reference as needed, while they are performing the procedures.
2. Review the safety precautions to be used in the laboratory.
3. Stress the importance of and procedures for keeping reagent samples and stock bottles uncontaminated.
4. Instruct students on safe disposal of wastes from the activities. It is suggested that the liquid wastes be collected in a large, centrally located bottle.

(Consult section on *Waste Disposal* in *Appendix*.)

Teacher-Student Interaction

1. Students will start slowly and increase in speed with experience. Observe their procedures and correct them if the techniques are sloppy or leading to contamination of stock reagents or samples.



2. Examine student observation sheets to insure that their observations are consistent with normal expectations. If they have aberrant observations suggest that they repeat the test. Do not tell them the correct answers.
3. You might want to divide this laboratory into parts. Have students do some of the tests and then hold a discussion on their observations. Pool their data and discuss writing equations. You might also use these discussions to explore the chemistry of these materials. Use the *Notes on Tests and Equations* in the *Answers to Data Analysis* section as a basis for discussion of student observations. Be prepared to rerun a test as a demonstration.

Anticipated Student Results

Reagent	Cation				
	Zn ²⁺	Ca ²⁺	Cu ²⁺	Al ³⁺	Co ²⁺
Test 1 PO ₄ ³⁻	White ppt	White ppt	Blue ppt	White ppt	Purple ppt
Test 2 Fe(CN) ₆ ³⁻	Yellow ppt		Yellow-green ppt		Red ppt
Test 3 OH ⁻	Initial ppt disappears	White ppt, but disappears by 2nd wash	Blue ppt	Initial ppt disappears	Blue ppt fading to pink
Test 4 NH ₃			ppt dissolves Dark blue solution		ppt remains
Test 5 H ⁺			ppt dissolves Pale green solution		ppt dissolves Pink solution

Answers to Data Analysis and Concept Development

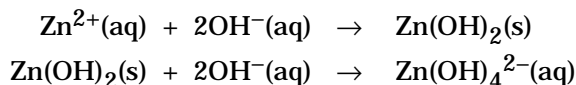
Equations

1. $3\text{Zn}^{2+}(\text{aq}) + 2\text{PO}_4^{3-}(\text{aq}) \rightarrow \text{Zn}_3(\text{PO}_4)_2(\text{s})$
2. $3\text{Ca}^{2+}(\text{aq}) + 2\text{PO}_4^{3-}(\text{aq}) \rightarrow \text{Ca}_3(\text{PO}_4)_2(\text{s})$
3. $3\text{Cu}^{2+}(\text{aq}) + 2\text{PO}_4^{3-}(\text{aq}) \rightarrow \text{Cu}_3(\text{PO}_4)_2(\text{s})$
4. $\text{Al}^{3+}(\text{aq}) + \text{PO}_4^{3-}(\text{aq}) \rightarrow \text{AlPO}_4(\text{s})$
5. $3\text{Co}^{2+}(\text{aq}) + 2\text{PO}_4^{3-}(\text{aq}) \rightarrow \text{Co}_3(\text{PO}_4)_2(\text{s})$
6. $3\text{Zn}^{2+}(\text{aq}) + 2\text{Fe}(\text{CN})_6^{3-}(\text{aq}) \rightarrow \text{Zn}_3[\text{Fe}(\text{CN})_6]_2(\text{s})$
7. $\text{Ca}^{2+}(\text{aq}) + \text{Fe}(\text{CN})_6^{3-}(\text{aq}) \rightarrow \text{N.R.}$
8. $3\text{Cu}^{2+}(\text{aq}) + 2\text{Fe}(\text{CN})_6^{3-}(\text{aq}) \rightarrow \text{Cu}_3[\text{Fe}(\text{CN})_6]_2(\text{s})$
9. $\text{Al}^{3+}(\text{aq}) + \text{Fe}(\text{CN})_6^{3-}(\text{aq}) \rightarrow \text{N.R.}$
10. $3\text{Co}^{2+}(\text{aq}) + 2\text{Fe}(\text{CN})_6^{3-}(\text{aq}) \rightarrow \text{Co}_3[\text{Fe}(\text{CN})_6]_2(\text{s})$

11. $\text{Zn}^{2+}(\text{aq}) + \text{OH}^{-}(\text{aq}) \rightarrow \text{N.R.} \xrightarrow{\text{NH}_3} \text{N.R.}$
 $\text{Zn}^{2+}(\text{aq}) + \text{OH}^{-}(\text{aq}) \rightarrow \text{N.R.} \xrightarrow{\text{H}^{+}} \text{N.R.}$
12. $\text{Ca}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{N.R. or Ca(OH)}_2(\text{s}) \xrightarrow{\text{H}_2\text{O}} \text{Ca}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq})$
 $\text{Ca}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{N.R. or Ca(OH)}_2(\text{s}) \xrightarrow{\text{H}^{+}} \text{Ca}^{2+}(\text{aq})$
13. $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Cu(OH)}_2(\text{s}) \xrightarrow{\text{NH}_3} \text{Cu(NH}_3)_4^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq})$
 $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Cu(OH)}_2(\text{s}) \xrightarrow{\text{H}^{+}} \text{Cu}^{2+}(\text{aq}) + 2\text{H}_2\text{O}(\text{l})$
14. $\text{Al}^{3+}(\text{aq}) + \text{OH}^{-}(\text{aq}) \rightarrow \text{N.R.} \xrightarrow{\text{NH}_3} \text{N.R.}$
 $\text{Al}^{3+}(\text{aq}) + \text{OH}^{-}(\text{aq}) \rightarrow \text{N.R.} \xrightarrow{\text{H}^{+}} \text{N.R.}$
15. $\text{Co}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Co(OH)}_2(\text{s}) \xrightarrow{\text{NH}_3} \text{N.R.}$
 $\text{Co}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Co(OH)}_2(\text{s}) \xrightarrow{\text{H}^{+}} \text{Co}^{2+}(\text{aq}) + 2\text{H}_2\text{O}$

Notes on the Tests and Equations

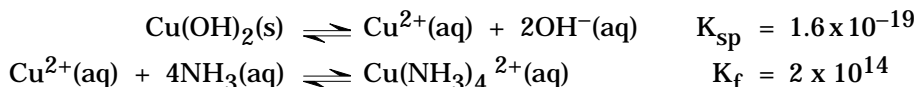
- Students may point out that most solubility rules would indicate that Zn^{2+} and Al^{3+} form insoluble hydroxides. Some may have noticed precipitates appearing when OH^{-} is added. This precipitate disappears when additional OH^{-} is added. Both Zn(OH)_2 and Al(OH)_3 are amphoteric (*i.e.*, they are soluble in both strong base and strong acid). They form soluble ions when an excess of OH^{-} is present. For example:



In a similar manner Al(OH)_4^{-} is formed. Students will probably not be able to write an equation for the complex ion formation and will merely indicate no reaction since that is consistent with their observations. The point of the test is that it allows the separation of Zn^{2+} and Al^{3+} from the other ions. Whether or not you discuss complex formation by these ions depends upon the background of students.

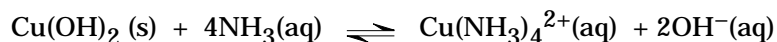
- The Ca(OH)_2 precipitate dissolves in the washings because although it is “insoluble,” it is relatively more soluble than most “insoluble” substances. ($K_{\text{sp}} = 5.5 \times 10^{-6}$)
- The Cu(OH)_2 precipitate is difficult to see until the solution is centrifuged. The green solution formed when the Cu(OH)_2 is acidified is very pale. Most students will probably observe no color. The dark blue solution formed when the ammonia is added to the Cu(OH)_2 is due to the formation of the complex ion $\text{Cu(NH}_3)_4^{2+}$. Students are unlikely to write the correct formula for the tetraammine copper complex, and might just write CuNH_3^{2+} . It is probably not important that they write the correct formula, although it does not do any harm just to tell them the correct formula.

The equations for the competing equilibria follow:





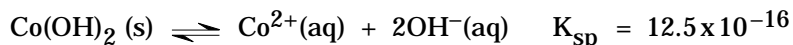
Because the equilibrium constants are close to offsetting each other, the combined reaction:



will dissolve the insoluble Cu(OH)_2 with a small excess of NH_3 . Similarly acid will dissolve the precipitate:



4. Co(OH)_2 can exist as either a blue or a pink precipitate. The blue form is believed to be more finely dispersed. It changes to the coarser pink form that is more stable. Air will oxidize Co(OH)_2 to Co(OH)_3 , which is a black/brown precipitate. If students do their cobalt ion tests during the same period, using fresh precipitates, the presence of Co(OH)_3 can be ignored. As with copper ions, it is possible to form the amine complex of cobalt ions. The equations follow:



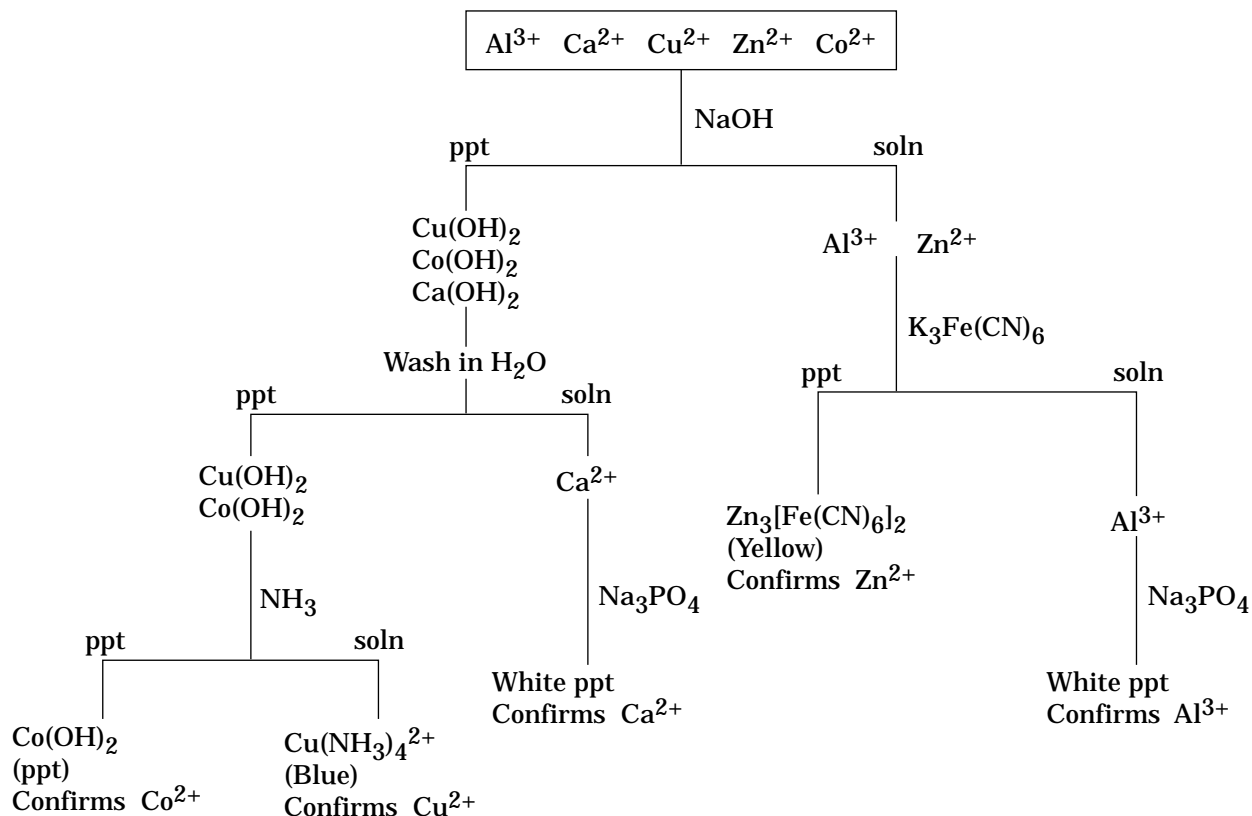
Because the relative sizes of the equilibrium constants favor the formation of insoluble Co(OH)_2 over $\text{Co(NH}_3)_4^{2+}$, the precipitate will not dissolve unless large excesses of ammonia are present. Thus, in contrast with Cu(OH)_2 , Co(OH)_2 does not dissolve. This gives a basis for separating and identifying Cu^{2+} and Co^{2+} .

5. Adjusting the pH of solution is not much of a problem in doing these tests. However, if an unknown or a combination of ions used to try out separations is used, pH control becomes more critical. If the 0.3 M solutions are too concentrated to make the pH adjustments, suggest that students make dilute solutions for this purpose.

Answers to Implications and Applications

1. Add NaOH to the solution, and, if Ca^{2+} is present, a white precipitate will form. Wash the precipitate, save the solution liquid, and add Na_3PO_4 to the solution liquid. The formation of another white precipitate confirms the presence of Ca^{2+} .
2. Add potassium ferricyanide and nitric acid solutions to the solution. A yellow precipitate confirms the presence of Zn^{2+} . Centrifuge and decant the solution liquid into another test-tube, and add Na_3PO_4 solution. If a white precipitate forms, Al^{3+} is confirmed. If both of these tests produce the expected precipitate, then both ions are present.
3. Addition of NaOH to this solution will result in the formation of precipitates of both Co(OH)_2 and Cu(OH)_2 . Cu(OH)_2 is soluble in concentrated NH_3 , and the $\text{Cu(NH}_3)_4^{2+}$ ion forms a dark blue solution. If all the precipitate dissolves, then Co^{2+} was not present. If the Cu(OH)_2 dissolves and there is a dark precipitate remaining, then the presence of Co^{2+} is confirmed by that Co(OH)_2 precipitate.
4. See the following flow chart.

Alternatives: Can dissolve Co(OH)_2 in acid and confirm with $\text{K}_3\text{Fe(CN)}_6$ or Na_3PO_4 . Can use a portion of the unwashed NaOH precipitate to look for Ca^{2+} by dissolving in acid, precipitating Cu^{2+} and Co^{2+} with $\text{K}_3\text{Fe(CN)}_6$ and then using Na_3PO_4 to look for Ca^{2+} .



Post-Laboratory Discussion

Discuss the reactions involved in the various tests with the class. Review the answers to the questions by having students submit their answers to the first three. Other ions may be the subject of similar questions raised to insure understanding of the chemical reactions and the laboratory techniques.

Extensions

While you may wish to include the analysis of an unknown as a learning assessment activity, such an activity could be used to support the knowledge gained from the laboratory work. The activity is included here.

Analysis of an Unknown

You will be given 10 mL of an unknown solution that might contain any or all of the cations: Zn^{2+} , Ca^{2+} , Cu^{2+} , Al^{3+} , and Co^{2+} . You are to analyze your solution in whatever manner you feel is appropriate to determine unequivocally which of the cations are present.

The laboratory report for this section should include the following:

1. **An experimental section.** This should be a step-by-step accounting of the activities conducted and the results observed. Include balanced ionic equations where appropriate.
2. **A conclusion section.** This should be a paragraph telling which cations you think were in your solution and telling why you reached this conclusion.



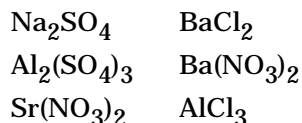
Notes to the Teacher

1. Use 0.15 M Al^{3+} in unknowns. The Al^{3+} test in our suggested Flow Chart is weak indicating that some Al^{3+} previously precipitated with NaOH. Make sure the unknown solutions are 0.1 M in each of the other metal ions used. Don't merely mix the test solutions since the concentrations will be too weak.
2. pH control called for in the tests is essential. Students should be reminded of this especially when doing the unknown.

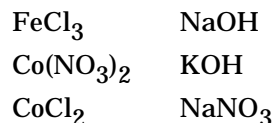
Prediction and Naming of Precipitates

Following are two sets of solutions. Each set contains six solutions. One of these sets will be assigned to you. Mix small amounts of each sample with every other sample and observe any evidence of a chemical reaction. How many possible combinations of two solutions are possible? Write balanced equations for any chemical reactions you observe. Identify any precipitate formed.

Set 1



Set 2



Notes to the Teacher

Another laboratory activity, which can enhance the learning regarding prediction and naming of precipitates, involves combining of solutions of known compounds. Two sets of chemicals are offered as suggestions. Many other sets are possible. 0.1 M solutions of each substance should be provided in dropper bottles. Students might make a 6 x 6 grid for recording information about reaction results. Set 1 is used as an example. [A grid similar to the one below (without the expected results) can be photocopied and overlaid with transparent food wrap such as Saran™ wrap. A drop of each reagent in the vertical column can be placed on the appropriate squares in the rows, and then tested with a drop of appropriate reagent from the set along the top of the chart. This procedure is quick, easy to accomplish, and easy to interpret.]

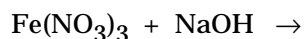
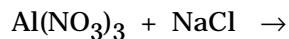
	Na_2SO_4	$\text{Al}_2(\text{SO}_4)_3$	$\text{Sr}(\text{NO}_3)_2$	BaCl_2	$\text{Ba}(\text{NO}_3)_2$	AlCl_3
Na_2SO_4	_____		White ppt	White ppt	White ppt	
$\text{Al}_2(\text{SO}_4)_3$		_____	White ppt	White ppt	White ppt	
$\text{Sr}(\text{NO}_3)_2$	White ppt	White ppt	_____			
BaCl_2	White ppt	White ppt		_____		
$\text{Ba}(\text{NO}_3)_2$	White ppt	White ppt			_____	
AlCl_3						_____

Assessing Laboratory Learning

A laboratory practical to identify ions in an unknown solution is an effective way to assess learning (see *Extensions*).

Questions

1. Based on your laboratory experience, write balanced equations for each of the following pairs of reactants. Assume that chemical changes occur with each set of reactants.



2. In which of the reactions in Question 1 would you be able to observe the formation of a precipitate?

A more challenging activity consists of providing students with a set of solutions labeled A, B, C, *etc.* Students are given the formulas of the chemicals in the solution and told to match the formula to the solution. Students make observations of reactions that occur when the solutions are mixed together. Many versions of this exercise exist—*e.g.*, Dreyfus Microscale Lab: Six Solutions #35 (Chen & Engels, 1987), but teachers can make up their own. Provide students with a solubility rules table and a table of common chemical tests (see *Appendix*).

CAUTION: Use appropriate safety guidelines in performing demonstrations.

DEMONSTRATIONS

Demonstration 1: Flame Tests

Description

Cations can be identified by characteristic colors, when a compound containing the cation is heated in a burner flame. The purpose of this demonstration is to show the colors and to associate them with the cations in the compounds.

Materials

Use samples of BaCl_2 , CaCl_2 , CuCl_2 , SrCl_2 , NaCl , KCl .

Procedure

Details of setting up and carrying out of a flame test demonstration can be found in the *Alkali Metals* module.

Remarks

It would be appropriate to review the demonstration by testing “unknown” solutions and asking students to identify the cation present because of the color. Explain that these flame tests provide a method for qualitative analysis for cations, if known samples containing that cation can be obtained and tested.

For reference, expected colors of the cations used in this demonstration follow.

Ba^{2+} Greenish-yellow

Ca^{2+} Brick-red

Cu^{2+} Green

Sr^{2+} Crimson

Na^+ Yellow

K^+ Violet (This color can sometimes be enhanced by observing the flame through cobalt blue glass, which absorbs all but blue and violet light.)

Demonstration 2: Spectrum Demonstration

Purpose

To show how colored substances absorb characteristic wavelengths of light in the visible spectrum.



Materials

Sheet of diffraction grating, 6.5- x 6.5- cm square (Edmund Scientific Company, 101 East Gloucester Pike, Barrington, NJ 08007-1380, Phone: 609-573-6260)

Cardboard with 6- x 6- cm square cut in middle

Overhead projector

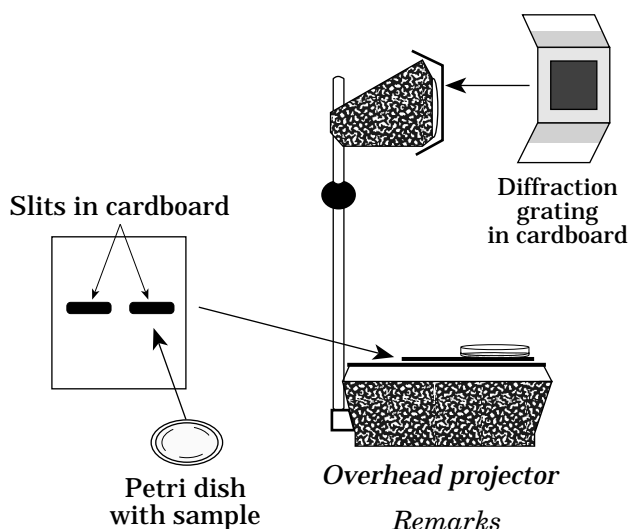
Cardboard large enough to cover the entire stage of the overhead projector

Safety

Be careful not to spill liquids on the overhead projector stage.

Procedure

Tape the 6.5- x 6.5- cm square of diffraction grating in the center of the piece of cardboard with a 6- x 6- cm square cut out of it. This grating will be placed over the upper projection lens of an overhead projector. Using the piece of cardboard large enough to cover the entire stage of an overhead projector, cut two rectangular "slits" (1- x 8- cm). The slits should be cut so that, when the cardboard is placed on the overhead stage, one slit is above the other (see illustration).



When the room is darkened, this arrangement will produce two continuous spectra on the screen. Over one of the slits, place a Petri dish. Pour a very dilute solution of potassium permanganate into the Petri dish. This "slit" will now produce a spectrum created by the presence of the compound in the Petri dish. This demonstration can be repeated for two other colored compounds (food coloring can also be used), showing that the bands produced are characteristic for the colors of these compounds.

Remarks

This demonstration will require practice to establish proper lighting in the room, arrangement for best projection of the spectra, and concentration of solutions that will produce the best results.

TIPS FOR THE TEACHER

Language of Chemistry

coordination complex compound or complex ion in which a metal ion or atom is bonded to one or more ligands.

filtrate liquid that has passed through a filter.

ligand molecule or ion bonded to the central metal atom in a coordination complex.

precipitate insoluble substance formed from soluble reactants.

qualitative analysis chemical analysis used to determine the identity of a sample.

quantitative analysis chemical analysis used to determine the amount of the constituents of a sample.

residue solid left after a liquid is evaporated or removed.

spectrometer instrument used to observe the absorption or emission of particular frequencies of light from a sample.

supernatant liquid solution remaining after a precipitate is formed.

Common Student Misconceptions

1. **“Insoluble means nothing dissolves.”**

Solubility is a relative term defined quantitatively by a solubility product (an equilibrium constant). At the qualitative level, an insoluble substance is defined in many texts as a substance that can only form solutions of less than 0.01 M.

2. **“Separations are clean.”**

No technique can completely and perfectly separate the components of a mixture.

3. **“Analysis is easy.”**

Chemical analysis is sometimes expected to answer such questions as: “What’s in this barbecue sauce?” It is thought that all that is necessary is to perform a simple test or analyze a sample with an instrument, and the answer reveals itself. Actually, quite a lot of intellectual effort is often necessary.

See “The Past 100 Years in Analytical Chemistry,” by G. Ewing in *References*.

Two anecdotes (Oesper, 1975) are related to flame tests and the qualitative identification of metal ions. The first of these concerns Frederick Soddy (1887-1956). During a visit to Paris, he received a few milligrams of radium bromide from Marie Curie. When he returned to London and the laboratory of William Ramsay, for whom he worked, he watched in horror as Ramsay used up most of his sample to do an unnecessary flame test to confirm that the sample contained radium. The second anecdote concerns Robert Wood (1868-1955), who studied chemistry under Ira Remsen at Johns Hopkins University. Wood took his meals at a boarding house. It seems that whenever steak was served for supper, beef hash was served at the following breakfast. The patrons of the boarding house suspected that table scraps were used in preparing the hash. One evening Wood left several pieces of steak on his plate after he sprinkled on it what looked like table salt, but was actually lithium chloride. The next morning he burned a piece of hash in the table lamp flame, saw the brilliant red color characteristic of lithium, and proved to the delight of his fellow diners that their suspicions were correct.

HISTORY: ON THE HUMAN SIDE

- | | |
|----------------------------|----------------------------|
| 1. Sing a song of Sulfide | When the cork is taken out |
| A beaker full of lime... | Fumes begin to reek... |
| Four and twenty test-tubes | Isn't that an awful mess |
| Breaking all the time. | To have five times a week? |

CHEM 13 NEWS, October 1969, p. 84

2. **TEN COMMANDMENTS FOR STUDENTS OF ANALYTICAL CHEMISTRY**

1. Thou shalt honor thy neighbor's olfactory sensibilities.
2. Thou shalt not kill unless thou canst prove that he did spit in thy unknowns.
3. Thou shalt not pencil titrate.
4. Thou shalt not take the name of thy teacher in vain.
5. Thou shalt not remain forever on the pans of thy balance.
6. Thou shalt keep thy desk spotless that thy days may be long in this laboratory.
7. Thou shalt not commit adulteration—of reagents.
8. Thou shalt love the lab assistant as thyself.
9. Thou shalt not covet thy neighbor's success, nor clean equipment, nor anything that is thy neighbor's.
10. Two days shalt thou labor, sweat and swear in thy laboratory; the other days are set aside for thy teacher's peace of mind and for the conservation of thine own sanity.

CHEM 13 NEWS, January 1978, p. 1292

HUMOR: ON THE FUN SIDE



3. LITTLE ION

Little Ion in my flask
Do you mind much if I ask
What your name is little ion?
Can't you see you've got me cryin'?
Are you silver, lead or tin?
Don't you see me growing weaker
As I gaze into my beaker?
Can you bear to see my fate
If my unknown is turned in late?

Come now do not be a fink,
Are you cobalt? Iron? Zinc?
Stir and centrifuge and test
I have done my very best.
Yet my prof will probably say—
Chloride, nickel, sulfate, nay.
Little unknown be a doll
Help me pass the course called "Qual."

Courtesy of Jerry Wellins, Arthur Breyer, and Marie Bonner.

4. Word Search (see Appendix for master copy)

S Y R L E T A T I P I C E R P A A
C U E W T F I L T R A T E E Z G F
O L P Y R G M F U X N S E V T G F
O M P E W T D V U U I V R I B E B
R Z O I R X T R A D I M E T G D B
D B C W N N I L U T I Y V A V A M
I O C J S D A E A J Q Y O T A H J
N W V A H U B T J E U A P I K O F
A P J T X L I D A C O F L T X P D
T S C V O L I G A N D C D N R N Y
I Y Z K A G V Q G B T X U A W X R
O C C U S Y R J O Z W T Y U X Z W
N V Q A F I W J Z D A T G Q P M C

Words about the concepts in this module can be obtained from the clues given. Find these words in the block of letters:

1. Solid left after a liquid is evaporated or removed.
2. Type of chemical analysis used to determine a sample's identity.
3. Adjective describing the liquid remaining after a precipitate is formed.
4. Liquid that has passed through a filter.
5. Type of chemical analysis used to determine the amount of the sample's constituents.
6. Insoluble substance formed in an aqueous solution from soluble reactants.
7. Type of complex in which a metal ion is bonded to one or more ligands.
8. Molecule or ion bonded to a central metal atom.
9. The ion studied in the activity that forms a red precipitate with $\text{Fe}(\text{CN})_6^{3-}$.
10. The ion studied in the activity that forms a dark blue solution with NH_3 .

Answers: 1. RESIDUE 2. QUALITATIVE 3. SUPERNATANT 4. FILTRATE
5. QUANTITATIVE 6. PRECIPITATE 7. COORDINATION 8. LIGAND
9. COBALT 10. COPPER

5. See cartoons at end of module.

MEDIA

1. *Molecular Spectroscopy*, CHEM Study film/video available from Ward's Natural Science Establishment, Inc., P.O. Box 92912, Rochester, NY 14692-9012; (800) 962-2660.
2. Software published by *JCE: Software*, a publication of the *Journal of Chemical Education*, Department of Chemistry, University of Wisconsin-Madison, 1101 University Avenue, Madison, WI 53706-1396: (608) 262-5153 (voice) or (608) 262-0381 (FAX).
 - a. *Organic Spectroscopy*. Vol. II A, No. 2, for Apple II computer. Simulated IR, Proton and C-13 NMR, and mass spectra for a variety of compounds.
 - b. *HPLC – An Instrument Simulator*, Vol. I B, No. 2, for IBM PS/2 PC-compatible computers.
 - c. *Proton NMR Spectrum Simulator*, Vol. II C, No. 1, for Macintosh computer.
3. Software published by Project SERAPHIM, Department of Chemistry, University of Wisconsin-Madison, 1101 University Avenue, Madison, WI 53706-1396: (608) 263-2837 (voice) or (608) 262-0381 (FAX).
 - a. For the Apple Macintosh computer: MC 901
 - b. For the Apple II computer: AP 901, AP 903, AP 904
 - c. For IBM PCs and PC-compatibles: PC 3401

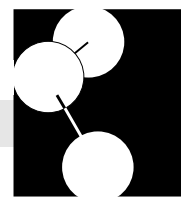
Six-Solution Problem – A simulation of the problem of determining the composition of six solutions by mixing them on a spot plate.

Canal 1,2,3,4,5 – Tests a student's knowledge of the traditional qualitative analysis scheme.

The Spectronic 20 is an instrument used to characterize solutions that can be useful in qualitative analysis. This type of instrument is sometimes found in high school laboratories.

EQUIPMENT AND INSTRUMENTATION

Links/Connections



WITHIN CHEMISTRY

1. Within chemistry, the most obvious connections are with chemical equilibrium and acid/base chemistry. The separations of most inorganic qualitative analysis schemes depend on either the relative solubility of the metal ions with various anions or upon the ability of these ions to form complex ions. pH very often plays a role in these processes. The ability of H^+ to dissolve insoluble hydroxides, and for amphoteric hydroxides [e.g., $Al(OH)_3$] to dissolve in excess base depends on competing equilibria. The solubility of other substances in ammonia depends on the formation of stable complex ions, another equilibrium consideration. There are also links to the *Transition Elements; Rocks, Minerals, and Gems; and Photochemistry* modules. The *Industrial Inorganic Chemistry* module has an exercise on the analysis of fertilizers.
2. **Alloys.** The purpose of preparing an alloy is to improve the specific properties of the principal component. Properties such as hardness, melting point, etc. can be controlled by alloying.
 - a. Most gold jewelry is really a gold alloy containing gold, copper, and silver, since pure gold is too soft for ordinary use. 24 carat gold is 100% gold. 12 carat gold is 50% gold, 35% copper, and 15% silver. Coinage gold is usually 90% gold and 10% copper. White golds are mixtures of gold with either palladium or platinum. German silver and nickel silver contain copper, nickel, and zinc in different proportions.
 - b. Copper is the principal component of brasses and bronzes. Red brass is 90% copper and 10% zinc, yellow brass is 67% copper and 33% zinc. Gun-metal bronze is 90% copper and 10% tin; bearing bronze is 82% copper, 16% tin, and 2% zinc; hardware bronze is 89% copper, 9% zinc, and 2% antimony.
 - c. Sodium melts at 97.8 °C, potassium melts at 63.7 °C, but an alloy of 22% sodium and 78% potassium melts at -11 °C. Soft (lead) solder is composed of tin, lead, antimony, copper, and bismuth, whereas hard (silver) solder contains 10-80% silver and varying amounts of copper and zinc.

TO THE CONTEMPORARY WORLD

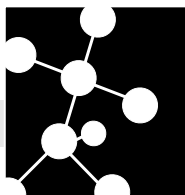
See *Forensic Chemistry* module.

Water purity.

Environmental hazards (Pb, NO, sulfur in industry).

Community resources: water treatment plant, soil conservation (county agent), sewage treatment facility, forensic facility.

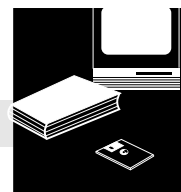
Technicians use qualitative testing methods to test pool water for chlorine and acid and indicators to decide if the pH is too high or too low.



Extensions

Chromatography is an analytical technique that is easily done in a high school laboratory. *Demonstration 3* in the *Separations* and *Solutions* modules illustrates paper chromatography. *Demonstration 4* in the *Solutions* module describes a column chromatography activity. (See also Dreyfus *Microscale Lab: Paper Chromatography*, Brogie, 1987.)

References



Module drafted by Mike Abraham, Donna Deen-Coshow, and Bill Fix, the Oklahoma team.

Abraham, M., and Pavelich, M. (1991). *Inquiries into chemistry* (2nd Ed.). Prospect Heights, IL: Waveland Press.

“Chemical Tests,” “Laboratory Procedures,” and “Qualitative Ion Analysis Using Semi-Micro Techniques” are taken from this work.

Barber, J. (1985). *Crime lab chemistry* [LHS GEMS] (Teacher’s Guide). Berkeley, CA: Lawrence Hall of Science, University of California.

From the Great Explorations in Math and Science (GEMS) series, this contains activities relating to chromatography, separating mixtures, pigments, and solubility. Although written for Grades 4-8, the activities are also suitable for high school students. Order this book from Lawrence Hall of Science, University of California, Berkeley, CA 94720.

Brogie, E. (1987). *Dreyfus microscale lab: Paper chromatography* [Woodrow Wilson-Dreyfus Institute, 1987, #6]. Princeton, NJ: Princeton University.

Chen, C., and Ingels, R. (1987). *Dreyfus microscale lab: Six solutions* [Woodrow Wilson-Dreyfus Institute, 1987, #35]. Princeton, NJ: Princeton University. Published by University of Nebraska in *Microscale Experiments for High School Chemistry Classes*, 1988. [Write to David Brooks, 118 Henzlik Hall, University of Nebraska, Lincoln, NE 68588-0355.]

Chen, C., and Ingels, R. (1987). *Dreyfus microscale lab: Qualitative analysis* [Woodrow Wilson-Dreyfus Institute, 1987, #36]. Princeton, NJ: Princeton University.

Cole, G. M., and Waggoner, W. H. (1983). Qualitative analysis: The current status. *Journal of Chemical Education*, 60, 135-140.

The current status according to these authors is that qualitative analysis is making a comeback. The article is a reconsideration of the qual scheme to make it more suitable for modern times.

Elo, H. (1987). Is thioacetamide a serious health hazard in inorganic chemistry laboratories? *Journal of Chemical Education*, 64, A144, A146.

The short answer from this article is “yes.”

Ewing, G. W. (1976, April 6). The past 100 years in analytical chemistry. *Chemical and Engineering News*, 54(15), 128-145.

This is a good source of history for this topic.

Gerber, S. M. (Ed.). (1983). *Chemistry and crime*. Washington, DC: American Chemical Society.

Numerous fictional and nonfictional references to chemistry and its relationship to crime detection.

Lagowski, J. J., and Sorum, C.H. (1991). *Introduction to semimicroqualitative analysis* (7th Ed.). Englewood Cliffs, NJ: Prentice-Hall.

This is the latest edition of a widely used text on inorganic qualitative analysis. It is cited here only as an example. There is a large number of such texts available and any one of them is a useful reference not only for qualitative analysis but for general chemical information.

Oesper, R. E. (1975). *The human side of scientists*. Cincinnati, OH: University of Cincinnati Publications.

This is the source of the two anecdotes that are related to flame tests and the qualitative identification of metal ions (in *History* section).

Rich, R. L. (1984). Qualitative analysis, with periodicity, for “real” solutions. *Journal of Chemical Education*, 61, 53-58.

This article describes two qualitative analysis schemes based on bromide and iodide ions. It is claimed that these schemes make more “sense” in terms of periodicity.

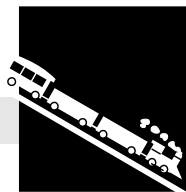
Solomon, S., Fülep-Poszmik, A., and Lee, A. (1991). Qualitative analysis of eleven household compounds. *Journal of Chemical Education*, 68(4), 328-329.

This article contains a scheme for identifying compounds commonly found in the house. All of the chemicals used, including reagents, can be obtained as consumer items.

West, P. W., and Vick, M. M. (1959). *Qualitative analysis and analytical chemical separation* (2nd Ed.). New York, NY: MacMillan.

This is a qualitative analysis scheme based on hydroxide and benzoate ions instead of sulfide.

Appendix

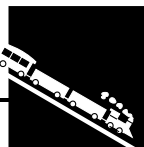


- **Transparency Masters**
 1. Identification of Selected Substances
 2. Qualitative Ion Analysis Using Semimicro Techniques
 3. Word Search
- **Humor**

Identification of Selected Substances

Products of chemical reactions can be identified by their chemical or physical properties. Following is a key to identifying some common substances.

Formula	Color	Odor	State at 25 °C	Solubility in H ₂ O	Miscellaneous
H ₂	None	None	Gas	No	Burns explosively
O ₂	None	None	Gas	No	Makes glowing splint flame
CO ₂	None	None	Gas	Yes	Turns limewater milky
NO ₂	Brown	Choking	Gas	Yes	Solution is acidic
I ₂	Purple	Medicine-like	Solid	Yes	Purple in hexane (or cyclohexane)
Br ₂	Red-brown	Choking	Liquid	Yes	Orange in hexane (or cyclohexane)
Cl ₂	Pale green	Choking	Gas	Yes	Yellow in hexane (or cyclohexane)
NH ₃	None	Choking (smelling salts)	Gas	Yes	Solution is basic
SO ₂	None	Suffocating	Gas	Yes	Solution is acidic
H ₂ S	None	Rotten eggs	Gas	Yes	Solution is neutral
HCl	None	Choking	Gas	Yes	Solution is acidic
H ₂ O	None	None	Liquid	—————	CoCl ₂ paper turns pink



Qualitative Ion Analysis Using Semimicro Techniques

In these experiments you will be concerned with determining what particular ions are in a solution or solid. This concern with only what is present is called qualitative analysis. (A concern for how much is present is called quantitative analysis). You will be working with very small amounts of solutions, usually about 1/4 to 1/2 of a milliliter. Thus the experimental procedures are called semimicro (small amount) techniques. Two other definitions of importance are:

Precipitate a solid formed when two solutions are mixed (abbreviated ppt).

Supernatant liquid the liquid phase of a solid plus liquid mixture (abbreviated spn).

The basic idea in these experiments is to add a reagent to a solution of ions and to note any and all changes that occur. In particular, one looks for and notes down:

1. The formation and color of a ppt.
2. Changes in color of the solution or spn.
3. Redissolving of a ppt.
4. Evolution of a gas.

Additions and Mixing

The amounts of solutions to be mixed in each test are often critical to the results and must be carefully watched. The amounts are usually controlled by counting drops of solution used (18 drops = 1 mL). When making additions suspend the end of the dropper just above the opening of the test-tube and gently apply pressure to the dropper bulb so that the drops fall one at a time into the test-tube.

When test reagent has been added to a solution, mix the two completely before you write down your observations. The results that you see when one solution is layered on top of the other may not be the same as when they are mixed. To mix the solutions move a clean stirring rod gently up and down within the test-tube; *i.e.*, use a “butter churn” type motion. Watch that the liquid does not overflow the test-tube. Be careful not to “punch” a hole in the bottom of the test-tube.

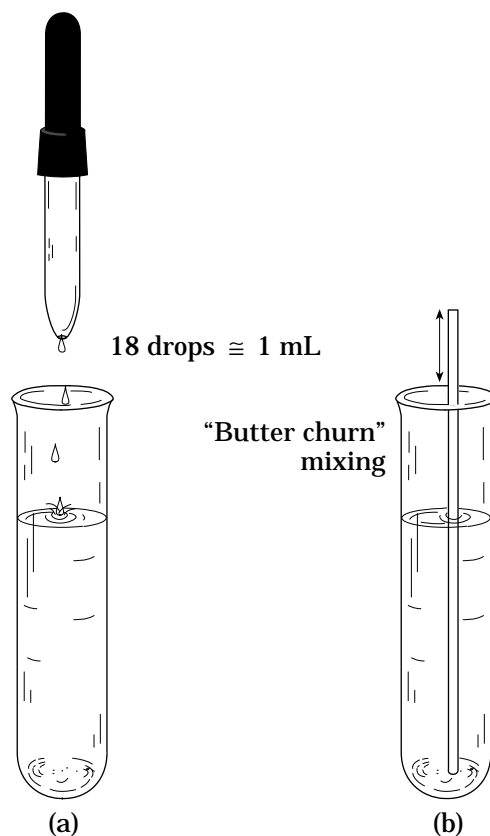


Figure A. (a) Dropwise addition and (b) mixing.

The Centrifuge

The centrifuge is a machine that spins a test-tube solution at high speeds causing the heavier and lighter parts of the mixture to separate. In essence, it accelerates what gravity would have done anyway. The centrifuge is used to separate a precipitate from the supernatant liquid. Usually only 1 or 2 min of centrifuging are needed to separate the ppt and spn.

Precautions to be observed in using the centrifuge are as follows. (1) Do not have the test-tubes more than 4/5 full. This will prevent spilling in the centrifuge. (2) Place the tubes in the centrifuge in a symmetrical pattern such as one tube directly across from another. If you are centrifuging just one test-tube, place another containing an equal volume of water across from it. If the placement of the tubes is not balanced, the spinning will be uneven and the centrifuge will move (“walk”) on the bench. Allow the centrifuge to slow down by itself. Helping it slow down can cause injury. Also if the slow down is too abrupt the ppt and spn will remix.

Decanting

This is the term used to denote removing a supernatant liquid from a precipitate. There are two decanting methods. The first is to simply pour the spn out of the test-tube leaving the ppt behind. The second method is to draw the spn out of the test-tube using a dropper. This dropper method is used when the ppt is not a firm pack and might pour out with the spn. It is also the preferred decantation method where the spn is to be saved for later use. It is often difficult to pour directly from one small test-tube to another.

When using the dropper method be sure to compress the bulb **before** inserting the dropper. A rush of bubbles in the spn will cause the ppt and spn to remix.

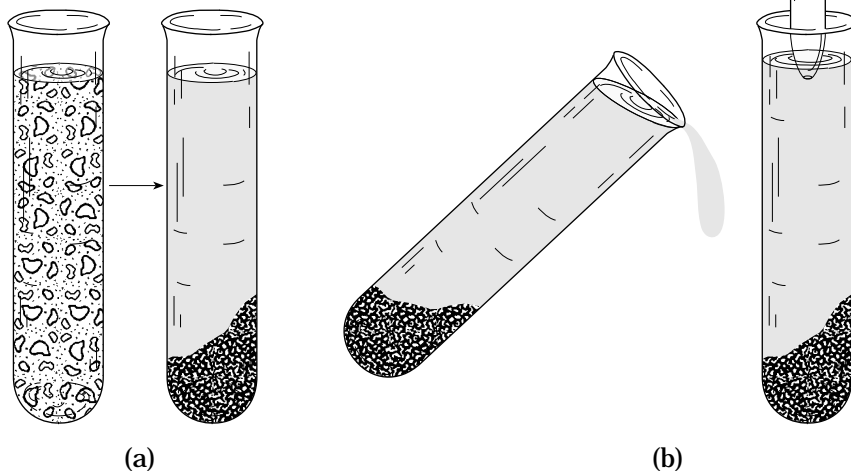


Figure B. (a) Effect of centrifuging and (b) decanting by pouring or drawing.

Washing a Precipitate

Not all of the supernatant is removed from the test-tube when one decants. A few drops remain on top of the precipitate, some clings to the test-tube walls and some is trapped within the packed precipitate. If a later test on the ppt requires that the chemicals in the spn be absent, these traces of spn are removed by a procedure called washing.

Washing a ppt involves the following sequence of steps. (1) Decant the original spn. (2) Add to the ppt about 1 mL of distilled water (or other prescribed liquid). (3) Completely mix the H_2O and ppt. This allows the H_2O to leach out any spn trapped in the packed ppt. (4) Centrifuge the mixture. (5) Decant the liquid, discarding it. (6) Repeat the process. Usually two such water washes are sufficient as they will leach out about 97% of the spn originally left in the test-tube.

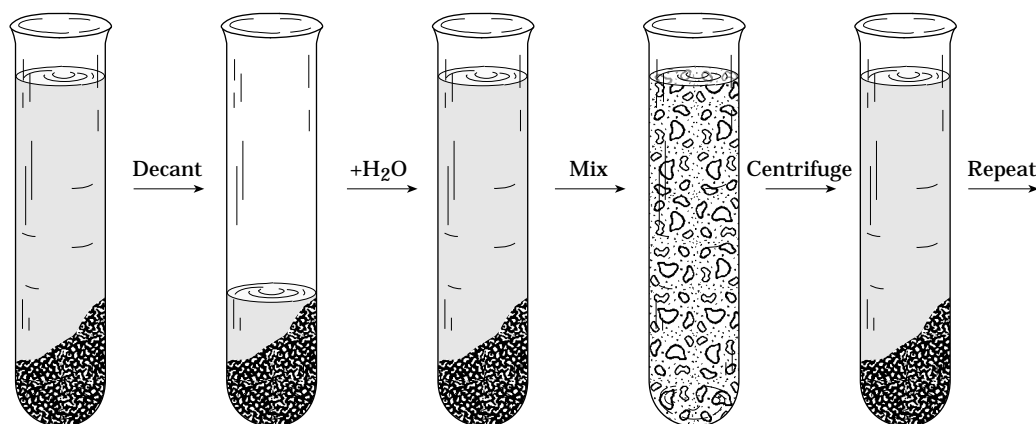
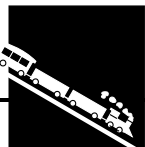


Figure C. Washing a precipitate.



Heating

You should **not** use a burner flame to directly heat liquid in a small test-tube. Such direct heating is too intense and too localized. It will usually cause the liquid to blow out of the test-tube. This practice can cause injury and, in any case, ruins the experiment.

Heating is to be accomplished by resting the test-tubes in beakers of heated water. This heating is more even and keeps the temperature from exceeding 100 °C. If a test-tube heating platform (also called an aluminum holder) is available it should be used in the beaker. Otherwise use a beaker large enough to keep the test-tubes from protruding too far over the beaker lip.

Adjust the water level in the beaker so that only the lower 1/2 to 3/4 of the test-tube is submerged. Move the burner flame in and out as needed to keep the water hot but to prevent violent boiling (see Figure D).

pH Measurement and Control

It is important in many reactions that the solution's pH be carefully controlled. The control is achieved by adding an acid or base solution dropwise until the required pH is obtained. The pH of the solution should be measured after each dropwise addition by using litmus or pH paper. Do not dip the pH paper into the solution. This practice can lead to chemicals from the paper getting into the solution. The proper procedure is to mix the solution with a stirring rod and then to touch the wet end of the rod to the test paper.

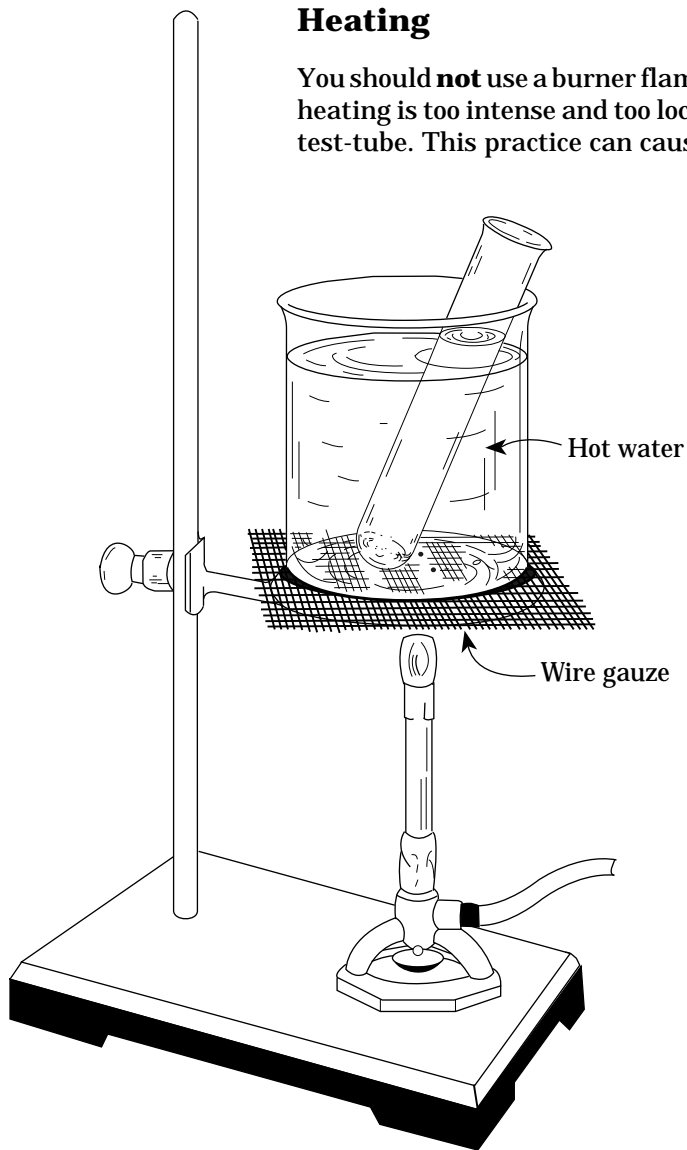


Figure D. Heating a sample.

The pH of a gas bubbling out of a solution can be tested by wetting the test paper with water and then holding it over the mouth of the test-tube.

Contamination

Even the smallest contamination of your ion or reagent solutions can change the test results. Contamination is a particular problem with these experiments because you reuse equipment so often. **Clean and distilled water rinse each piece of equipment after each use.** Thus you should clean a dropper before using it in a different solution, clean a stirring rod before using it to mix a second solution, *etc.*

Often cleaning requires only that the test-tube, rod, *etc.* be rinsed several times with tap water then 2 to 3 times with distilled H₂O. The equipment does not have to be dry. Use a test-tube brush to clean out stubborn ppts. A dropper can be cleaned by removing the bulb and running water down the barrel.

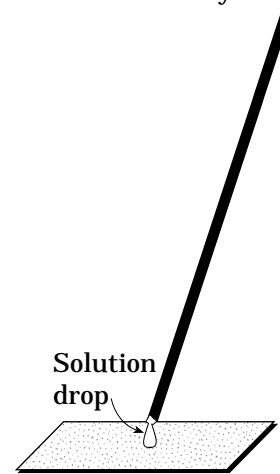


Figure E. Testing pH.

Complete Precipitation

In analysis of an unknown you will often depend on precipitating an ion completely from solution to separate it from the other ions. It is important that this precipitation reaction be complete.

After centrifuging the ppt plus spn mixture, add a drop or two more of the test reagent. If new ppt forms in the spn, add 2 more drops of test reagent, mix, and recentrifuge. Again add a drop of reagent. Continue this 1 to 2 drop testing of the spn until no new ppt forms. At this point the reaction is complete (see Figure F).

Organization and Reagents

It is strongly suggested that you take a few minutes to plan your work. You will be performing 25 or more individual tests in one period; thus, some organization is needed to save time and not to confuse your results. The following ideas might be helpful.

1. After using a dropper, immediately return it to its reagent bottle. Do not have two droppers out of their reagent bottles at one time. This will keep you from returning a dropper to the wrong reagent bottle.
2. Label each test-tube and assign it a labeled position in your test-tube rack. Never have two test-tubes out of the rack at the same time; always return one before picking up another. This keeping track of test-tubes is needed because the label on the tube can be smeared in handling. Be sure the label on a tube is clear before you put it into a centrifuge.
3. Put all used, dirty glassware in one section of the bench so as not to confuse it with clean glassware.
4. When you are to run a test on several different ion solutions, run the test on all of them simultaneously. Set up a labeled tube for each ion solution in the rack. Put the proper ion solution into each test-tube. Next go from one to the other adding the test reagent and mixing. This simultaneous testing saves time and allows you to make direct comparisons of the results for different ion solutions.
5. Both lab partners should observe the results of each test and record their own perception of the results. Also describe fully your observation; do not simply put down "Reacted." You will need to recall exactly what you saw happen when you analyze your unknown.

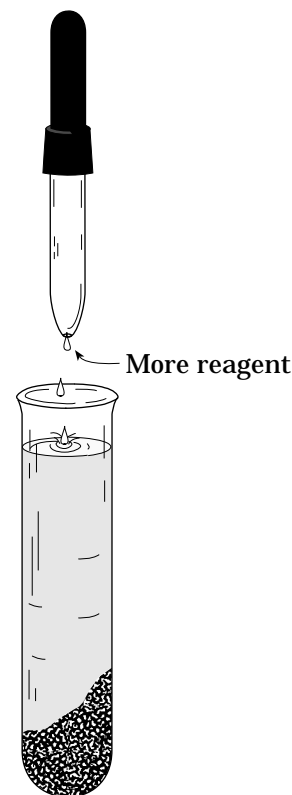
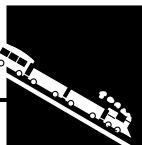


Figure F. Testing for complete precipitation.

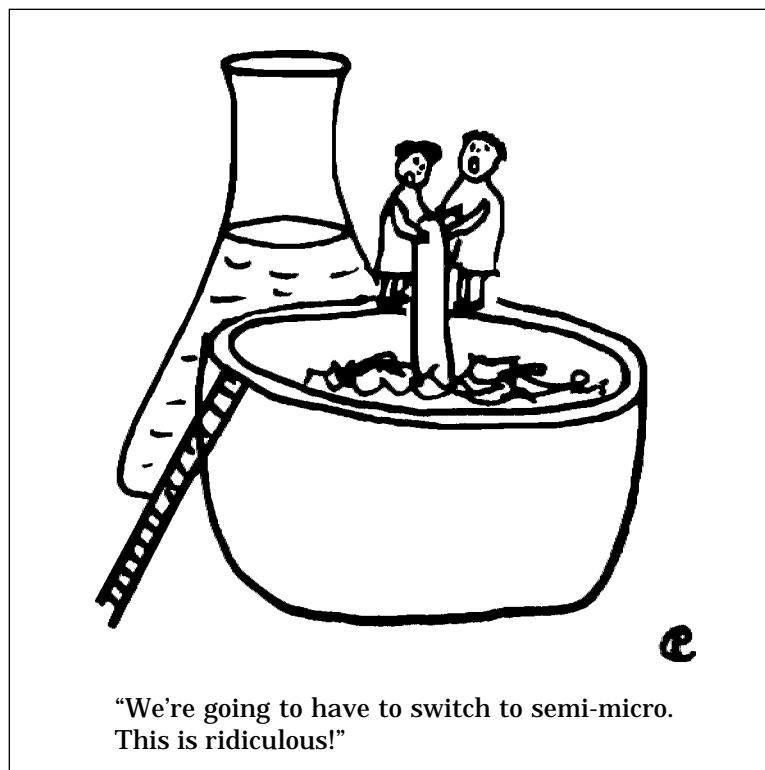


Word Search

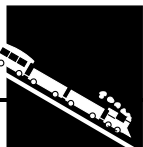
S Y R L E T A T I P I C E R P A A
C U E W T F I L T R A T E E Z G F
O L P Y R G M F U X N S E V T G F
O M P E W T D V U U I V R I B E B
R Z O I R X T R A D I M E T G D B
D B C W N N I L U T I Y V A V A M
I O C J S D A E A J Q Y O T A H J
N W V A H U B T J B U A P I K O F
A P J T X L I D A C O F L T X P D
T S C V O L I G A N D C D N R N Y
I Y Z K A G V Q G B T X U A W X R
O C C U S Y R J O Z W T Y U X Z W
N V Q A F I W J Z D A T G Q P M C

Words about the concepts in this module can be obtained from the clues given. Find these words in the block of letters:

1. Solid left after a liquid is evaporated or removed.
2. Type of chemical analysis used to determine a sample's identity.
3. Adjective describing the liquid remaining after a precipitate is formed.
4. Liquid that has passed through a filter.
5. Type of chemical analysis used to determine the amount of the sample's constituents.
6. Insoluble substance formed in an aqueous solution from soluble reactants.
7. Type of complex in which a metal ion is bonded to one or more ligands.
8. Molecule or ion bonded to a central metal atom.
9. The ion studied in the activity that forms a red precipitate with $\text{Fe}(\text{CN})_6^{3-}$.
10. The ion studied in the activity that forms a dark blue solution with NH_3 .



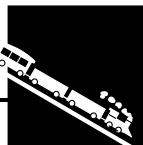
CHEM 13 NEWS, December 1980, p. 3. Reprinted with permission.



CHEM 13 NEWS, October 1978. Reprinted with permission.



CHEM 13 NEWS, November 1978. Reprinted with permission.

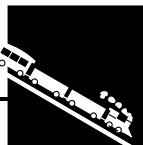


**IT MUST HAVE BEEN CRACKED
WHEN I GOT IT.**

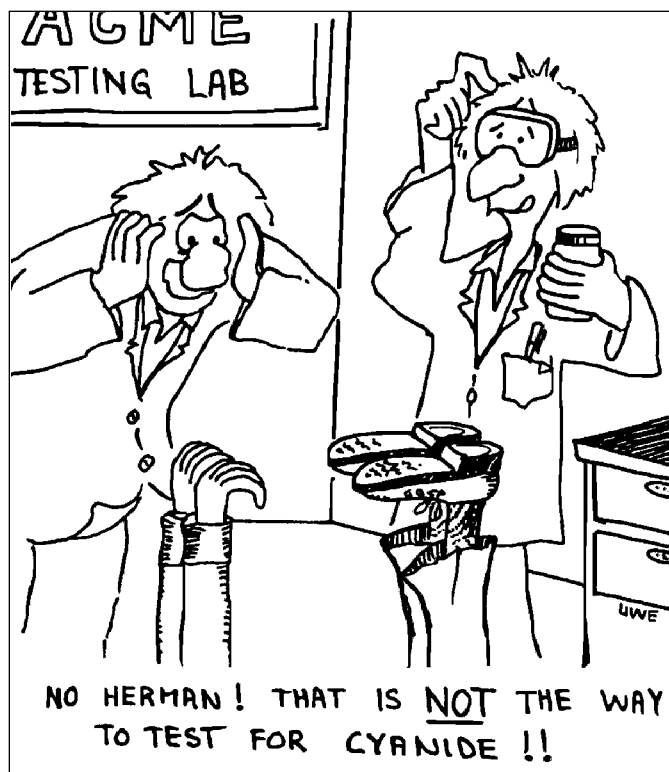
CHEM 13 NEWS, March 1980, p. 6. Reprinted with permission.



CHEM 13 NEWS, March 1981, p. 9. Reprinted with permission.



CHEM 13 NEWS, September 1981, p. 5. Reprinted with permission.



CHEM 13 NEWS, February 1983, p. 13. Reprinted with permission.