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Pleasing precipitation performances – the microscale way

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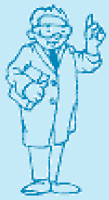
Dropping out: Learn about the chemistry of precipitation and introduce your students to chemical reactions that form colourful new compounds using microscale chemistry methods that are cheap, quick, and easy to do.

Seeing precipitates suddenly appear is magical to students (and many think it is magic).[1] Two salt solutions are mixed and suddenly a solid appears. Sometimes the solid is a different colour to the solutions. If 1 M copper sulfate solution is mixed with sodium hydroxide solution, a blue solid appears. The solid is copper hydroxide, which is insoluble in water. If lead or silver nitrate solution is used, then a yellow solid suddenly appears.

Traditional methods using test tubes waste a lot of resources. There is an awful lot of material thrown down the sink or saved in waste bottles. A previous article on pH indicators showed how procedures can be carried out on a plastic folder. The instructions are printed on a worksheet, below the plastic surface, so there is no immediate distraction. These activities are suitable for students aged 11–18.

Activity 1: Solubility rules

When students perform precipitation reactions, they can often fail to appreciate where the solids are coming from, since they never see the original salts being dissolved. Getting students to make the solutions adds a lot of time to lessons. This microscale experiment is a quick and simple way to introduce the concept of solubility.



Safety note

The solutions are of low hazard. Eye protection should be worn, and hands should be washed with soap and water after the experiment in case of contact with copper(II) chloride, which can cause skin irritation.

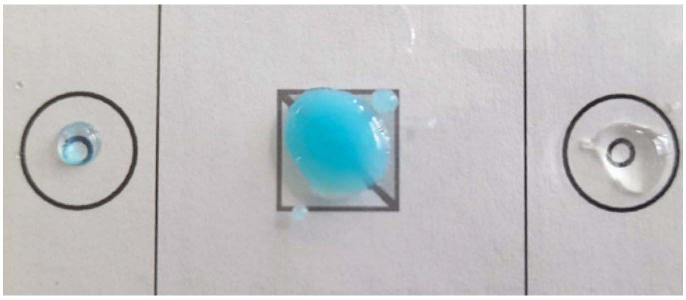
Materials

- Printout of Worksheet 1 (with an optional precipitation activity)
- Plastic pocket or folder (alternatively, laminate the worksheet)
- Copper(II) chloride
- Sodium carbonate
- Water
- Wooden splint or toothpick

Procedure

1. Insert the worksheet into the plastic folder.
2. Add copper chloride and sodium carbonate crystals to the boxes shown on the worksheet.
3. Add water with a pipette to fill the whole circle and stir with a freshly cut, pointed splint to dissolve the solid.

4. With a pipette, move two drops of the copper chloride solution in the circle into the square in the middle.
5. With another pipette, move two drops of the sodium carbonate solution in the circle on the right into the square in the middle.
6. Stir the contents of the square with a freshly cut, pointed splint.
7. Record or photograph the results.
8. Disposal: wipe the plastic surface with a paper towel and dispose of the towel in the waste.



When solutions of copper(II) chloride (left) and sodium carbonate (right) are combined (centre), a blue precipitate of insoluble copper carbonate forms.

Image courtesy of the authors

Discussion

The results allow students and teachers to discuss where the chemicals are coming from, and they provide more belief in the chemical equation:

$CuCl_2 + Na_2CO_3 \rightarrow CuCO_3 + 2NaCl$

The reaction works because sodium carbonate and copper chloride are soluble in water, but copper carbonate is not very soluble, and this is the reason why a precipitate forms. It is really a saturated solution of copper carbonate in water.

Every student knows sodium chloride is soluble in water. So, now students can complete a table of results and observations.

Salts soluble in water	Salts insoluble in water
Copper chloride Sodium carbonate Sodium chloride	Copper carbonate

Extension

Try reactions between the following salts:

1. copper sulfate and sodium carbonate
2. sodium chloride and potassium nitrate
3. sodium chloride and silver nitrate
4. sodium sulfate and barium nitrate
5. sodium carbonate and magnesium sulfate

There will be no precipitate with the second one, which implies that sodium nitrate and potassium chloride are also soluble in water. The fifth reaction is a 'green' reaction with chemicals available from the local shop. Using test tubes would generate a large amount of cleaning at the end. Now with the use of a plastic sheet, the procedure is rapid, giving the teacher time to generate student feedback on their observations, with very little waste generated.

Activity 2: Identification of positive ions

Metal ions can be identified in solids or in 0.1 M solutions using precipitation techniques. The technique of adding reagents to a plastic surface, with instructions underneath, is also ideally suited to the study of transition metals.

Safety note

The solutions are of low hazard. Eye protection should be worn, and hands should be washed with soap and water after the experiment in case of contact, as solutions of transition-metal ions can cause skin irritation.

Materials

- Printouts of Worksheets 2 and 3
- Plastic pockets or folders (alternatively, laminate the worksheets)
- Iron(II) sulfate solution (0.1 M) or a few grains of solid and water
- 0.4 M sodium hydroxide solution
- Eye dropper style bottle with above solutions or dropping pipette
- Wooden splint or toothpick

Procedure

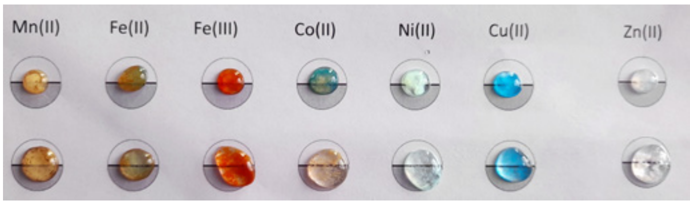
1. Insert Worksheet 2 into the plastic folder.
2. Add two drops of 0.1 M iron(II) sulfate solution to the circle on Worksheet 2 labelled Fe^{II}, or place a few grains of the solid, add water, and stir with a wooden splint.
3. Add one drop of 0.4 M sodium hydroxide solution to the puddle and stir again. A green precipitate of iron(II) hydroxide is obtained.

Iron(II) sulfate crystals (left), iron(II) sulfate solution (centre), and iron(II) hydroxide (right)

Image courtesy of the authors

Extension

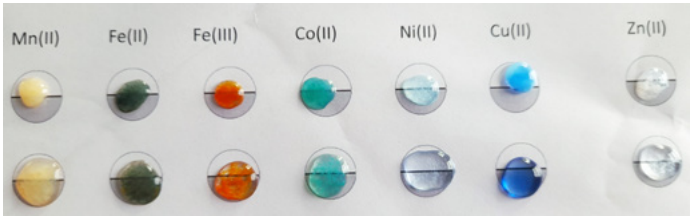
The activity can be extended to study the chemistry of transition metals (Worksheet 2), which can give a variety of pleasing colours that can be observed when they form a metal hydroxide upon the addition of 0.4 M sodium hydroxide solution to a 0.1 M solution of the transition-metal ion.



Transition-metal-ion solutions with one drop of 0.4 M sodium hydroxide (top row) and six drops 0.4 M sodium hydroxide (bottom row)

Image courtesy of the authors

The addition of 2 M ammonia solution can also show a wide range of colourful precipitates (Worksheet 3).



Transition-metal-ion solutions with one drop of 2 M ammonia (top row) and six drops of 2 M ammonia (bottom row)

Image courtesy of the authors

Activity 3: Identification of negative halide ions

Negative ions can also be identified using microscale techniques by the addition of silver nitrate.

Safety note

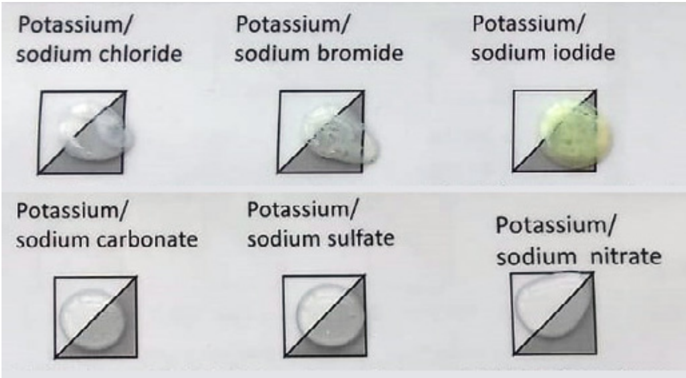
The solutions are of low hazard. Eye protection should be worn, and hands should be washed with soap and water after the experiment in case of contact, as solutions of silver nitrate can stain skin.

Materials

- Printout of Worksheet 4
- Plastic pocket or folder (alternatively, laminate the worksheet)
- Solutions of sodium or potassium chloride, bromide, iodide, sulfate, carbonate, and sulfate
- 0.4 M nitric acid
- 0.05 M silver nitrate
- 2 M ammonia solution
- Eye-dropper-style bottles containing the above solutions or dropping pipettes
- Wooden splint or toothpick

Procedure

1. Insert Worksheet 4 into the plastic folder. Use the top row of squares.
2. Add two drops of the relevant sodium or potassium salt to each of the squares on the sheet.
3. Add two drops of 0.4 M nitric acid and two drops of 0.05 M silver nitrate. Stir the mixtures with a wooden splint.
4. Record observations before adding two drops of 2 M ammonia solution.



Potassium salt solutions after addition of 0.4 M nitric acid and 0.05 M silver nitrate

Image courtesy of the authors

Discussion

The results illustrate one of the strengths of this technique. Only three of the six salts show positive responses to the addition of silver nitrate solution. The responses of the other negative ions are negative, since the silver nitrate test is unique to halides. On addition of 2 M ammonia, silver chloride dissolves, silver bromide partly dissolves, and silver iodide is unaffected.

Extension

To identify carbonate ions in a salt solution, add two drops of pH indicator, which should indicate an alkaline pH. Add two drops of 0.4 M hydrochloric acid to the sample. Bubbles of carbon dioxide will be released. A template for this is shown in the middle row of Worksheet 4.

To identify sulfate ions in a salt solution, add two drops of 0.4 M hydrochloric acid to a metal sulfate solution (0.1 M), followed by two drops of 0.1 M barium chloride. A white precipitate of barium sulfate will form. A template for this is shown in the bottom row of Worksheet 4.

Activity 4: An insight into how reactions work – diffusing precipitates

"We did precipitates last year" is a not unknown comment from some students. It is important for teachers to have something new that not only substantiates what has been taught before, but also presents new information, aiding further understanding of these reactions. This activity combines dissolving, diffusion, and precipitation. It needs to be done after the ionic nature of salts has been introduced.

Safety note

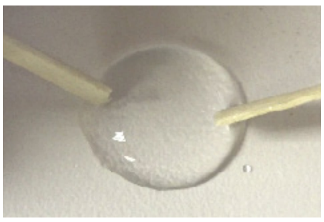
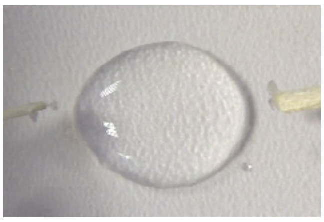
The solids are mostly of low hazard. The compounds of some metals, such as lead, cobalt, and nickel, may be banned from use in some countries. Readers should always follow local rules. However, one of the advantages of the microscale approach is that so little of the substance is used, there is not enough to cause a health issue, and waste can be collected and stored for disposal. Eye protection should be worn, and hands should be washed with soap and water after the experiment in case of contact.

Materials

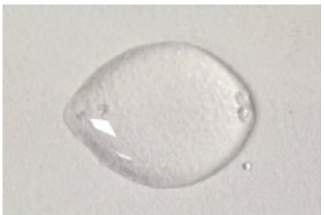
- Plastic pocket or folder (or laminated sheet of paper)
- Silver nitrate (a few crystals)
- Potassium iodide (a few crystals)
- Water
- Wooden toothpick or splints

Procedure

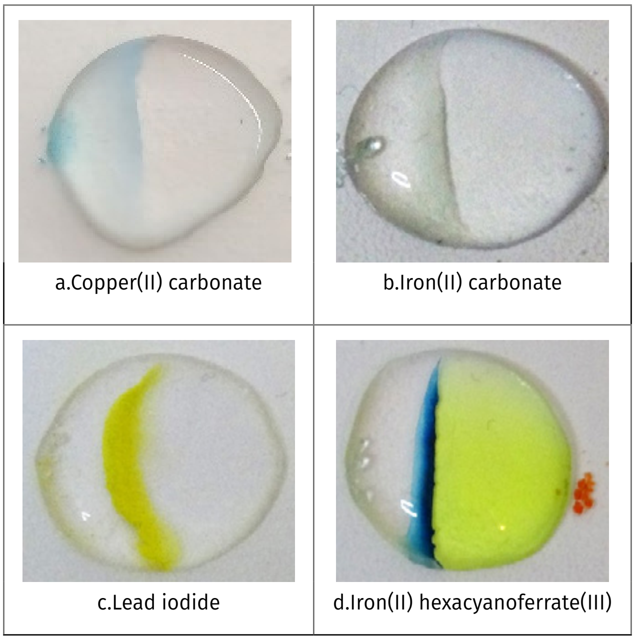
1. Place a small puddle of pure water on the plastic surface. Place a few grains of the two solids, say silver nitrate and potassium iodide, either side of the puddle.
2. There are two ways to proceed:
   - Dampen the ends of two splints in water and touch each to one of the solids to pick up small amounts. Insert the two solids, one on either side of the puddle.
   - Use the flat end of a splint to push a grain into each side of the puddle.

3. In less than a minute, the two solids dissolve and diffuse within the water, and when the chemicals meet, a precipitate is formed.




4. Try some more diffusing precipitate reactions:
   a. Copper(II) sulfate and sodium hydrogen carbonate
   b. Iron(II) sulfate and sodium hydrogen carbonate
   c. Lead nitrate and potassium iodide
   d. Iron(II) sulfate and potassium hexacyanoferrate(III) – the product, iron(II) hexacyanoferrate(III), is the pigment Prussian blue



Examples of diffusing precipitate reactions

Image courtesy of Bob Worley

Discussion

Discuss the following questions with your students:

- How would you describe the bonding and structure of the salt crystals?
- Were the compounds soluble when they were pushed into each side of the water drop?
- What happened to the ions after they dissolved?
- What precipitate was formed and seen after observation, and what was the soluble product?

If we use the reaction involving silver nitrate and potassium iodide as an example, we can see that both compounds are soluble in water. These are ionic compounds with an ionic lattice structure. As the ions are released from the ionic lattice during dissolving, the ions diffuse and migrate towards each other until a precipitate is formed near the middle. The precipitate formed is silver iodide. The other soluble product is potassium nitrate.

A visualizer or USB microscope is ideal for a highly visual classroom demonstration. «

References

[1] Worley B et al. (2019) Visualizing dissolution, ion mobility, and precipitation through a low-cost, rapid-reaction activity introducing microscale precipitation chemistry. *Journal of Chemical Education* **96**: 951–954. doi: 10.1021/acs.jchemed.8b00563

Resources

- Learn how to make indicators from butterfly tea: Prolongo M, Pinto G (2021) Tea-time chemistry. *Science in School* **52**.
- Read an introduction to microscale chemistry in the classroom: Worley B (2021) Little wonder: microscale chemistry in the classroom. *Science in School* **53**.
- Teach about pH chemistry using microscale methods: Worley B, Allan A (2021) Little wonder: pH experiments the microscale way. *Science in School* **54.**
- Try a classroom activity to extract essential oils from fragrant plants: Allan A, Worley B, Owen M (2018) Perfumes with a pop: aroma chemistry with essential oils. *Science in School* **44**: 40–46.
- Watch the precipitation of potassium iodide reacting with lead nitrate.
- View a fantastic infographic by *Compound Interest* on the colours and chemistry of indicators.
- Read a *ChemEd X* article on the many uses of red cabbage extracts.

AUTHOR BIOGRAPHY

Dr Adrian Allan is a teacher of chemistry at Dornoch Academy, UK. He was selected to represent the UK at the Science on Stage conferences in 2017 and 2019. He has presented Science on Stage webinars on microscale chemistry and using magic to teach science.

Bob Worley, FRSC, is the (semi-retired) chemistry advisor for CLEAPSS in the UK. He taught chemistry for 20 years, and in 1991 he joined CLEAPSS, which provides safety and advisory support for classroom experiments. In carrying out these duties, he gained an interest in miniaturizing experiments to improve safety and convenience.