PRECIPITATION AND PUDDLE CHEMISTRY

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ABSTRACT

In order to be accepted as a useful technique in practical chemistry, the microscale technique has to offer more to the teacher and students than the traditional method. This account precipitates of diffusing shows that the understanding of the mechanism can be enhanced with extremely simple equipment. The technique uses worksheets which are laminated or inside a polypropylene folder, so the student can see the instructions, while at the same time placing puddles of water or solutions on the plastic hydrophobic surface. Salts are added to the



puddle, dissolve and migrate via diffusion to form a line of insoluble salt. An examination of the mechanism for precipitation incorporates a discussion on the Johnstone Triangle, encompasses many of the core ideas of chemistry developed by Professor Peter Atkins and the amazing videos produced by Roy Tasker. It shows how even the wording of exam syllabuses can be influenced by descriptions of events at the nano-level, and the relevance of precipitation to students. The evidence of solvated ions is demonstrated by a simple conductivity indicator. This article is based upon a keynote lecture given at the 10th International Symposium on Microscale Chemistry (ISMC) held at the University of the North West, Potchefstroom, South Africa. [African Journal of Chemical Education—AJCE 9(3), November 2019]

INTRODUCTION

The simple procedure, using "puddles" allows teachers and students to carry out "test tube" reactions on an inexpensive polymer surface such as,

- a polypropylene folder in which the instructions are inserted,
- a laminated card (or paper) encapsulating the instructions, or
- a polystyrene Petri dish on a mostly white background.

This article concentrates on the formation of precipitates when two soluble salts in water are mixed together and another salt suddenly appears as solid, so that the liquid is no longer transparent. Other chemical reactions such as displacement and acid/base can also be carried out in puddles and given the same comprehensive treatment that precipitation receives here.

THE MICROSCALE APPROACH

There were originally 4 strands to the microscale approach. These are shown in the first column of Table 1. As teachers and technicians in the UK tried these methods, further advantages soon became apparent as shown in the second column. As one teacher said during the 10th ISMC in South Africa "I felt really focused on what I was doing and in the chemistry taking place!" Other comments made in another workshop that resonated with me were "In a little I can see a lot", "That is just beautiful" and "That saves me doing a lot of washing up".

Table 1

	Initial reasons for microscale techniques	The added attractions of carrying out microscale procedures
1. 2. 3. 4.	It improves safety in science education It reduces costs in equipment in materials It assists in instigating green and environmental concerns It promotes practical science in developing countries	 It improves classroom management Students are more focused It helps to reduce overload on the short-term working memory so students work efficiently allowing the teacher more time to teach. It is visually very pleasing It helps students develop improved understanding of difficult chemical concepts New and original experiments are available

What is a puddle?

The puddle is formed by adding several drops of water or an aqueous solution onto a hydrophobic surface such as a polypropylene sheet where the contact angle is over 90° (Fig 1). The angle is slightly smaller with plastics such as polystyrene, polythene and polyesters (the covering on laminates) but the experiments still work.

The addition of organic reagents reduces the angle making the puddle spread out. The contact angle for water on glass is as low as 15°, so the droplet really spreads out.



Fig 1 A puddle of 1 M copper sulfate solution on polypropylene showing the contact angle just greater than 90°.

A simple precipitation reaction (uses in ion analysis)

Simple precipitation reactions are straightforward to perform on the plastic sheet. In Fig 2, a puddle of 0.1 M copper sulfate is placed on the surface of the plastic. This is achieved using a dropping bottle or a plastic transfer pipette. A small test tube, taped to a bottle to hold the pipette, reduces cross-contamination between bottles when using transfer pipettes. Beneath the plastic are the instructions. The printed square on which the puddle is placed is half-grey and half-clear. This means the student can see any white precipitates. Two drops of 0.4 M sodium hydroxide solution are added and the blue precipitate of copper(II) hydroxide appears.



Fig 2 A simple precipitation reaction carried out on a plastic sheet showing the formation of the precipitate of copper(II) hydroxide

Fig 3 shows the full sheet, which includes tests for the ions specified by one English exam board



Fig 3 The full sheet showing test on positive ions

Steps 1, 2 and 3 introduce the technique. Students can then use the technique on the other salt solutions providedⁱⁱ. That Step 8, to test for ammonium ions, produced a result, was a surprise to me. If there is no precipitate, it could be an ammonium salt. Traditional methods require boiling the salt solution in sodium hydroxide and the gas evolved is ammonia, which is alkaline. However, the reaction can proceed at room temperature. Alkaline ammonia gas diffuses onto the moist red litmus to change the colour to blue. If none of these tests provide a conclusion on an unknown solution, then sodium or potassium ions are present. A similar approach can be carried out with testing for negative ions such as chlorides, sulfates, etc.ⁱⁱⁱ

The procedure can also be used to establish solubility rules for salts.

ISSN 2227-5835

Why should teachers in the UK care about this approach? After all, they are lucky enough to have all the right equipment in their schools. In order to be accepted over any traditional methods, the microscale approach has to offer more.

By seeing and reading the instructions directly, the student is focused into the experiment and teachers in the UK have found classroom management easier. This style of instruction sheet is called an Integrated Instruction Sheet [1] as it brings together diagrams and numbered, limited instructions together rather than a list of written instructions. It is thought it reduces the load on their short-term working memory, allowing the student to work on their own without asking the question "what do I do next"? Also, teachers should see a green agenda as there is less waste.

A More Advanced Technique for Precipitates (Uses in Challenging Misconceptions)

To students seeing the precipitation reaction for the first time, it must seem like magic: two colorless liquids on mixing suddenly produce a white (and sometimes a colored) solid. Teachers spoil the student by making up solutions, which are transparent and often have no color, for them to simply add together. To most students, the colorless, transparent solutions appear to be water, nothing else – it is what they are familiar with. (This is the long term-memory providing the student with information of what they are seeing.)

In this microscale procedure, the student actually inserts the solid at each end of the puddle and students see the solid "disappear" as it dissolves.

Fig 4 shows the work sheet for this procedure. Any pairs of solids which dissolve in water and when mixed form a precipitate can be used. (Powdered sodium hydroxide can be used but it is a very corrosive solid and absorbs water quickly so better not used by students.)

ISSN 2227-5835

AJCE, 2019, 9(3)



Fig 4 A worksheet, with integrated instructions and diagrams, which is inserted into a plastic folder or laminated

Figs 5 to7 show some photos of what students see.



deeper blue basic copper(II) carbonate. I am not convinced; they seem the same color.)

Fig 7 shows the formation of the silver iodide, which is a beautiful cream/yellow color. The line of silver iodide forms very quickly. Silver iodide is very insoluble in water and only tiny amounts of silver nitrate and potassium iodide (two expensive chemicals) are required.



Fig 7 Producing the precipitate of silver iodide

The student has seen two salts dissolve and then another salt appear as a band in the puddle.

Already the microscale approach is showing more than just adding two clear liquids together. More

information can be obtained when questions are asked about how and why this happens.



ISSN 2227-5835

This procedure for showing precipitates was first demonstrated at the 8th ISMC in Mexico City. Professor Bruce Mattson saw how it could be used with his first year university students and subsequently produced a paper for the Journal of Chemical Education. [2] We were honoured on having the procedure featured on the front page of the May 2019 issue (Fig 9).



Now text books will quote formulae in equations to represent the reactions:

$$\begin{split} &KI(aq) + AgNO_3(aq) \rightarrow AgI(s) + KNO_3(aq) \\ &CuSO_4(aq) + Na_2CO_3(aq) \rightarrow CuCO_3(s) + Na_2SO_4(aq) \end{split}$$

These equations representing precipitation reactions lead to all manner of explanations from students such as "the salts want to change partners". Giving particles human attributes is known as anthropomorphism. Energy changes drive chemical reactions, not romantic liaisons. Research has shown that state symbols such "(aq)" are very confusing to students. Water is at the center of these reactions and links the Explanation of these reactions with the Representation.

Interlude 1: The Johnstone Triangle



ISSN 2227-5835

Experienced teachers of chemistry will move around the Johnstone Triangle with ease, changing from the macro event to representation with equations, working out yields, etc and to explaining the situation with ions, solvation and diffusion, etc. Students will not be able to do this and after the initial joy of chemistry through macro observations, they will find the going tough at first.

I believe the microscale approach assists with "Explanations" and "Representation". First water, the substance often missing from equation but necessary for the reaction to work, needs to be addressed.

Water

Students have to come to terms with several initially difficult concepts which fly in the face of common-sense, that deep-seated reservoir of facts set in our long-term memory. Matter consists of very tiny particles (Peter Atkins' Core Idea number 1 in Interlude 2), yet ice is a solid block and diving into water can hurt. How can water and ice possibly be made of small particles?

Fifty years ago state symbols in equations were not used in school teaching; we would write equations as

$$KI + AgNO_3 \rightarrow AgI \downarrow + KNO_3$$

and the downward arrow (\downarrow) , representing the precipitate, was not always required either.

Now state symbols, in this case "aq" and "s", need to be added. The ionic equation is often required and students need to understand that potassium and nitrate ions remain in water as "spectator" ions, taking no actual part.

$$I^{-}(aq) + Ag^{+}(aq) \rightarrow AgI\downarrow(s)$$

ISSN 2227-5835

Teaching this material is not easy. There is also the recurring question from some students, "Why do I need to know this?" (See Interlude 4)

Visualizing water at the sub-micro or nano level is very difficult but the computer simulations by Roy Tasker^{iv}, in Fig 11, help. The screen shot shows ice with water molecules in a rigid but vibrating structure, the random movement in the liquid and faster moving molecules in the vapor along with the formula and state symbols. If the 0.9 cm³ (Fig 1) puddle suddenly became a vapor, it would occupy a cube with sides about 11 cm long. That is 1,333 times the volume of the puddle. The particles are, in comparison to their size, very far apart, the particles themselves do not expand (a common misconception)! That there is nothing between these particles in the gaseous phase is also difficult to grasp.



Fig 11 Models of water molecules

Water consists of tiny particles called molecules

The water particles are called molecules and these contain smaller particles, hydrogen and oxygen atoms in the ratio 2:1 (H₂O). The volume of the puddle (Fig 1) is about 0.9 cm³. That puddle will contain 0.3×10^{23} molecules; a huge number of molecules. It is a number which we all find hard to visualize. There are far more molecules of water in that puddle than the number of stars you can see from our planet!

Water molecules are moving

In ice, the molecules are fixed in position but vibrating; in the liquid and gas they are vibrating, rotating and moving. The puddle of water might appear stationary but inside there is a seething environment of motion, with energy taken in from the surroundings and spread amongst the various states of energy (See Fig 12). Some molecules have so much energy that they break away from the surface. Liquid crystals which change color with temperature can be incorporated into the plastic surface on which the puddle stands. What happens to a puddle of water on this plastic? See Fig 12.





The temperature range of these thermochromic plastic sheets, from red to blue, is 5°C. This is within the 20 - 25°C range.

Fig 12. The Puddle of water on thermochromic plastic. Room temperature is "green". At the edge of the puddle is a "red" cooler area as energy is drawn into the water. The puddle itself is cooler still as energetic particles leave the puddle (evaporation). Over 24 hours, the puddle would "disappear" due to evaporation.

Water molecules are polarized (Fig 13)

If an electrostatically charged acrylic rod, rubber balloon or even a comb (after combing your hair) is brought close to a stream of water, the path of water is deflected. Distortion can also be seen in a puddle (Fig 14).



So there is a side richer in electron density (slightly -ve or Fig 13: A model of the water δ -) and a side which is poorer in electron density (slightly +ve or

molecule illustrating polarization

ISSN 2227-5835

 δ +). Water molecules are attracted to the opposite charge on an acrylic rod or nylon comb and the stream of water deflects or the puddle is distorted. This electrostatic attraction is between two particles of opposite electrical charge. It is energetically more stable. Reduction in the energy of a system is a part of the reason why chemical reactions occur (the other concerns entropy). (These are Peter Atkins' Core Ideas number 6 and 7 in action)

Polarization is explained by assuming the electrons in the bonds in a bent water molecule are not shared equally, they are polarized (These are Peter Atkins' Core Ideas number 3 and 4 in action and it leads to Core Idea number 5)



ISSN 2227-5835

Water dissolves many chemicals

These pictures in Fig 15 from Roy Tasker's website show how the water molecules surround the ions. For sodium ions, the oxygen atoms (with a slight excess in negative charge) in the water molecules surround the positive sodium ion. The hydrogen atoms in water molecules surround the negative chloride ion.

A substance like calcium carbonate has ionic bonding but is not soluble in water. In this case the attraction between the ions far outweighs the energy of attraction between the ions and water molecules.

There are non-ionic solids which are soluble in water. The obvious ones are glucose and sucrose. These molecules contain numerous –OH groups and water molecules solvate these groups breaking up the crystals.

There is one notable difference between sugar solution and salt solution; **the ability of salt solutions to conduct electricity.**



Interlude 2: Peter Atkins' Core Ideas of Chemistry [4]

- 1. Matter is atomic
- 2. Elements display periodicity
- 3. Chemical bonds form when electrons pair (and the pair of electrons are not always shared equally even forming charged species called ions)
- 4. Molecules have shape
- 5. There are residual forces between molecules
- 6. Energy is conserved
- 7. Entropy tends to increase
- 8. There are (kinetic) barriers to reaction
- 9. Reactions occur when electron(s) falls into a hole (*Atkins discusses 4 types of reaction but ultimately admits there is just one. When a negative electron falls into a positive hole, there is a reduction in potential energy to a more energetically stable state.*)

Using a Conductivity Indicator for more evidence



Here you have direct evidence that solvated ions from the dissolving ionic solids allow electricity to pass through the solution by means of electron transfer (redox reactions, specifically electrolysis) reactions at the electrodes. The influence of the electrode is very localized (See Interlude 3).

An issue to be resolved now is whether the solid salt consists of neutral molecules or charged atoms (ions). An important piece of evidence suggesting that the solids are ionically bonded is that salts conduct electricity when molten. It is traditionally a difficult procedure in schools but the microscale approach makes it accessible^{vi}. Further evidence has been supplied by

X-ray crystallography.

Interlude 3

When an ionic compound is melted or dissolved in water, the ions are free to move about within the liquid or solution. These liquids and solutions are able to conduct electricity and are called electrolytes. Passing an electric current through electrolytes causes the ions to move to the electrodes. Positively charged ions move to the negative	The statement is mistaken in emphasizing that the electricity causes the ions to move to the electrodes. The solvated ions are already free to move about the solution.
electrode (the cathode), and negatively charged ions move to the positive electrode (the anode). Ions are discharged at the electrodes producing elements. This process is called electrolysis.	The statement has arisen because writers of the syllabus have taken the macro
Fig 18	observation of electrolysis and applied it
The Statement in Fig 18 is from an English	directly to the explanation at the nano-level.
Examination Board Chemistry syllabus for	Teachers' knowledge of what happens at
students up to 16 years old ^{vii} .	electrode surfaces is not well founded.
	Helmoltz double layers are hardly taught,
	even in University courses, but the range of
	influence of the charge on the electrodes on
	diffusing water and solvated ions away from
	the electrode, is as low as 10 ⁻⁵ mm, ie, 10nm;
	it does not span the whole distance between
	the two electrodes.

How does the Diffusing Precipitate (Fig 7) occur?

Electrostatic attraction

The precipitation reaction is an example of the interplay between electrostatic attractions between ions and water molecules at the nano-level. **Electrostatic attraction between oppositely charged or partially oppositely charged particles is fundamental in understanding chemistry.** It may become more sophisticated and more mathematical but in schools, the foundations have to be laid. Electrostatic attraction is present between sub-atomic particles, in covalent, ionic and metallic bonds, in solvation between ions and solvent molecules, complexes between ligands and metal ions, in attraction between molecules (eg, hydrogen bonds and dipole attractions) and even between molecules of inert gases.

Water

The structure of water is fundamental to dissolving (Martin Chaplin^{viii}). The bent structure of the molecule, lone pair of electrons and the electrostatic attraction of electrons towards oxygen in the O-H bonds all come together to make the molecule have a dipole. Evidence for this can be shown in deflecting a narrow stream of water by bringing a rubber balloon close to it after rubbing the balloon on a woollen sleeve to develop an electrostatic charge on the balloon.

Macro event 1:

The solids are outside the puddle of water

Nano-level explanation

The solids comprise of charged particles called ions. (Evidence; when molten, these salts conduct an electric current). The ionic bonds are represented in 2 dimensions in Fig 19. Potassium +ve ions(blue) and iodide –ve ions (mauve) are in a crystal structure held together by electrostatic attraction between ions. Silver +ve ions(grey) and nitrate –ve ions (yellow) are in a crystal structure held together by electrostatic attraction between ions.



Symbolic representation

KI(s), $AgNO_3(s)$. Perhaps a better representation is $K^+I^-(s)$ and $Ag^+NO_3^-(s)$? This symbolic representation was once tried in the UK but then seemed to fall out of fashion. It was just too difficult for the majority of students to understand. The words "atoms" and "molecules" are even familiar to primary school children and so fully established in their long term memory. The term "ions" is not and introduced in secondary education. This seems to make acceptance difficult.

Macro event 2:

The solids are pushed into the puddle from either side and the solids "disappear".

Nano-level explanation

There is no magical disappearing; the solids dissolve! The electrostatic attractive forces between the water particles and the charged particles (ions) overcomes the electrostatic attractive forces between the ions in the solid. This is represented in Fig 20. This comes as a big surprise to many students as ionic bonds are often described as strong bonds requiring lots of energy to break them apart because they have high melting points.



Symbolic representation

 $K^{+}(aq), I^{-}(aq), Ag^{+}(aq), NO_{3}(aq)$

The "(aq)" symbol is used to show the solvated ions. It is possible to work out the ratio between the number of water molecules and the ions.

Let us say we use 5 mg of potassium iodide (M(KI)=166 g mol⁻¹) and 5 mg of silver nitrate ($M(AgNO_3)$)=170 g mol⁻¹). That is 3 x 10⁻⁵ mole of each salt as they have nearly the same molar mass. Each salt will produce 6 x 10⁻⁵ moles of particles. With 2 salts present, there are 12 x 10⁻⁵ moles of ions present in the puddle when they dissolve. How much water is there in the puddle? The molar concentration of pure water (1 liter is 1 000 g) is 1000/18 or about 55 mole per liter. Our puddle has a volume of about 1 cm³ so 0.055 moles of water present. So there are 0.055/(12 x 10⁻⁵) or a ratio of 460 water molecules per each ion.

Those close to the ion are held quite strongly by electrostatic attraction. If I were to put those water molecules on the diagram of Fig 20 it would be quite a mess so I leave them out but it can cause a false impression in visualizing what is happening. The concentration of these salts in the solutions is in the region of 0.03 M.

ISSN 2227-5835

Macro event 3:

A yellow solid appears down the centre of the puddle (Fig 21).

Nano-level explanation

To obtain the precipitate line, the charged particles, surrounded by water molecules (ie, solvated ions), MUST be moving through the other water molecules in the puddle. It is a



Figure 21: The precipitate of silver iodide

random movement driven by energy taken from the surroundings and converted into kinetic energy. It is called diffusion.

Fact from a students' long-term memory: Salt (sodium chloride, NaCl(s)) dissolves in water. In

fact, group 1 salts are soluble in water. So the

precipitate must be silver iodide.

The electrostatic attractive forces between the silver and iodide ions MUST be stronger than electrostatic attractive forces between the silver and

iodide ions and water. (It is the only way we can explain the yellow line.) So it comes out of the solution as a solid. There is a problem for you with
 Figure 22: A model of the precipitate of silver iodide
 Fig 22. I have not put any water molecules in the diagram. I try to show the situation with water present in Fig 23 it is now getting very complicated.

Symbolic representation

$$K^{+}(aq) + I^{-}(aq) + Ag^{+}(aq) + NO_{3}^{-}(aq) \longrightarrow AgI(s) + K^{+}(aq) + NO_{3}^{-}(aq)$$



Muddying the waters

There is a saying in the United Kingdom "Muddying the waters". It means to make a situation unnecessarily complicated and less clear. To ensure that Fig 22 is clear and not complicated, all the water molecules have been omitted. In this case, though, water "muddies" the vision we have in our minds of what is happening. Even Fig 23 with water molecules added is simplified as it is drawn in 2 dimensions. The atoms are not circles but spheres and there are tens of water molecules more that have been missed out. The center shows the silver iodide precipitate with water attached to the surface and around them are the spectator ions.



Fig 23:

CONCLUSION

Alex Johnstone (Interlude 1) said that when introducing a topic, only deal with two corners of the triangle at any one time until you get to revision time. Teachers should be able to move from corner to corner because of their knowledge and experience. In doing so you bring together all of Chemistry into a holistic science as shown by Peter Atkins' core ideas (Interlude 2). It is very easy for students, teachers and even exam-question setters to be side-tracked from what is happening at the nano-level by the macro event and to give particles human characteristics (Interlude 3). Norman Reid wrote an appreciation of Alex Johnstone^{ix}. In it he states "*The aim was to allow the young learners to look at the world around from the perspective of chemistry and to see what insights and understandings chemistry offered in making sense of their world.*" [5] (See Interlude 4)

I started teaching chemistry in 1970 when we made precipitates by mixing two liquids together. On starting the microscale chemistry project, I became more focused into the micro events happening, and I began to understand the nano-level interpretation. I doubt that most students at school level will ever get this far, but teachers need to. Teachers need to be ahead of the game. There will be some students who do understand and they may be the ones to go on to be chemistry teachers, chemists at University or in Industry. One of the pleasures in teaching is after 40 years, former students come back and say, thank you. It may only be a few but it makes the profession so rewarding.

Interlude 4: Why is precipitation of solids in chemistry important?

"Why do we have to know about this?" is a cry from students. Water is necessary for life. Our bodies are on average between 57 and 60% water and as high as 70% in infants. Any water we consume needs to be free of biological and chemical additives so that humans and animals are not poisoned. Precipitation is used to clean water of chemicals, both natural and polluting, such as heavy metals from naturally occurring rocks, land water and in the sewer system. The sea precipitates out carbon dioxide as calcium carbonate and keeps the pH reasonably constant. A decrease in sea-water pH affects coral growth and affects the ecology of the system.

Precipitated mordants are used in the dye industry. They are used as pigments in paint.

ACKNOWLEDGMENTS

I would like to thank Dr Marie du Toit and the other organizers of the 10th International Symposium of Microscale Chemistry for inviting me to the symposium. I would like to thank Professor John Bradley for inviting me to contribute to this publication and his continuing encouragement to me in developing microscale techniques. I would like to thank Professor Bruce Mattson of Creighton University, who initiated a spark in my brain to research these techniques further and for publishing this idea in the Journal of Chemical Education. I would like to thank Steve Jones, Director of CLEAPSS in the UK, for enthusiastically supporting me in this venture to delve further into alternative techniques in practical chemistry. The aim to make it safe and enjoyable. I would like to thank Mary Owen, Senior Technician at CLEAPSS, who has contributed so many ideas to the presentation of the equipment.

(http://science.cleapss.org.uk/) in the UK. He studied chemistry at the University of Sheffield. He then taught students for 20 years, 16 years as Head of Department at Norwich School.

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In 1991, he joined CLEAPSS as the Chemistry advisor.

In carrying out these duties, Bob gained an interest in miniaturising experiments. His enthusiasm was supported by CLEAPSS and the family of international chemists who support the International Symposia on Microscale Chemistry.

You can see more about these procedures and how they help students understand chemistry on www.microchemuk.weebly.com .

ⁱⁱ Iron(II) sulfate is a green solid. Do make sure it is green. When damp it oxidises to brown iron(III) salts. Some text books recommend dissolving the green solid in 1 M sulfuric acid. To obtain the precipitate, extra sodium hydroxide solution needs to be added to neutralise the acid. I prefer making the solution in water immediately before the students use it.

ⁱⁱⁱ If readers would like a copy of these sheets, then please send a communication to bob.worley@clepass.org.uk ^{iv} <u>http://www.scootle.edu.au/ec/pin/HVFJXB?userid=71715</u>

^v <u>https://youtu.be/JxXDKk6H-NQ</u>

^{vi} The evidence that salts contain electrically charged atoms (ions) is their ability to conduct electricity when molten. You can find a microscale version of the electrolysis of molten lead bromide (0.5 g used) on

<u>https://youtu.be/LwwmRP8Zpaw</u>. If lead salts are banned from use by your employer, then silver bromide may be used in place of lead bromide.

vii https://www.aqa.org.uk/subjects/science/gcse/chemistry-8462

^{viii} <u>http://www1.lsbu.ac.uk/water/martin_chaplin.html</u> (go to site index). This site is a most comprehensive website about water.

^{ix} A tribute to Professor Alex H Johnstone (1930–2017) His unique contribution to chemistry education research Norman Reid, <u>https://doi.org/10.1515/cti-2018-0016</u>

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