Guide for contributors to the Australian Journal of Education in Chemistry

Introduction

The Australian Journal of Education in Chemistry publishes refereed articles contributing to education in Chemistry. Suitable topics for publication in the Journal will include aspects of chemistry content, technology in teaching chemistry, innovations in teaching and learning chemistry, research in chemistry education, laboratory experiments, chemistry in everyday life, news and other relevant submissions.

Manuscripts are peer reviewed anonymously by at least two reviewers in addition to the Editors. These notes are a brief guide to contributors. Contributors should also refer to recent issues of the Journal and follow the presentation therein.

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Articles should not exceed six pages in the printed form including tables illustrations and references - ca. 5000 words for a text only document. Short, concisely written articles are very welcome. Please use headings and subheadings to give your article structure.

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2. On another separate page provide an abstract of 50 to 100 words;

3. All photographs should be scanned and saved in JPEG format.

4. All chemistry structures, and schemes should follow the guidelines set for ACS publications. It is preferred that Schemes, Tables etc be arranged to fit in a column 7 cm wide, although full page width will be accepted.

Reference Styles

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In this issue ……….

**Kabapinar** from Marmara University in Istanbul (p. 5) investigates an intermediate strategy between computer animations and simulations (to which some students may not have access) and single figure textbook diagrams (which can leave so much to interpretation) to represent phenomena at the sub-microscopic level. Her study probes prospective teachers' perceptions of stepwise multi-frame illustrations of events such as chemical change upon collision of molecules. In reporting the mixed bag of findings, the author recognises that this is a preliminary study that needs considerable follow-up.

Few would deny the enormous challenge of teaching the concepts related to the structure of matter to 13-year-old students with negligible prior chemistry conceptual knowledge. **Korolija and Mandic** at the University of Belgrade, Serbia (p. 11) describe their strategies for doing so, in a framework designed to present the atom as a continuously developing model, with a focus on how and why the model changed with time. The quality of student questions at the end of the lessons suggests that at least some have their curiosity aroused.

Does the mixing of a solution of a lead salt with a solution of potassium iodide produce a precipitate of lead iodide, PbI₂? **Monković et al** from Sts. Cyril and Methodius University, Macedonia (p. 17) report an interesting investigative demonstration that uses IR spectroscopy to convince that the precipitate formed with lead nitrate is different from that produced with lead acetate. The hydroxy iodide formed in the latter case is probably a consequence of the solution pH.

In today's nano-science world, it is timely that **Kumar**, at Florida Atlantic University, USA (p. 20) has chosen to probe the understandings of related concepts amongst prospective teachers in an undergraduate science education course. Problems are identified with understanding scale, as well as meaning of terms, including the etymology of 'nano'.

**Lim et al**, Deakin University, Victoria, (p. 23) report an experiment accepted into the APCELL database involving ³¹P and ⁷⁷Se NMR spectra to monitor the extent and regioselectivity of oxidation of tris(2-diphenylphosphinoethyl)phosphine on addition of sulfur and selenium. As for all experiments in this database, the report includes a detailed summary, an educational analysis, and an evaluation of the student learning experience.

**Sun**, Baotou Teacher’s College, Inner Mongolia, China, (p. 27) proposes calculation of the sum of electronegativity difference of an atom bonded to a benzene ring an other atoms or groups bonded to this atom as a means of categorising whether substituents are *ortho-, para-, or meta-*directing. Some weaknesses in the method are pointed out.

**Pare et al** from Madhav Science College in India, along with **Fogliani** at Charles Sturt University (p. 30) describe an undergraduate general chemistry experiment in which the effects of various parameters on the pseudo first-order rate constant of ZnO-catalysed degradation of acridine orange dye are studied. The experiment gives students an insight into the mechanism of the photocatalytic degradation, and how a catalyst works. The reaction can be taken as a model of photocatalytic degradation of pollutants in the treatment of coloured waste waters.

**Palmer** at Charles Darwin University (p. 33) gives us a fascinating look into science and society in the second half of the eighteenth century and a little beyond through his readings about Richard Watson, Bishop of Llandaff in a variety of books – some very old and rare, including some written by Watson himself. This is a most unusual historical article.
Editorial

Our education revolution: an opportunity for science education

The planets are in alignment. Our new prime minister, Mr Kevin Rudd, campaigned heavily before the recent election on a promise of an ‘education revolution’, promising that education would be seen as an investment in Australia’s future productivity and well-being. Immediately after the election, he seemed to verify his intent by appointing the deputy prime minister, Julia Gillard, as the Minister for Education and Minister for Employment and Workplace Relations (as well as Minister for Social Inclusion). This is an eminently sensible linkage (if she can manage such a workload). And no doubt education has as high a status as it has ever had.

Meanwhile, science is also enjoying a high status. Witness the focus on the science of the recent debates concerning global climate change, and GM crops. Witness the dependence of the previous government on a report from Australia’s chief scientist, Mr Jim Peacock, with regard to the siting of the proposed new paper mill at Bell Bay in Tasmania. Witness the status among the public of Nobel prizewinners Peter Doherty and Barry Marshall. Witness that Ian Frazer was 2006 Australian of the Year, and that in 2007 Tim Flannery again kept the sportmen and sportswomen at bay to win the same award. Witness that some states are offering financial incentives for young people to graduate as science teachers. Witness that the President of the Business Council of Australia, Mr Michael Chaney, who is also Chancellor of the University of Western Australia and a director of some of our biggest companies, called for science teachers salaries to be upgraded to $100 000.

Let’s ignore for the moment the rumours that the Carrick awards for teaching excellence at the tertiary level will no longer be presented (because they are rumours).

The time is right for the science education community to win support to make a great leap forward. All that it needs to do is to reach some consensus on the priority areas for change, evolution or revolution. Compulsory enrolments in science at secondary schools? Revision and/or standardisation of curricula? Four-year degrees at the tertiary level? Upgrade the number and quality of science teachers? Bring back the focus on laboratory work and field experiences? Re-think the nature of laboratory work in schools and universities? Think through how best to take advantage of the new electronic technologies? Devise ways to increase the numbers of science graduates? Should the objective be to produce more Peter Dohertys and Barry Marshalls, or to develop science literacy (whatever that means) in the general population (if it must be one or the other)?

So there: if we make up our collective mind what is important for the way ahead, we may never be heard with as much empathy as now. Speak with diverse voices and diverse opinions and the opportunity may be lost.

Chemistry educators, life science educators, physics educators, geology educators (I’m sorry to compartmentalise here, when the boundaries are now too fuzzy to even recognise in some cases), and even mathematics educators and perhaps engineering educators, should act together to grasp the moment. Are there leaders out there who can make it happen? Of course there are.

RBB

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Prospective teachers’ perception of multi-frame illustrations as a way of teaching chemistry

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Abstract

Multi-frame illustrations on which chemistry concepts are represented by portraying submicroscopic changes that they involve might be used as a potential visual strategy in teaching chemistry concepts. This study presents the results of a case study concerning preservice chemistry teachers’ perception of multi-frame illustrations as a way of learning and teaching chemistry. Findings indicated that the responses of prospective teachers to the multi-frame illustrations were generally highly positive. They think that multi-frame illustrations increase motivation and learner involvement due to their visually appealing nature and enable elicitation of students’ submicroscopic ideas. They also seem to think that as a way of teaching chemistry multi-frame illustrations can promote understanding by representing the causal relationships between submicroscopic particles and displaying all intermediate stages of the chemical phenomenon at the submicroscopic level. However, some prospective teachers felt that preparation of them might be a difficult task.

1. Introduction

Within the domain of chemistry, students are expected to be able to switch between submicroscopic and symbolic representations to explain macroscopic events (Herron, 1996; Kozma, 2000). However, research indicated that students have difficulties in understanding submicroscopic and symbolic representations and translating the representations of chemistry (Ben-Zvi, Eylon & Silberstein, 1988; Driver, 1989; Gabel, Samuel & Hunn, 1987; Johnstone, 1991). Molecular visualisations are suggested as a way of addressing such difficulties. Empirical research supported this as molecular visualisations were found to be effective in helping students to see invisible side of the chemistry by visualising dynamic processes (Schnotz & Kulhavy, 1994; Van Sommern, Reimann, Boshuizen & De Jong, 1998). Connected Chemistry (Stieff & Wilensky, 2003), Vischem (Tasker, Dalton, Sleet, Bucat, Chia & Corrigan, 2002) and ChemDiscovery (Jones, 1999) are few examples. Dynamic processes can also be represented via static visuals appear on printed materials. This could be possible by multi-frame illustrations which are made up of a series of chronologically ordered static pictures each of which portrays a change in a chemical process. A multi-frame illustration on chemical reaction can be seen in Figure 1.

Figure 1. Submicroscopic multi-frame illustration concerning chemical reaction

Several researchers have concentrated their efforts on multi-frame illustrations. They found that such illustrations were effective in promoting retention and understanding scientific phenomena (Mayer & Gallini, 1990; Mayer, Steinhoft, Bower & Mars, 1995). The question of how multi-frame illustrations are perceived by teachers/student teachers has not been addressed. Therefore, this study aimed to introduce multi-frame illustrations as a teaching model and to investigate prospective chemistry teachers’ perception about it.
2. Research Design
In the line of the focus of the study, research questions can be stated as follows;
• Were multi-frame illustrations perceived by chemistry prospective teachers to have some value in teaching chemistry?
• Which aspects of multi-frame illustrations might have been influential in forming their judgement?
• In what ways did prospective teachers think this visual strategy can promote or impede understanding?

Research was carried out during a preservice teacher-training programme conducted between 2004-2005 at the department of chemistry education. A total of 45 prospective teachers took part in the study. They were 4th year students who completed their chemistry courses as well as general pedagogy courses. Data was obtained through four main sources. These were; video-records of the intervention, field notes based on the researcher’s observations, face-to-face interviews with some of the prospective teachers and examples of their work.

Interviews were carried out with 22 prospective teachers during the week following the intervention. They generally lasted for 20-30 minutes. During the interviews an attempt was made to ask identical questions though it was often appropriate to formulate questions in different ways depending on the points raised by the interviewee. All interviews were audio taped and transcribed verbatim.

The criterion used in selecting the prospective teachers to be interviewed was based on their responses to whole class discussion topics. To illustrate, three prospective teachers were selected from those who mentioned disadvantages, three from those who put forward a range of advantages, four were from those who touched upon the difficulties in preparation of multi-frame illustrations. The rest was chosen from those who volunteered.

The data obtained through video-records, observations and interviews were analysed to categorise respondents’ ideas concerning the multi-frame illustrations. After this initial categorisation an attempt was made to identify the existence of specific patterns within these categories. The reliability of analysis was carried out in terms of the degree of consistency among the raters.

3. Teaching intervention on multi-frame illustrations
A five-hour teaching section was devoted for the intervention on multi-frame illustrations. It started by asking prospective teachers to prepare molecular drawings to visualise the process of chemical reaction in pairs. They displayed their drawings in the class and commented the nature of them. They were then invited to test the suitability of drawings to explain the process of chemical reaction and to revise them upon necessity. It was thought that this would help prospective teachers to realise different models used to explain chemical reaction and representations are theoretical models that can be tested, revised and changed in time.

Then a multi-frame illustration (Figure 1) focused on the process of chemical reaction was displayed on the board. Prospective teachers were encouraged to reflect on the nature of the illustration, including design of objects in the frames and representation of key actions in a chemical reaction. This was followed by the researcher explaining the nature of multi-frame illustrations and displaying different examples including dissolving (see Figure 2), chemical equilibrium, phase changes, rusting and chemical bonding.

Figure 2. Multi-frame illustration concerning dissolving of sugar in water. Note that a simple model of dissolving is used for illustration how the process of dissolving occur. The figure does not aim to illustrate why dissolving occurs or the role of water in solvation.
Having discussed their nature and issues regarding their preparation, prospective teachers were asked to create a multi-frame illustration. These were to be used in teaching of evaporation of a liquid, expansion of a solid, the relationship between temperature, volume and pressure of a gas, physical equilibrium. They were asked to work in pairs while preparing multi-frame illustration concerning one of the topics above. All illustrations created were displayed for evaluation. After inadequacies were pinpointed, each group was requested modify its previously created illustration. The intervention continued by introducing the range of uses of multi-frame illustrations as an explanatory, summarising and assessment tool in teaching chemistry.

Then prospective teachers, in pairs, were invited to evaluate multi-frame illustrations as a teaching model. Pair discussions were followed by a whole class discussion which ran around 45 minutes. The discussion was centred around three main issues. There were features and limitations of multi-frame illustration as a model, efficiency of content knowledge of the creators and advantages and disadvantages of multi-frame illustration as a teaching strategy.

4. Findings

Results of data analysis indicated that responses of prospective teachers to multi-frame illustrations as a way of teaching chemistry were generally highly positive. However, they felt that preparation of multi-frame illustrations is a difficult task. Categories determined as a result of data analysis were examined under four main headings below. In the presentation of transcripts some abbreviations were used. In quoting prospective teachers’ responses PT was used together with a number that indicated the identification number. R was used to indicate the statements used by the researcher.

4.1. Motivation and learner involvement

Field notes indicated the picture of highly motivated prospective teachers while preparing and working with multi-frame illustrations. During the interview they were asked how they felt when the researcher introduced illustrations for the first time. Nearly all of them (n=20) commented on the motivating nature of multi-frame illustrations. Some of them (n=12) also noted their own increased involvement. One of them concluded that:

“It is like a game, it just captures your attention and drags you in its world. I found myself wondering what is going on in the frames.” (PT5)

Similar sort of answers were put forward to the question of what might be the advantages of multi-frame illustrations as a way of teaching chemistry. All of the prospective teachers (n=22) attempted to expect that regardless of their educational level students would be highly motivated. Some of them (n=10) also attempted to give reasons for the high level of learner involvement. For example they (n=9) commented upon the appealing nature of coloured design and pointed to the visual appeal of the multi-frame drawing style. Among those, three commented on the value of the drawing style in creating curiosity for the new coming frame and thereby in maintaining learner involvement.

“You just wonder what is going to happen next and move on to the next card with curiosity. This happened to me till I saw the last card.” (PT10)

Having accepted the motivating nature of the multi-frame illustrations, some of the prospective teachers speculated that repeated use of the multi-frame illustrations might no longer create motivation.

4.2. Elicitation of students’ ideas

One of the interview questions was “During the teaching different ways of uses of multi-frame illustrations were put forward. Which ones do you think you can benefit most? And why?”. More than half of the group (n= 15) commented on the value of multi-frame illustrations in making students’ thinking accessible at submicroscopic level and in pinpointing misunderstandings as the following transcript illustrates.

“I imagine myself in using it before the teaching to find out if students posses any misunderstandings. Or I might use it during the teaching so that I can be able to correct students’ misconceptions immediately. It could also be used after teaching to find out if any misunderstanding remains.” (PT9)

Similarly, the rest expressed their view that multi-frame illustrations are useful in enabling students to think and talk about submicroscopic ideas. However, they seemed to focus on the importance of students’ prior ideas only.

“We might use multi-frame illustrations to find out the ways in which students think about chemistry concepts. This is important because only then we can plan our teaching effectively.” (PT17)

All prospective teachers noted the possibility of using multi-frame illustrations for elicitation purposes. They were aware that the strategy has some value in making learners’ thinking accessible at submicroscopic level.

4.3. Developing understanding by visualising chemical phenomenon at the submicroscopic level

On the basis of classroom observations, it is possible to say that all respondents commented favourably on the submicroscopic nature of multi-frame illustrations. This perception was confirmed by interview data. When prospective teachers were asked to explain “In what ways multi-frame illustrations can promote understanding?”, all of them noted that multi-frame illustrations focused their thinking on submicroscopic particles and their behaviour. Two examples were;

“Because atoms and molecules were the main characters in each card, I automatically started thinking about the way they move and collide with each other ” (PT2)

“Particles are animated in the cards. This gives you the feeling that you are there too and see them. They help you to base your thinking on particles” (PT3)

The stepwise nature of multi-frame illustrations was also
seen as an important aspect of the strategy to promote understanding by most of the prospective teachers interviewed (n= 19). However, they indicated different notions concerning how this aspect improves students’ understanding. Majority (n= 15) stressed the possibility of representing the causal relationships.

“Chemical phenomenon is portrayed step-by-step. Students can easily build the relationships between cause-and-effect and understand it better.” (PT20)

They also commented on the possibility of displaying all intermediate stages of the chemical phenomenon at the submicroscopic level through this visual strategy.

“All intermediate stages, even those we can not express verbally, are possible to portray in this strategy. What could we want more? This is it, this is understanding.” (PT19)

The rest (four out of 19) on the other hand, touched upon a rather different point. They put forward that stepwise feature of the illustration helps students conceptualise chemical phenomena as being endless. On further probing, it became clear that they perceive multi-frame illustration representing the reality. They thought that it reflects the invisible world existing somewhere out there and failed to perceive multi-frame illustration uses scientific models, which are theoretical, constructs of chemists’ interpretation of nature. The following interview transcript illustrates this;

R: You said stepwise feature of the illustration helps students conceptualise chemical phenomena as being endless. Does this help or hinder students’ understanding?

PT: It does help understanding of course.

R: How does it help understanding?

PT: First you know the event and how it happens from your daily life. Then somebody tells you why that event occurs in that way by using tiny invisible characters. It is difficult to believe. But then that person makes them visible with this new strategy. These characters are so visible that you can empathise their world. This is exactly what happens to me. After seeing a number of cards in chemical reaction I thought that this is only a fragment of a big story. This story does not end there. Then I started imagining what might happen in the previous and subsequent cards.

R: How did this further imagination help your understanding of the phenomenon?

PT: I already understand the phenomenon but it helped me to think that they are all there even if I do not draw them in the cards. I gained a general way of looking events, whatever the phenomenon is. We can make students to develop such a general thinking too by using multi-frame illustrations. They will know that even if they cannot see, particles are there. They will surely believe and feel the existence of this new imaginary world. (PT8)

This prospective teacher seems to believe that equipped with this conceptualisation, students might believe and feel the existence of this new imaginary world without any blockages in their thinking. She herself believes this imaginary world as if it is real and possibly thinks of multi-frame illustrations as representing the facts.

During interviews prospective teachers were asked to explain in what ways multi-frame illustrations might impede understanding. Majority of them commented on its value of promoting understanding and seemed to think that it does not impede students’ understanding. Having accepted potential benefits of it, five prospective teachers drew attention to a potential drawback due to its visually based nature as one of them explained;

“Students never forget pictures. In one of my chemistry classes, I noticed that nearly all students left big spaces between particles of a liquid. Then I realised that I left some spaces between the particles as I was in a hurry to finish the lesson on time. Although I corrected my earlier drawing and explained that it was wrong, a few students still remained to use the first drawing” (PT15)

The findings indicated that majority of prospective teachers commented on the benefits of multi-frame illustrations in learning and teaching chemistry. This might be strongly related to their preferred learning styles. They might be visual learners and therefore might prefer visual learning environments.

Some prospective teachers (n= 5), on the other hand, noted that multi-frame illustrations might cause misconceptions about particles. On probing, they made clear that the basic tendency might be to attribute anthropomorphic qualities to particles.

“Atoms and molecules are animated in the illustration. Students might think of them as alive.” (PT4)

Indeed, multi-frame illustrations have a potential to empower anthropomorphic views. Drawings of particles as having features or feelings of living things (such as angry eyes, happy face and so on) might be one source. Another source might be the language used in captions. Careful use of language and drawings might diminish the possibility of such anthropomorphic view.

4.4. Ease of preparation and use

Field notes and observations highlighted the picture of tense atmosphere when prospective teachers were requested to create a multi-frame illustration. Nearly half of the prospective teachers (n=12) seemed confused upon the request of preparation. On probing, they underlined the difficulty of preparation of multi-frame illustrations. Interview data revealed that the reasons behind the difficulty of preparation were of different in nature. Some of the prospective teachers (n= 4) explained their state of being unconfident by their feeling of having not enough knowledge on the topic at the submicroscopic level. Typical comments were:

“I felt very uncomfortable because I thought that I did not know how to explain physical equilibrium at the molecular level” (PT8)

“We were asked to prepare a molecular illustration about
sugar dissolving in water. Suddenly I lost my confidence despite three years of teaching experience. I did not admit it at the class, but actually I did not know how to draw it at the molecular level.” (PT5)

Some (n=3) on the other hand, blamed their poor drawing ability. Observations also confirmed this. These respondents knew what to draw but they did not know how to draw it. Observations also noted the ease with which they completed their illustrations when they were provided information on visual techniques.

The rest (n=5) noted the difficulties that they experienced when planning the illustration. These focused mainly on two issues. One was difficulty experienced in deciding the number of stages.

“Our topic was easy. Salt dissolving in water. We could think of three cards: salt at the bottom, salt starts dissolving and salt ions are distributed evenly in water” (PT12)

The second difficulty identified was representation of actions of the event at the molecular level. Similar to the first one, this difficulty was also reported by the majority of the respondents.

“We knew the relationship between the pressure of a gas and temperature. We knew that we are supposed to show low temperature-low pressure and vice versa. But we had difficulty in representing low pressure till you said low pressure-less collisions with the container.”

For the rest of the prospective teachers (n=8), being informed about the nature of multi-frame illustrations was enough to start preparation. Yet, a small group (n=4) pointed out repeated drawings of multi-frame illustrations cost effort and time.

Contrary to respondents’ perception regarding the preparation phase of multi-frame illustrations, all prospective teachers commented favourably on the ease of use of multi-frame illustrations. Many pointed that once it is prepared it is easier to use it in the class.

“The difficult part is to complete the necessary thinking and drawing. Then you can just put them in front of students’ eyes. They all speak for themselves.” (PT17)

In short, prospective teachers feel that multi-frame illustrations are difficult to prepare, expensive for time and effort but easier to use as a teaching approach.

5. Conclusions and implications

This study has presented a case study concerning prospective chemistry teachers’ perception of multi-frame illustration as a way of teaching chemistry. Results of the case study provided evidence that prospective teachers perceived them to have value in the teaching of chemistry concepts. They seem to focus on two aspects of learning: motivation and visualisation. Most prospective teachers thought that multi-frame illustrations increase motivation due to their visually appealing nature. They also seem to think that such illustrations may promote understanding by visualising chemical phenomenon at the submicroscopic level and representing the causal relationships between particles.

Research indicates that learners have different learning styles and that student motivation and performance improve when instruction is adapted to student learning styles (Butler & Mautz, 1996; Dunn, Giannitti, Murray, Rossi & Quinn, 1989). Based on this, one might argue that this highly positive perception of multi-frame illustrations might stem from prospective teachers’ preferred learning style. It may well be that they had strong preference to the visual learning style. This may affect their perception of multi-frame illustrations that are more focused towards the visual learners. This study did not detect prospective teachers’ learning style. Future research may attempt to find out the relationships between learning styles and perception/use of multi-frame illustrations. Having said that designing intervention on multi-frame illustrations to allow verbal and social interactions might attract all students and produce similar sorts of positive effects on their learning.

The present study highlighted prospective teachers’ fairly limited perception of multi-frame illustration as a model despite of their knowledge on modelling in science. None of them mentioned the success or limitations of multi-frame illustrations as teaching models. On the contrary some seemed to think of multi-frame illustrations as representing the reality rather than modelling of ideas in chemistry. This finding coincides with the findings of Van Driel and Verloop (1999) who investigated experienced science teachers’ knowledge of models and modelling in science. They found that despite their experience some of the chemistry teachers supported the positivist notions of models. As explained earlier, the prospective teachers received instruction on chemical modelling in the previous school term and they were reminded modelling in chemistry prior to the intervention. This attempt seems inadequate for them to build relationship between multi-frame illustrations and modelling. It may become more desirable to assess their knowledge and perception of modelling in science prior to the introduction of multi-frame illustrations as a teaching model. The future research might focus on designing and evaluating teaching intervention as such.

Multi-frame illustrations have been found to be valuable by prospective teachers. However, concern raised during the study centred on issues relating to the difficulty of preparation. The findings highlighted the content knowledge of the creators as being the main reason for the difficulty. In explaining the content knowledge they focused their attention simply on the absence of knowledge on the subject matter. This would be the simplest way of conceptualisation of the content knowledge. Creators also need to have pedagogical content knowledge related with models and modelling. To be specific, they need to differentiate between different models offered in the subject area (for instance chemical bonding) and select the one best works for the theoretical and instructional objectives. In addition to knowledge on modelling,
creating and using multi-frame illustrations require pedagogical content knowledge concerning instructional theories, pedagogical strategies and teaching methods.

A subsidiary reason, which seems to contribute to the level of difficulty in preparation was the drawing ability of the prospective teachers. Providing a few drawing techniques during the method course, however, had some bearing on this issue. Another solution for poor drawing ability might be benefiting from technology to prepare illustrations. Having planned the sequence of the frames, it might be possible to create professional drawings via a drawing program. By doing so, the uniformity in size and shape of the submicroscopic particles or the hackwork associated with repeated drawing of them might not be a problem.

The paper proposes multi-frame illustrations as a teaching model and investigates how prospective chemistry teachers perceived them. However, there is much we have yet to learn about multi-frame illustrations such as whether they are more or less effective than any other strategies at leading learners towards more scientifically acceptable ideas. It is also important to make comparisons between students’ learning resulting from multi-frame illustrations that differ in design and conceptual level. As in any teaching strategy, the nature of teaching intervention is also significant. Therefore, it is important to carry out research to examine and compare the extent and stability of students’ learning fostered by different teaching interventions concerning the pedagogy based on the use of multi-frame illustrations.

6. References

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and follow the links.
Introductory lessons for the teaching topic The Structure of Matter. How to make the first contact with the concepts atomic structure and valence easy and entertaining?

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Abstract
The introduction and explanation of atomic structure and valence at the start of studying chemistry is a very complex and demanding task. This paper presents the introductory lessons for the topic ‘The Structure of Matter’ that may help elementary school students easier understanding these concepts. The introductory lessons were helped by the use of didactic games with models: balloon in balloon - BIB models, balloon and balloon – BAB models, two-dimensional atomic models, molecular model puzzles. Through these introductory lessons the students gained their first ideas of these concepts, they acquired a basis for elaborating the concepts in subsequent lessons, and were motivated to adopt the abstract concepts of this topic.

Introduction
The introductory lessons to any course serve to introduce the student to the new course, its goals and tasks, contents, the approach to be followed, work evaluation, and the way it is organised. The introductory lessons can also be used at the beginning of the study of an extensive teaching topic to enable the student to: obtain background information (provide perspective) about concepts, the content and extent of which they will be covered; stimulate the imagery about a concept or model, the possibilities of presentation. Furthermore, it helps the exchange of experiences, assumptions, and imageries; prepare to gain new knowledge through active learning; assess one’s place and role and assignment in future lessons; stimulate the student to learn by stimulating his/her activities in the teaching process. On the other hand, the introductory lessons are also very important for teachers because they enable them to assess to what extent the concept contents are close to or removed from the students; to identify convenient teaching/learning methods and various aids, as well as student/teacher activities.

This paper presents the introductory lessons for the teaching topic The Structure of Matter, which includes the following concepts: particulate structure of matter, atomic structure, element valence and symbol and chemical bond. The aim of the lessons was for students to gain images about concepts that they will later adopt, e.g. anticipate that:
• The structure of matter is discontinuous,
• Particles of matter move incessantly,
• The smallest particle of an element is an atom,
• Atoms have complex structures,
• Atoms of different elements differ from each other,
• There are rules (valence) for the bonding of atoms,
• Different combinations of atoms are possible during collisions,
• In nature only stable entities of united particles (combinations of particles) are found.

The students start to study chemistry at age 13-14, at the beginning of the development of their formal operations. The importance and abstractness of the concepts from the sub-micro world required approaches appropriate for the elaboration of above mentioned terms (Johnstone 2000, Gabel 1999, Tsaparlis 1997). The use of analogies (Thiele 1994), associations and models (Treagust et al. 2003, Gupta and Parkash 1999, Bentzinger, and Meyer 1994) facilitates the familiarization and clarification of concepts, the definitions of which are not accessible to the senses. The selection of appropriate atom models for the early stage of studying chemistry is very important as the students form their mental image of atom on the basis of models. Misunderstanding of this model may prevent meaningful learning at later stages (Ben-Zvi 1986).

The best way to introduce students to the world of concepts of which they have no previous knowledge, is to encourage maximal activity, independent work, asking questions and seeking answers (Treagust et al. 2003, Denny et al. 2000). The internal motivation of students to study may be enhanced through didactic games (Russell 1999, Milton 2001), which enable spontaneity, freedom of expression, imagination and logical reasoning (Casta-Acuna et al. 1999).

In order to motivate students to study the atomic structure and valence, and to achieve their greatest involvement during the introductory lessons, analogies and models in didactic games were applied.

Methodology
The above mentioned goal was achieved through several steps: the demonstration of various teaching aids, discussion about the stated facts, a demonstrational educational game and an educational game for independent student assignments (team work). Overview of the realization steps of these introductory lessons (elements of the concepts, strategies, student’s activities and teacher’s demonstrations) is given in Table 1.
Table 1. Overview of the realization steps of introductory lessons: elements of the concepts, strategies, student’s activities and teacher’s demonstrations

<table>
<thead>
<tr>
<th>Elements of the concepts</th>
<th>Strategies</th>
<th>Student’s activities</th>
<th>Teacher’s demonstrations</th>
</tr>
</thead>
<tbody>
<tr>
<td>The structure of matter is discontinuous. The smallest particle of an element is an atom.</td>
<td>Historical review of the knowledge about atom from Leucippus and Democritus to the present days.</td>
<td>Observation and discussion about atom models</td>
<td>Photograph of sand beach; Pictures of Leucippus, Democritus and Dalton and their representations of the atom</td>
</tr>
<tr>
<td>The size of atom.</td>
<td>Use of analogies</td>
<td>Finding the connection between the balls and atom symbols and ball and atom radius</td>
<td>Balloons for various sports; Berzelius symbols and names of elements</td>
</tr>
<tr>
<td>The complexity and diversity of the atomic structure of individual elements</td>
<td>Demonstrational educational game with atoms models</td>
<td></td>
<td>Models of atoms: - balloon - Dalton’s model - BIB models of hydrogen and oxygen atoms - modern model - models of subatomic particles</td>
</tr>
<tr>
<td>Demonstrational educational game with atoms models Explosion of the model balloons.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Collision of atoms, their grouping, regrouping, forming of complex particles (molecules); the bonding of atoms; the valence of elements</td>
<td>Demonstrational educational game - forming of molecules models</td>
<td>Observation and discussion</td>
<td>Balloon and balloon (BAB) models</td>
</tr>
<tr>
<td>Educational game for working with groups of students - forming of molecules models</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The demonstration of various teaching aids, discussion about the stated facts

The difficulties that students have in adopting the particulate model of matter are not surprising since it took mankind about 2000 years to develop it (Ben-Zvi 1986). Therefore, at the beginning of this introductory lesson, a short historical review of the idea and knowledge about the atom from Leucippus and Democritus to the present day was presented (photograph of a sand beach – discontinuous structure of matter, pictures of Leucippus, Democritus, Dalton and their representations of the atom) (Smith 1989). In the discussions students used their knowledge of history, mathematics, geography and physical education to suggest reasons why the idea of the ancient Greeks about the particulate structure of matter was neglected till the beginning of the 19th century. Which social, scientific and technical conditions determined that the idea of the atom be revived only after the appearance of Dalton?

There were a large number of inflated balloons of various sizes (on the desks, on the floor, between the desks) in the chemistry classroom. After the afore-mentioned discussion about the atom, the conclusion was reached that the balloons were models of atoms. When analyzing the size of atoms, balls for various sports were used beside balloons (Pinto 1998) above which Berzelius’ symbols of the elements were positioned. The students were successful at finding the connection between the balls and the atom symbols – the ball radius. The students compared the size of the ball and the size of the atom, estimating how many times the atomic radius of a certain element had to be increased in order to obtain the radius of the corresponding ball. At the same time they were connecting knowledge of mathematics and chemistry in an entertaining and simple fashion.

Demonstrational educational game

Two- and three-dimensional models of atoms were reproduced for the educational games. The goal of the demonstrational didactic game was to explain the complexity and diversity of the atomic structures of individual elements. The game started with the demonstration of two balloons. One balloon (an indivisible entity) contained air, while the second one contained air and a smaller internal balloon (balloon in balloon - BIB model). BIB model of atom contained the models of subatomic particles. In external balloon of BIB model were models of electrons while in the internal balloon (atomic nucleus) were models of protons and neutrons. The number of protons and electrons was equal. The question ensued: Which of the two given balloons presents Dalton’s and which the modern model of the atom? In addition to the answer to the question, the students were requested to elaborate their reply. The movement of both models of atom produced a sound only in the case of BIB model: it was thus apparent to the students that some smaller objects were contained inside the balloon. As the students could recognise Dalton’s model (on the basis of the information that the Dalton atom is indivisible, which was obtained at the beginning of the introductory lesson and by the fact that its model will not produce sound during movement) the other should be the modern model (a divisible entity). During the demonstration it was explained that the sound was used only as mean for differentiation of two atom models and that it is not real analogy of the atom.

The discussion about the possibility of checking the correctness of the answers led to a proposal by the students to cause an explosion (fission) of the model.
balloons. After the explosion, an inner balloon appeared in the case of the modern atomic model, as well as the particles (model of electron) from the external balloon (electronic shell). The following ensued: counting of the particles from the external balloon, explosion of the internal balloon (atomic nucleus), observation of two types of particles of the same size and of different color in the internal balloon (nucleus), counting of the particles from the inner balloon, discussion about the different charge of subatomic particles, i.e. about the neutrality of the atom.

The second part of the demonstrational educational game had the purpose of showing that atoms may be bonded, and build molecules by utilizing atomic models (balloon and balloon- BAB models). By constructing models of molecules the students gained the imagery about forming more complex structures as well as information about the concept of valence. The following elements of the game were presented and discussed:

- Collisions of two balloons, connecting of balloons with double-sided scotch tape - the construction of a model of the diatomic hydrogen molecule; adding of the third balloon does not result in new bond (H3 does not exist)
- familiarization with the valence of hydrogen,
- collisions of atoms of various elements – the construction of a model of the four-atom ammonia molecule (by using double-sided scotch tape at certain positions of the model of the nitrogen atom),
- familiarization with the valence of the nitrogen molecule.

Educational game for working with groups of students

In order to enable the students to practise with as many models as possible, and to assess better the basic concepts previously presented by the demonstrational educational game, a didactic game was developed with two-dimensional atomic models of various elements for work in student groups. Atomic models of hydrogen, nitrogen, oxygen, carbon and sulfur were made of colored cardboard (Figure 1). The name and symbol of the element was written on the model. The number of convex and concave surfaces on the model corresponded to the valence of the atom. The colour of the model corresponded to the international convention on the representation of atoms of various elements. Non-metals were chosen for this game as we thought that for presenting the process it is the best to start from atoms (the simplest particle) to molecules (complex particle). Introducing the terms, ion and ion pair would make the introductory lesson on submicroworld particles too difficult.

The selection of atomic models for four individual model groups was made according to the following criteria: the chosen elements are commonly in the curriculum of the first year of studying chemistry; each group contained the same number of elements (three); the atomic models could be used to construct molecular models of elements and compounds that are studied during this year of study.

The number of atomic models of elements per group ranged from six to seven. The molecular models that could be made from them varied in complexity, from diatomic to penta-atomic ones. Four to five molecular models could be constructed from the atomic models in each of the groups. The presentation of the formulae of the molecules of elements and compounds, the models of which could be constructed in the groups are presented in Table 2.

Table 2. Presentation of the formulae of the molecules of elements and compounds in the groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Number and type of the atomic models</th>
<th>Models of molecules that can be formed from them</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>4 H, 1 C, 2 Cl</td>
<td>H2, Cl2, HCl, CH4, CH3 Cl,</td>
</tr>
<tr>
<td>II</td>
<td>3 H, 1 N, 2 O</td>
<td>H2, NH3, H2O, H2O2, NH2OH</td>
</tr>
<tr>
<td>III</td>
<td>3 H, 2 N, 1 S</td>
<td>H2, N2, NH3, H2S</td>
</tr>
<tr>
<td>IV</td>
<td>2 H, 1 N, 3 O</td>
<td>H2, O2, H2O, HNO3</td>
</tr>
</tbody>
</table>

In the didactic game the students:

- noticed and wrote down the valence of the atom presented by the model,
- noticed that the position of convex and concave surfaces on the atomic models of elements do not have to be the same on all models of the same element, although their number (valence) is equal (Figure 1). The aim was to realize that the various positions of the convex and concave surfaces on the two-dimensional atomic models enable the obtaining of a two-dimensional representation of that molecule that approximates to reality – bond angles – when constructing the model. Models of the ammonia molecule could be constructed from the atomic models of both groups two and three, producing two very different molecular models. These could be used later to discuss which of them was closer approximation to the real structure and why,
- constructed molecular models based on atomic models of elements and drew molecular models,
• discussed whether the model constructed represented the molecular model of an element or compound.

During the educational games the students wrote down their observations and conclusions on worksheets. The answers in the worksheets were analyzed and discussed.

In order to assess the effects of these introductory lessons, i.e. what ideas about certain concepts were gained by the students, five-minute exercises (short questions in written form) were held during classes in which the terms were elaborated.

207 Elementary school students (year 7, age 13) participated in the experiment. In Serbia “elementary school” covers the first eight years of schooling, i.e. our students (the 7th year) correspond to lower secondary school.

Results and Discussion
The aim of the demonstration of various teaching aids and atomic models, and the discussion about the stated facts at the beginning of introductory lesson was to show to the students how and why the model of the atom changed with time. The atom is presented as a continuously developing model, the characteristics of which change to accommodate new facts that have to be explained. The intention was to avoid possible misconceptions and wrong mental images of atoms and their structure, which may result from flaws of the applied atomic models. Bearing in mind that these were introductory lessons (which were to be followed by detailed study of the structure of atoms) it was assumed that the use of several different kinds of atom models (simple two-dimensional coloured cardboard models and three-dimensional - balls, balloons, BIB and BAB models) will later facilitate acquiring of images of abstract concept of atom. Experience of many our colleagues and our own is that when only one atom model, e.g. ball is offered to students they wrongly accept rigid ball as real atom.

Through the demonstrational game with BIB models the students gained imagery about the complexity of atomic structure (Table 3). The differences in atomic structure were displayed by exploding BIB models of hydrogen and oxygen atoms. In this lesson elementary school students were introduced for the first time to the most important subatomic particles (electron, proton, and neutron). By means of the model of subatomic particles and the atomic model, the following was shown: the location of particles in the atom; the volume in which the electrons moved in the atom (nucleus shell) was large compared to the size of the nucleus and electrons; the number of negatively and positively charged particles in the atom was equal. The students can count particles and drew the conclusion that the atom is a neutral entity. Balloons filled with ‘particles’ in the nucleus and shell, which moved constantly, as three-dimensional atomic models helped the students to create the concept that the atom was a dynamic entity. Using BAB models students got the idea on the collision of atoms, their grouping and regrouping, forming of more complex particles (molecules) as well as the valence (Table 3).

The students accepted the demonstrational game with BIB and BAB models very well and actively participated in the demonstration of atomic and molecular models, in the discussion and the drawing of conclusions.

Table 3. Elements of the demonstrational game used to introduce students to the concepts of the atomic structure and valence of elements, and student observations and conclusions

<table>
<thead>
<tr>
<th>Game elements</th>
<th>Observations and conclusions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dalton’s and the modern atomic model – work with BIB models (balloon in a balloon)</td>
<td>The atom is indivisible (Dalton). The atom is divisible (modern model).</td>
</tr>
<tr>
<td>‘Atomic fission’ of hydrogen (model)</td>
<td>The atom contains a nucleus and a shell. The atoms of elements differ by the number of particles in the shell and in the nucleus. The atomic nucleus contains one or two types of particles. The number of protons and electrons is equal.</td>
</tr>
<tr>
<td>‘Atomic fission’ of oxygen (model)</td>
<td></td>
</tr>
<tr>
<td>‘Fission of the atomic nucleus’ of oxygen (model)</td>
<td></td>
</tr>
<tr>
<td>Introduction of the concept of the valence of elements</td>
<td>The valence of an element indicates how many monovalent hydrogen atoms it can bind.</td>
</tr>
<tr>
<td>The bonding of element atoms into new stable entities</td>
<td>The bonding of atoms occurs by means of the shell. An unstable particle formed by the bonding of atoms cannot survive.</td>
</tr>
</tbody>
</table>

The construction of a molecular model from the atomic models of elements has great educational significance. When working with models, the students are indirectly introduced to the concepts of valence and chemical bond, basic concepts of the sub-microworld. The various valences of one element indicate the complex structure of its atom to the students and the various possibilities of connecting it with other elements. At the age when abstract thought begins, such exercises may stimulate the development of combinatorial skills.

Table 4. Elements of the didactic game for student team work used to introduce the students to the concept of the valence of elements and the combination of atoms into more complex entities

<table>
<thead>
<tr>
<th>Game elements</th>
<th>Observations and conclusions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Two-dimensional atomic models of various elements</td>
<td>The number of convex and concave surfaces on the atomic model corresponds to the valence of the element. The valence of an element can differ.</td>
</tr>
<tr>
<td>The construction and drawing of molecular models based on atomic models. Total number of molecular models which can be constructed is 13.</td>
<td>The valence of atoms when constructing a molecule should be saturated. Hydrogen is always monovalent.</td>
</tr>
<tr>
<td>The differentiation of molecules based on the constructed models</td>
<td>There are molecular models that are composed of identical or different atomic models.</td>
</tr>
</tbody>
</table>

The elements of the educational game for working with groups, as well as the student observations and conclusions, are presented in table 4. Based on the supplied atomic
models and by using the puzzle technique, it was possible to construct thirteen models (four models of molecules of elements and nine models of molecules of compounds).

Students constructed several molecular models for each of four groups (I-IV) presented in Table 2. The total score obtained for each group of models is presented in Figure 2, while the score of constructing molecular models of various complexities is presented in Table 5.

Table 5. The total score achieved constructing molecular models of elements and compounds

<table>
<thead>
<tr>
<th>Model of the molecule</th>
<th>Formula of the molecule</th>
<th>No of students</th>
<th>No of correct formulae</th>
<th>% of correct formulae</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H₂</td>
<td>207</td>
<td>131</td>
<td></td>
<td>63.3</td>
</tr>
<tr>
<td>N₂</td>
<td>50</td>
<td>48</td>
<td></td>
<td>96.0</td>
</tr>
<tr>
<td>O₂</td>
<td>51</td>
<td>41</td>
<td></td>
<td>80.4</td>
</tr>
<tr>
<td>Cl₂</td>
<td>52</td>
<td>50</td>
<td></td>
<td>96.2</td>
</tr>
<tr>
<td>Compound</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HCl</td>
<td>52</td>
<td>44</td>
<td></td>
<td>84.6</td>
</tr>
<tr>
<td>H₂O</td>
<td>105</td>
<td>102</td>
<td></td>
<td>97.1</td>
</tr>
<tr>
<td>H₂S</td>
<td>50</td>
<td>48</td>
<td></td>
<td>96.0</td>
</tr>
<tr>
<td>NH₃</td>
<td>104</td>
<td>101</td>
<td></td>
<td>97.1</td>
</tr>
<tr>
<td>CH₄</td>
<td>52</td>
<td>48</td>
<td></td>
<td>92.3</td>
</tr>
<tr>
<td>H₂O₂</td>
<td>54</td>
<td>42</td>
<td></td>
<td>77.8</td>
</tr>
<tr>
<td>CH₃Cl</td>
<td>52</td>
<td>40</td>
<td></td>
<td>76.9</td>
</tr>
<tr>
<td>NH₂OH</td>
<td>54</td>
<td>41</td>
<td></td>
<td>75.9</td>
</tr>
<tr>
<td>HNO₃</td>
<td>51</td>
<td>29</td>
<td></td>
<td>56.9</td>
</tr>
</tbody>
</table>

The results indicate successful model construction (56.9–97.0%). It may be concluded that the selected way to form models helped the students to observe the valence of elements easily. The number of convex and concave surfaces (valences) and the complementarity of the model surfaces enabled their simple combination and construction.

By comparing the results of constructing molecular models of elements (the simplest diatomic molecules), it may be seen that very good results were obtained for all the elements (80.4–96.2%), except hydrogen (63.3%). Lower scores for hydrogen might be the consequence of the simple model of this atom and the fact that to construct it the idea of the complementarity of shape has to be disregarded. It was designed in the shape of a full circle, without convex or concave surfaces as indications of valence (Figure 1). The size of the model of the monovalent atom of hydrogen (the smallest one compared to other atomic models) and its use in the construction of a large number of other molecular models, dictated its shape. We were of the opinion that the students would construct a two-dimensional model of the diatomic hydrogen molecule just by placing two disks next to one another, in view of the fact that a three-dimensional model of this molecule (the BAB model) was constructed during the demonstrational didactic game.

The molecular models of compounds consisting of two elements (hydrogen chloride, methane, ammonia, hydrogen sulfide and water) were successfully constructed due to the easy observation of the valence on the atomic models (84.6 – 97.1%, table 5). Molecular models consisting of three elements (methyl chloride and hydroxylamine), as well as molecular models of compounds in which atoms of the same element are connected (H₂O₂), were also constructed very successfully (76.9%, 75.9% and 77.8%, respectively). The success in constructing molecular models was reflected in the fact that the students, while respecting the valence of the elements, the number of available atomic models and to some extent the complementarity of their surfaces, also constructed unexpected molecular models that may be presented by the formulae:

H-N=N-S-H  H-N=N-H  H₂N-S-H  H-O-O-O-H

These models served as a good basis for a renewed discussion that not all atom combinations were stable (they do not exist in nature). Thus, one of the above mentioned goals of the introductory lessons was completely achieved.

With the desire to construct as many molecular models as possible and satisfy all the valences of the element, the students added atomic models of hydrogen, oxygen and chlorine. Some of the constructed models were correct, but were not accepted because they did not obey the rules of the puzzle game – the construction of molecular models only on the basis of the available number of atomic models.

The effects of the introductory lessons in forming the images of atomic structure and valence were accompanied by five-minute exercises (short questions in written form) during lessons in which these concepts were further elaborated. According to the course structure the time span between the introductory lesson and the elaboration of the concepts was 2 to 3 weeks. The students successfully responded to the questions: Which particles are found in the nucleus of the atom? Where are the electrons located in the atom? Are atoms electrically charged? What does the valence of an element indicate? (77.9%, 78.1, 53.9%, and 64.0%, respectively). The results indicated that during the introductory lessons most of students got the initial informations about the structure of atoms and valence. In such way the basis for an understanding of these key chemical terms at sub-microscopic level was provided. After the introductory lessons more complex methods for
acquiring knowledge were used later: discovery learning during the school lessons (independent or guided simple experiments), students’ research projects - application of knowledge in real life, learning by problem solving at an early stage of studying chemistry. Thus, active learning/teaching was used during these lessons on the atomic structure and chemical bond. The students who had introductory lessons were more independent, showed more initiative and positive attitude towards chemistry as it was observed at later lessons.

At the end of the worksheet was the question “What will you ask us at the end of the lesson?” The answers of students indicated their increased interest for further learning. Some typical answers were: How many atoms has the biggest model that has ever been made? Is it in the Guiness book of records? Does every collision of particles gives new particles? What in the real world is represented with the Scotch tape (which connects two balloons in the model)? How many elements do not have neutrons?

Instead of a conclusion
Analysis of the results achieved in five-minute exercises and the results achieved during didactic games with atomic and molecular models indicate that the students accepted this way of introducing concepts of the sub-microworld very well. The application of experiments by which one enters from the macroworld into the sub-microworld at the very beginning of studying chemistry is difficult to implement because of the age of the students and their low level of knowledge. Consequently, solutions for familiarizing students with concepts of the sub-microworld must be sought in other teaching means. The didactic games that were applied during the introductory lesson are one of the solutions that may be applied in the teaching and checking of concepts that are not available to our senses. Educational games of this type can also produce divergent answers which lead to discussion among the students, the development of their imagination and the seeking of support for the defence of one’s opinions.

Introductory lessons for the teaching topic The Structure of Matter, which were carried out as described, enabled partial familiarization of the students with the concepts of atomic structure and valence (as well as chemical bond), increased the students’ internal motivation for further study and thus aided the elaboration of these concepts in subsequent lessons. Therefore, we recommend them as an introduction to the teaching topic The Structure of Matter.

Acknowledgements
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Pinto, G.,(1998), Using balls from different sports to model the variation of atomic size, Journal of Chemical Education, 75, 725-726.

An invitation
The editors invite readers to make contributions to this Journal. As well as papers submitted for peer review, we welcome any of the following:

- Short papers on chemistry topics or concepts, from an educational perspective
- Reflective papers teaching and learning chemistry - general or specific
- Letters to the editor
- Announcements
- Forthcoming events
- Books to review
- News about people or places
Demonstrations as a Tool for Ironing-Out Misconceptions: III. A Note on the Reaction \( \text{Pb}^{2+}(\text{aq}) + 2\text{I}^-(\text{aq}) \rightarrow \text{PbI}_2(\text{s}) \): Exceptions from the \textit{a priori} Expectations

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Abstract

The precipitates formed upon the action of aqueous solution of potassium iodide on aqueous solutions of lead(II) nitrate and lead(II) acetate, respectively, are different. The assumption that in the presence of acetate a double salt \( \text{Pb(CH}_3\text{COO})\text{I} \) has been formed, was ruled out by IR spectroscopy. Some simple additional experiments prove that the precipitate, in the latter case, is actually due to lead(II) hydroxide iodide, in good agreement with the appearance of the IR spectra.

Introduction

In the general chemistry course, but also in analytical chemistry (when talking about characteristic reactions used in qualitative analysis of cations/anions [1,2]) one finds the following reaction:

\[
\text{Pb}^{2+}(\text{aq}) + 2\text{I}^-(\text{aq}) \rightarrow \text{PbI}_2(\text{s})
\]

and it is often mentioned as one of the reactions suitable for identification of the presence of lead in a given solution, for the precipitate is readily recognized by its bright yellow color. Also, there are several nice demonstrations that are based on the above reaction [3–5] (similar demonstrations could also be found on the World Wide Web too [6–8]). The most effective are those including cooling of hot and saturated water solution of lead(II) iodide [4]. The latter salt has a property of being sparingly soluble in cold water, but fairly soluble in hot water. On cooling of its saturated water solution, \( \text{PbI}_2 \) crystallizes in the form of small golden-yellow needles that are readily precipitated (due to the relatively high density of the salt, as well as due to the large size of the \( \text{PbI}_2 \) particles obtained in this way).

The important thing is that, the way eq. 1 is written (i.e. in ionic form), it is a source of a misconception. One may conclude that no matter what lead(II) salt is used, the addition of aqueous solution of potassium iodide (or, for that matter of any other soluble ionic iodide) will result in precipitation of yellow \( \text{PbI}_2 \). This is not true, as will be demonstrated further. In what follows, we will describe a set of research procedures that enabled us to identify the nature of unknown precipitate.

The Problem

Knowing for many years the above chemical reaction, and working always with aqueous solutions of lead(II) nitrate, it happened once that we simply used stock solution of lead(II) acetate, for we thought we know it doesn’t matter. A pale yellow precipitate was formed immediately. Although the color is similar to the bright yellow color of \( \text{PbI}_2 \) (cf. Fig. 1) the pale yellow precipitate was practically insoluble both in cold as well as in hot water. It became obvious that the two precipitates are chemically different. One had to answer the question: what is the chemical nature of the pale yellow substance, and why are the precipitates obtained from nitrate and acetate different?

Figure 1

The precipitate obtained upon the action of \( \text{KI(aq)} \) on aqueous solution of lead(II) acetate (left) and lead(II) nitrate (right).

At first it seemed logical to assume that a double salt was formed, possibly \( \text{Pb(CH}_3\text{COO})\text{I} \). However, this assumption had to be confirmed in some way. IR spectroscopy seemed to be both fast and convenient method for the above task.

IR Spectroscopic Measurements

The IR spectra of the pale yellow precipitate and of \( \text{KCH}_3\text{COO} \) were recorded on the Perkin Elmer System 2000 FT–IR interferometer. The substances (1–2 mg of each) were homogenized with 300 mg KBr and pressed on a hydraulic press, at a pressure of 1 GPa. The potassium acetate was recorded for purposes of comparison (if the two salts contain acetate anions, the spectra are expected to show some resemblance).

Experimental conditions. Both background and sample spectra were acquired with 8 scans, on an unpurged instrument. For more efficient elimination of problems
due to non-compensations of H₂O and CO₂ bands, a Perkin-Elmer Spectrum Software package [9] was used. A standard resolution of 4 cm⁻¹ was used. Other data regarding the choice of instrument parameters were: apodization strong; OPD velocity 0.2 cm/s; interferogram bidirectional, double sided.

For low-temperature FT–IR spectroscopy, a Graseby-Specac low-temperature cell was used. Spectra were again analyzed from KBr pellets, while liquid nitrogen was used as a coolant.

Perkin-Elmer Spectrum Software package [9] and Grams/386 software package [10] were used for spectra acquisition and manipulation respectively.

IR Spectroscopic Results:
Motivation for Further Checks
The spectra are presented in Figure 2.

![Figure 2 a, b] FT IR spectra of potassium acetate (a) and the pale yellow precipitate (b). Note the distortion of the sharp band at ~3500 cm⁻¹, due to Christiansen effect.

Even an amateur in IR spectroscopy could safely conclude that the spectra are dramatically different. Obviously, the pale yellow substance contains no acetate groups. A more experienced spectroscopist will also note that, apart from appreciable quantities of “hygroscopic” water (water absorbed by the sample), the spectrum of the unknown does not seem to give evidence for a presence of any polyatomic anion. Two bands (at 3500 cm⁻¹ and at 604 cm⁻¹) strongly indicate a presence of OH⁻ anions. The sharp band at 3500 cm⁻¹ could easily be attributed to the O–H stretching vibration (the relatively high frequency and the band shape reveal that the OH⁻ anions are only weakly hydrogen bonded). The latter, rather broad band could be attributed to libration (a hindered rotation) of the OH⁻ anions. For a more detailed discussion of the IR spectra see Appendix 1.

What is the chemical nature of the pale yellow precipitate? The absence of other polyatomic anions but OH⁻ strongly suggests that the composition is Pb(OH)I, or at the very least, Pb(OH)(I)ₓ. If this is correct, then the differences between the nitrate and acetate salts could be explained in terms of the different pH values: water solution of lead(II) acetate is expected to have a higher pH value than the solution of the lead(II) nitrate (this is due to partial hydrolysis of both salts in solution, and the fact that acetic acid is weak, while nitric acid is a strong one). PbI₂ is formed from the nitrate, as already known, but the conditions in the lead(II) acetate solution are more favorable for formation of the hydroxide salt. Let us also mention that the solubility of Pb(OH) in water is much lower compared with that of PbI₂ [5], so at certain pH values the formation of the hydroxide salt instead of PbI₂ is fully justified.

If the above is correct, then one might reason that if the suspension of the pale yellow substance in water is acidified (say with CH₃COOH, to exclude the presence of other ions), then the Pb(OH)Iₓ could be converted to PbI₂, following the equation:

\[
Pb(OH)I_x(aq) + xCH₃COOH(aq) \rightarrow (1-\frac{1}{2}x)PbI₂(s) + \frac{1}{2}xPb(CH₃COO)₂(aq) + xH₂O(l) \quad (2)
\]

We then performed the above test: a water solution (c = 2 mol/L) of acetic acid was added to the pale yellow precipitate. The precipitate instantaneously turned bright yellow, in a complete accord with the expectations. It was then safely to conclude that the pale yellow precipitate is indeed Pb(OH)I, or Pb(OH)(I)ₓ. One more experiment (suggested by the referee), illustrates this notion even more vividly: small portions of diluted aqueous solution of KOH were added to a suspension of PbI₂ in water. The yellow color of the precipitate becomes paler and paler, till it is almost white (cf. Fig. 3). One could say that with each added portion, x increases from 0 tending to reach its maximum value of 1.

![Figure 3] Changes in the color of aqueous suspension of PbI₂, upon addition of KOH(aq).

Conclusions
The experiment is of undisputable educational value. It teaches several things:
• chemically different substances can have similar (and sometimes almost identical) color;
• conclusions drawn by analogy (i.e. it is the same whether one should use a nitrate or an acetate salt) could be wrong;
• for many precipitates it is important to monitor the pH value of the medium, since hydroxide salts can often be obtained instead of normal salts (e.g. bismuth salts transform very often to hydroxide salts, if the medium is not properly acidified);
• once the research procedure has been performed as explained, it could be effectively modified into a serial (i.e. “a sequence of small experiments leading, step by step, to the desired goal” [11]), without using IR spectroscopy at all (this is important, as the use of IR instrument during lectures is highly impractical, if not impossible).

One should note that in a research experiment (procedure), it is not necessary that the result is indeed new and original.
The point of it is that the result is a novelty for the class or for the class and the instructor. A working assumption is formulated: in our experiment we assumed that the pale yellow precipitate is a double salt lead(II) iodide-acetate. Then, a strategy has to be developed (like using IR spectroscopy) to confirm the assumption. In our case the assumption failed, as no presence of acetate ions could be detected. Further, in order to reveal the unknown (the chemical nature of the pale yellow precipitate), another logical assumption might be necessary (the spectra suggested it is a hydroxide salt and an independent proof of this was offered by a simple chemical reaction of the pale yellow precipitate with acetic acid, performed in a test tube).

Safety Tips and Disposal
Safety goggles and gloves should be worn at all times during the demonstration.

All water soluble lead salts are very poisonous! The waste must be disposed according to local safety regulations. It might be useful to know that PbS is practically insoluble precipitate, so the waste could first be converted into PbS and then disposed (e.g. flushed under the drain).

References

Appendix 1
To further confirm the assignment, we decided to run some spectra at the temperature of liquid nitrogen (LNT). Experience shows (there are sound theoretical reasons too) that on cooling the vibrational bands due to the bending O–H vibrations get sharper (the same applies to the OH stretchings, but the effect is less pronounced). Frequency shifts are also often evident: for medium and strong hydrogen bonds, on cooling the OH stretchings exhibit red shift and the bending (librational) bands exhibit blue shifts. If the hydrogen bonds are both weak and highly bent, the stretching OH band might show a blue shift, the latter being also characteristic for bifurcated hydrogen bonds [12]. The room temperature (RT) and liquid-nitrogen temperature (LNT) spectra are given in Fig. 4.

Figure 4 The OH stretching (left) and bending (right) regions of the FT IR spectra at room (a) and liquid nitrogen (b) temperature of the pale yellow precipitate.

It is evident that both intense bands are much sharper at LNT. There is a minor blue shift on cooling (5 cm⁻¹) of the OH stretching band. These findings confirm that the only type of polyatomic anions present are OH− anions (these participate not only in formation of weak, but also of rather bent hydrogen bonds, as evidenced by the blue temperature shift of the OH stretching band).
Nanoscale Science and Technology in Teaching

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Abstract

A study of prospective teachers’ general knowledge of nanoscale science and technology is reported. A voluntary sample of 109 prospective teachers in an undergraduate science education course participated in the study by responding to a ten-item questionnaire. The items demanded various types of knowledge of nanoscale science and technology such as etymology, chemical ingredients of materials such as sunscreens, and an understanding of physical scale. The average score for all ten items was 6.13 (SD = 1.34). The study revealed a lack of understanding of the underlying physical scale of nanoscience and nanotechnology, and the etymology of the term “nano” among prospective teachers.

Nanoscience and Nanotechnology

A study of the nanoscale science and technology knowledge of students (prospective teachers) in several sections of an undergraduate science education course is reported in this paper. Nano refers to dwarf, sizes of the order of one-billionth of a meter. Nanoscience refers to the scientific study of materials one billionth of a meter in size, and nanotechnology refers to various technologies to produce materials of extra high precision and dimensions on the scale of one-billionth of a meter (The Royal Society, 1994; National Science and Technology Council (NSTC) Committee on Technology, 1999). Nanotechnology is of great interest to researchers, business and governments around the world, and its impact is felt in almost all areas of life. For example, in 1971 it cost 10 cents to manufacture a computer chip containing 2,300 transistors with clock speed 0.8 million cycles/second (Semiconductor Industry Association, 2003). By 2003 the cost of manufacturing a computer chip (e.g., Pentium) containing 108 million transistors with clock speed 3,000 million cycles/second was 1/1000 cents thanks to developments in nanotechnology. The number of articles (registered by Institute for Scientific Information) with “nano” as a topic increased from approximately 60 in year 1995 to over 3000 by year 2004 (Kumar, 2006). Nanoscience and nanotechnology are areas of great interests in developed as well as developing nations.

Relevance to Science Teaching

The field of nanoscale science and technology provides teachers the opportunity to develop motivating and engaging interdisciplinary learning experiences for students. “It brings a new excitement to many subjects. Students see amazing applications that result from nanoscience, and learn that an understanding of basic science is necessary to make them happen. For example, CD’s wouldn’t exist without nanotechnology; neither would the new colo[u]r changing paints on luxury cars and motorbikes; transparent sunscreens and many new cosmetics use nanoparticles; the technology behind stay-clean chinos (and even now school uniforms) is nanotechnology. These developments depend on a fundamental knowledge of chemistry, physics and biology.” (How is the Nanotechnology Relevant to the Scottish School Curriculum? n.d., p. 1).

Yet, as the National Commission on Teaching and America’s Future (1996) stated, the teachers’ knowledge and their teaching are the most significant factors that affect student learning. Therefore, to accomplish systemic reform in science education, it is imperative that classroom teachers are equipped with contemporary knowledge to teach science meaningfully to students (National Research Council, 1996). In this context the study attempted to answer the following question: What general knowledge do undergraduate science education (K-9) students have about nanoscale science and technology?

Method

The study implemented the Nano Quiz (Appendix 1) containing ten multiple-choice items used with permission from the National Institute of Standards and Technology (U.S. Department of Commerce, n.d.). The purpose here was to gauge the general knowledge level of study participants about nanoscale science and technology. No discussion on nanoscience and nanotechnology took place prior to administering the quiz.

Nano Quiz item 1 deals with the etymology of the prefix “nano” which means dwarf and has both Greek and Latin roots (NSTCCT, 1999). Item 2 requires an abstract understanding of perception of distance and an ability to make logical comparisons. One billion millimeters equals 1,000 kilometers or about 620 miles. Item 3 requires an understanding of sizes of atoms and especially an abstract understanding of the perception of width. For example, a hydrogen atom has a diameter of 1/10 nanometer (NSTCCT, 1999). A general knowledge of the chemical composition of day-to-day materials such as sunscreens is required for answering item 4. Other related information includes stain resistant nanowhiskers coated on khaki pants, nanoparticles used to seal certain tennis balls, and nano films used on read heads in computer hard drives.

It should be noted that for items 5, 6, 7, 9, and 10 the respective terms “nanonewton,” “qubit,” “flying qubit,” “self-assembled monolayer,” and “spintronics” are not often addressed in the science content courses taken by prospective teachers. According to the National Science and Technology Council Committee on Technology (NSTCCT, 1999) a nanonewton is one billionth of the force needed to hold 20 nickels against the force of gravity. Qubits in quantum information processing represent bits at nanoscale capable of holding large amounts of information spanning from 0 and 1. Photons used to
process quantum information represent flying qubits. Self-assembled monolayers are uniform single layers formed as a result of atoms or molecules spontaneously attaching to specific surfaces. Spintronics represent the use of pattern (up or down) electronic spins to process information. Item 8 demands knowledge of a collection of atoms doing the same thing the same time and the Nobel Prize winning history behind the Bose-Einstein Condensate.

Sample
The participants were student volunteers (prospective teachers, N = 109) enrolled in several sections of an undergraduate science education course in an ethnically diverse urban university in the southeastern United States.

Results
The average score for all ten items was 6.13 (SD = 1.34). Scores for items 5, 6, 7, 8, 9 and 10 were above 60%. For item 4 the respective score was below 60% but above 40%. The scores for items 1 and 2 were between 25% and 40%. The score for item 3 was far below 25%. See Table 1 for a summary of Item Response Distribution, Item Difficulty, and Point Biserial Item Discrimination results.

Discussion and Recommendation
The findings must be interpreted with caution. Some of the items require an abstract knowledge of the underlying physical scale of nanoscience and nanotechnology. Though most of the items on the quiz required knowledge of the etymology of the terminology used in nanoscience and technology, item 1 registered an item difficulty of 0.275. See Sarma (2006) for details on etymology. Items 2 and 3 dealt with physical scale and had item difficulty values 0.257 and 0.073 respectively. Especially item 3 required knowledge of the diameter of hydrogen atom. The distracter in item 5 “a miniature pop singer,” “a tiny lizard,” and item 7 “a prehistoric bird,” seem rather easy to eliminate.

It is recommended that science teacher education programs provide prospective teachers in depth opportunities to discuss and develop cognitively engaging and motivating ways of teaching nanoscale science and technology. Particular attention must be paid to the etymology of science and technology terms and the physical scale upon which the field of nanoscience and nanotechnology is developed.

Acknowledgements
The author would like to acknowledge Dr. John Enger at Florida Atlantic University and the two anonymous reviewers of AJEC for thoughtful critique and feedback, and Ms. Mindy Kessler at FAU for editorial assistance. The items in the Nano Quiz were reprinted courtesy of the National Institute of Standards and Technology, Technology Administration, U.S. Department of Commerce.

References


Table 1. Summary of Item Response Distribution, Item Difficulty, and Point Biserial Item Discrimination

<table>
<thead>
<tr>
<th>Item</th>
<th>Topic</th>
<th>Item Response Distribution</th>
<th>Item Difficulty</th>
<th>Item Discrimination (Point Biserial) (p &lt; 0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Etymology of “Nano”</td>
<td>N 18</td>
<td>30 (16.5)</td>
<td>0.275</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(27.5) (37.6)</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Physical Scale</td>
<td>N 42</td>
<td>20 (38.5)</td>
<td>0.257</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(18.3) (15.6)</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Physical Scale</td>
<td>N 27</td>
<td>8 (24.8)</td>
<td>0.073</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(7.3) (30.3)</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Chemical Ingredients</td>
<td>N 2 2</td>
<td>1 (1.8)</td>
<td>0.431</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(2.8) (0.9)</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Unit of Force</td>
<td>N 2 2</td>
<td>104 (1.8)</td>
<td>0.954</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(18) (95.4)</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Quantum Mechanics</td>
<td>N 8 1</td>
<td>70 (7.3)</td>
<td>0.642</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(0.9) (64.2)</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Quantum Information Processing</td>
<td>N 1 2</td>
<td>104 (0.9)</td>
<td>0.954</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(1.8) (95.4)</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Atomic Physics and History of Science</td>
<td>N 2 4</td>
<td>14 (1.8)</td>
<td>0.835</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(45.0) (12.8)</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Surface Chemistry</td>
<td>N 94</td>
<td>4 (1.8)</td>
<td>0.862</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(64.2) (2.8)</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Pattern of Electron Spin</td>
<td>N 92</td>
<td>13 (86.2)</td>
<td>0.844</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(%)</td>
<td>(84.4) (11.9)</td>
<td></td>
</tr>
</tbody>
</table>
Appendix 1: Nano Quiz Items and Multiple Choice Responses

1. The prefix “nano” comes from a Greek word meaning ______________________
   a) billion;  
   b) dwarf  
   c) invisible  
   d) infinite

2. If a nanometer were about as big as the width of a pinhead, about how long would a meter be?
   a) as long as the pin shaft  
   b) as long as a ladder  
   c) as long as a blue whale  
   d) as long as a trip between Washington, D.C., and Atlanta, Georgia

3. How many hydrogen atoms lined up “shoulder to shoulder” would fit in a one nanometer space?
   a) less than one  
   b) ten  
   c) 1 thousand  
   d) 1 billion

4. Which of the following products contain nanoscale manufactured parts or materials?
   a) sunscreen  
   b) khaki pants  
   c) tennis balls  
   d) devices that read computer hard drives  
   e) all of the above

5. What is a nanonewton?
   a) a new kind of cookie  
   b) a miniature pop singer  
   c) approx. amount of force required to break a single chemical bond between two atoms  
   d) a tiny lizard

6. What is a qubit?
   a) a unit of measure used in ancient Egypt  
   b) a cover for the tip of the stick used in billiards  
   c) a unit of information that takes advantage of the laws of quantum mechanics  
   d) a pair of atoms used to store digital information

7. What is a flying qubit?
   a) a prehistoric bird  
   b) a muon  
   c) a photon used to transport quantum information  
   d) a gluon

8. What is a Bose-Einstein Condensate?
   a) a new type of “smart” speaker that works under water  
   b) a collection of atoms doing exactly the same things at exactly the same time  
   c) a lumpy gray substance invented by Bose and Einstein  
   d) a physics “Holy Grail,” achieved in 1995, leading to a Nobel Prize in 2001  
   e) b and/or d

9. What is a “self-assembled monolayer”?
   a) atoms or molecules that spontaneously form uniform single layers  
   b) a type of clothing that gets thicker in response to colder temperatures  
   c) an optical device that puts itself together  
   d) a fuzzy logic circuit

10. What is spintronics?
    a) using the spins of electrons to carry information  
    b) electronics made of textiles  
    c) a rock group  
    d) a type of dance music

(Correct Responses: 1 b, 2 d, 3 b, 4 e, 5 c, 6 c, 7 c, 8 e, 9 a, 10 a.)

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CONFERENCE NOTICE

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Phosphorus and Selenium NMR Spectroscopy: An APCELL experiment*

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Abstract

Most undergraduate chemistry textbooks on spectroscopy focus on $^1$H and $^{13}$C nuclear magnetic resonance (NMR). In this laboratory exercise, $^{31}$P and $^{77}$Se spectra are used to monitor the extent and regioselectivity of the oxidation reactions of tris(2-diphenylphosphinoethyl)phosphine on the addition of 1, 2, 3 and 4 equivalents of sulfur and selenium.

Introduction

Nuclear magnetic resonance (NMR) spectroscopy is an important analytical technique in many fields of scientific endeavour. Heteronuclear NMR is used in inorganic chemistry, biochemistry and other fields. For example, $^{31}$P NMR is often used to probe biochemical processes. Despite this, most chemistry textbooks on spectroscopy focus on $^1$H and $^{13}$C NMR.

<table>
<thead>
<tr>
<th>Nucleus</th>
<th>% Abundance</th>
<th>Nuclear spin $I$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^1$H</td>
<td>99.99</td>
<td>$\frac{1}{2}$</td>
</tr>
<tr>
<td>$^{13}$C</td>
<td>1.11</td>
<td>$\frac{1}{2}$</td>
</tr>
<tr>
<td>$^{19}$F</td>
<td>100</td>
<td>$\frac{1}{2}$</td>
</tr>
<tr>
<td>$^7$Li</td>
<td>92.5</td>
<td>$\frac{3}{2}$</td>
</tr>
<tr>
<td>$^{14}$N</td>
<td>99.6</td>
<td>1</td>
</tr>
<tr>
<td>$^{31}$P</td>
<td>100</td>
<td>$\frac{1}{2}$</td>
</tr>
<tr>
<td>$^{33}$S</td>
<td>0.74</td>
<td>$\frac{3}{2}$</td>
</tr>
</tbody>
</table>

Table 1: Nuclear spin properties of various nuclei

In this experiment NMR spectroscopy is used to follow the reaction of tris(2-diphenylphosphinoethyl)phosphine (also called “Tetraphos-2” or “PP3”) a commonly used ligand, with elemental sulfur and selenium.

Like most phosphines, PP3 is oxidised by reaction with chalcogens to form pentavalent phosphorus compounds. Students monitor the extent and regioselectivity of the reaction of PP3 with S and Se through the use of $^{31}$P (100% abundance) NMR spectroscopy after the addition of 1, 2, 3 and 4 equivalents of the oxidant.

* The complete documentation for this experiment is freely available on the APCELL web site [www.apcell.org]. It includes the educational template, a set of student notes, demonstrator notes and technical notes to allow ready implementation into a new laboratory.
1.4 Time Required to Complete
Prior to Lab 1.5 hours reading and pre-lab exercise
In Laboratory 2 hours laboratory and 2 hours for analysis of results
After Laboratory 2-3 hours report writing

1.5 Acknowledgments
This experiment was originally developed at Deakin University several years ago by one of the present authors in collaboration with other colleagues. We thank our colleague, Professor Dainis Dukternieks, for his assistance and support. KFL thanks Ms Jeanne Lee (朱靜) for encouraging and helpful discussions.

1.6 Any Other Comments:
The teaching-and-learning assessment described in this paper has been approved (EC 29-2002) by the Deakin University Human Research Ethics Committee.

<table>
<thead>
<tr>
<th>Learning Outcomes</th>
<th>Process</th>
<th>Assessment</th>
</tr>
</thead>
<tbody>
<tr>
<td>What will students learn?</td>
<td>How will students learn it?</td>
<td>How will students know they have learnt it?</td>
</tr>
<tr>
<td>Theoretical and Conceptual Knowledge</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Students should appreciate the operation of a modern FT-NMR spectrometer.</td>
<td>Students will use a modern FT-NMR spectrometer.</td>
<td>Students will interpret $^{31}$P and $^{77}$Se spectra to infer the bonding environment of P and Se.</td>
</tr>
<tr>
<td>Students should understand that NMR spectroscopy can be applied to nuclei other than $^1$H and $^{13}$C.</td>
<td>Students will measure $^{31}$P and $^{77}$Se spectra.</td>
<td>Students will apply the NMR information to inorganic chemistry by inferring similarities and differences in the reactivities of the two group 16 elements sulfur and selenium.</td>
</tr>
<tr>
<td>Students should appreciate the different aspects of FT-NMR spectroscopy, including heteronuclear coupling and nuclear relaxation.</td>
<td>Students must decide what to include or omit from a formal written report. They are given the demonstrator’s assessment and feedback pro forma. They are encouraged to seek help from the demonstrator.</td>
<td>There must be sufficient data, details and discussion in the main body of the report, so that a student (classmate) who has done everything as the student writer, except this exercise (or this unit), can understand the report.</td>
</tr>
<tr>
<td>Students should be able to relate spectroscopic information and concepts to bonding and reactivity concepts in other parts of the chemistry syllabus.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Students should be able to exercise judgement about what is (or is not) relevant in the context of the exercise, judgement about what is (or is not) significant in the context of the exercise, and judgement about what is (or is not) important in the context of the exercise.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scientific and Practical Skills</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Students should appreciate the operation of a modern FT NMR spectrometer.</td>
<td>Students assist the NMR operator in collecting spectra. Students can collect their own data if the laboratory has a teaching NMR spectrometer.</td>
<td></td>
</tr>
<tr>
<td>Generic Skills</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Students should be able to work in teams, and to plan and manage their time effectively.</td>
<td>Students must divide tasks between themselves at different stages of the laboratory exercise.</td>
<td>Students will complete the allocated tasks with minimal conflict.</td>
</tr>
<tr>
<td>Students should (further) develop communication and generic skills (2.3), including the ability to use appropriate computer programs (4). Note: The semester-long spectroscopy laboratory program at Deakin University is one of a series of</td>
<td>Students are given the opportunity to submit draft reports for comment. Students are encouraged to consult their demonstrator on the report writing style and use of appropriate computer programs</td>
<td>Students will present a formal written report, which satisfies the criteria set out on an assessment and feedback pro forma.</td>
</tr>
</tbody>
</table>
### Section 3 - Student Learning Experience

<table>
<thead>
<tr>
<th><strong>Laboratory programs specifically intended to foster report-writing skills and use of computer packages (eg word processors and spreadsheets). Students are given the opportunity to submit draft reports for comment. This aspect of the curriculum is not an integral component of the current exercise.</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>All of the above knowledge and skills</strong></td>
</tr>
<tr>
<td>By preparing a clear, well-structured formal report, students will organise their knowledge and understanding and to consolidate learning (5).</td>
</tr>
<tr>
<td>Students demonstrate that their knowledge, skills and understanding … satisfy the stated and implied criteria and they have met [or exceeded] all the other requirements … Note: This criterion is an extract from the Faculty guidelines on grading and assessment. It is clearly communicated to students during the semester and is the basis for assessment of all laboratory exercises and assignments.</td>
</tr>
</tbody>
</table>

3.1 **Did this experiment help you to understand the theory and concepts of the topic? If so, how, or if not, why not?**

| S1: | Yes after completing the prac I felt I had a much better grasp of NMR. The textbooks in the library were of great help. the experiment allowed for very comprehensible interpretation of spectra. |
| S2: | Yes it did. The explanation given by [the demonstrator] during the prac assisted in my understanding. |
| S3: | Yes, practical experience for theoretical concepts |
| S4: | Yes. It reinforced NMR work and understanding prior to examination |
| S5: | Yes. It was very informative. valuable to see how the NMR works |
| S6: | Yes. It helped explain multinuclear NMR |
| S7: | Yes, help with multinuclear component |
| S8: | Yes |
| S9: | Yes, This topic was very well explained during the prac leading to better understanding |

3.2 **How is this experiment relevant to you in terms of your interests and goals?**

| S1: | Experiment v relevant for future work in identifying compounds. I am v interested in NMR I find it more interesting than other I.D. techniques |
| S2: | To pass this subject and learn techniques to use in the workplace |
| S3: | Practical experience |
| S4: | Relevant because want to do well in this subject |
| S6: | It is only relevant to people who want to work with NMR |
| S7: | Put NMR into a better perspective |
| S8: | Yes, need to pass well |
| S9: | It is relevant in that it lead to further understanding which will be relevant in the goal of passing this subject |

3.3 **Did you find this experiment interesting? If so, what aspects of this experiment did you find of interesting? If not, why not?**

| S1: | Yes I found it very interesting. Good to work with other NMR-active nuclei other than $^1$C and $^1$H |
| S2: | How the NMR actually works |
| S3: | Yes. The practical component learning about the instrument |
| S4: | Yes. Good to look 1st hand at how the theory is applied. Quick and simple – not complicated write-up |
| S5: | Yes it was interesting to see how the NMR works and to apply it to all the theory |
| S6: | It was interesting … |
| S7: | Experiment was interesting because it put the NMR theory into practice and could see how NMR is used |
| S8: | Sort of, don’t mind the concept |
| S9: | Yes interesting as this is the first time we have been allowed near the NMR machine |

3.4 **Can the experiment be completed comfortably in the allocated time? Is there time to reflect on the tasks while performing them?**

| S1: | Yes … One week was adequate time. This prac tied in with [the lecturer’s] NMR assignment |
| S2: | Yes, … reasonable in terms of the prac write up and expectations |
| S3: | Yes |
| S4: | Yes. Not rushed … |
| S5: | Yes, this experiment was easily completed in the time allowed … |

Note: These comments were obtained from the 2003 cohort of 3rd-year students. The teaching staff have commented that this particular cohort are very focussed on getting good marks and grades, and less interested in being receiving a chemical “education”.

Note: This criterion is an extract from the Faculty guidelines on grading and assessment. It is clearly communicated to students during the semester and is the basis for assessment of all laboratory exercises and assignments.
S6: Yes, this was the most well explained prac because it had a handy reference
S7: Yes no probs with that
S8: Yes
S9: Yes experiment on be completed in time …

3.5 Does this experiment require teamwork and if so, in what way? Was this aspect of the experiment beneficial?
S1: Yes teamwork between classmate discussing spectra and sharing ideas. can talk together about difficult concepts. NMR is a very challenging topic
S2: Yes in being to interpret the spectra
S3: Yes each part of the experiment being done by each group
S4: Yes. Discussion is always beneficial
S5: Yes. the experiment was discussed in groups
S6: Not really
S7: Teamwork was required to perform the experiment which was useful because prac didn’t take as long
S8: Yes, help others
S9: Teamwork is always beneficial

3.6 Did you have the opportunity to take responsibility for your own learning, and to be active as learners?
S1: Yes, I took notes during the prac which helped me greatly. background reading to understand concepts
S2: Yes through the prac write-up
S4: Yes
S5: Yes
S6: Yes. I had to seek out a reference
S7: Yes
S8: Yes
S9: Yes

3.7 Does this experiment provide for the possibility of a range of student abilities and interests? If so, how?
S1: Some students are better at interpreting types of spectra than others. The students feed off each other’s knowledge
S2: Yes I think it does because the expectations are reasonable … in relation to varying student capabilities
S4: No. Only if interested in this. But did demonstrate range of abilities ie making chemicals, analysing etc
S6: Yes the demonstrator explained it in simpler terms
S7: Yes
S8: Yes
S9: Not really …

3.8 Did the laboratory notes, demonstrators’ guidance and any other resources help you in learning from this experiment? If so, how?
S1: The demonstrator’s assistance. [The demonstrator] was wonderful. She was v good at explaining background to NMR and helping with concepts. I was very impressed
S2: No but the explanation given by [the demonstrator] was extremely useful and thorough
S4: Not a great deal from work book but demonstrator’s explanations were excellent and easy to understand
S5: The demonstrator’s explanations were extremely useful and informative
S6: Demonstrator was helpful
S7: Yes lecture notes and also information given in prac itself were useful. (esp. prac info.)
S8: Yes
S9: The demonstrator explained the concepts extremely well

3.9 Are there any other features of this experiment that made it a particularly good or bad learning experience for you?
S1: Good learning experience because it was in tandem with [the lecturer’s] NMR assignments and demonstrator/lab staff/lecturer were v helpful
S2: No not really …
S6: Good – because info was available to help
S7: No bad experience. It was good tho [sic], as stated earlier to put NMR into perspective
S8: Not really

3.10 What improvements could be made to this experiment?
S1: I think this experiment was run well …
S7: No. It was a good lab and also an informative write-up
S9: We could do more NMR

3.11 Any Other Comments
No responses were received for this question.

References
A Practical method for the determination of the classification of substitution groups on benzene ring by addition of electronegativity difference

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Abstract

A semi-quantitative experiential ‘addition of electronegativity difference’ method is proposed and applied to the determination of the category of substitution groups on the benzene ring. The “addition of electronegativity difference” stands for the sum of electronegativity difference of the atom bonding to a benzene ring and other atoms or groups bonding to this atom. For the most common substituents, very good linear correlations between the electronegativity difference of the substituents and their directing capability are observed. Based on this method, the directing capabilities of different substitution groups are quantitatively classified.

I. Instruction

The substitution groups on benzene ring include ortho, para-director (the first category of the substituent) and meta-director director (the second category of substituent). The former directs the subsequent groups to ortho, para-position, the later to meta-position. Some experiential rules used for determining the type of substituent were reviewed in ref. [1a]. Among these methods, Vorlander’s rule [2] is the most concise: if the atom attached to the benzene ring is saturated, the substituent is of the first category; while it is unsaturated, the substituent should be of the second category. However, it is found that this rule is improper for many situations. For example, in -CCl₃, -CF₃, -CH=CHCOH, and -H=CHNO₂ etc. -CCl₃, -CF₃ are saturated groups, but they are of the second category of substituent; -CH=CHCOH, -H=CHNO₂ are of the first category although they are unsaturated groups. Hammick and Liingworth [3] proposed a modified rule, of which the main points are: (i) suppose the structure of a single substituted aromatic is C₆H₅-XY, if Y is in a higher group than X in the element periodic table, or the atom Y is smaller than X atom while they are in the same group, the substituent -XY is meta-director; (ii) in other cases, if both X and Y are single atoms, -XY is ortho, para-director; (iii) the positive charged substituent is meta-director, and the negative charged substituent is ortho, para-director. However, this rule could not tell the classification of the substituent groups, -CH₃, -CH₂Cl, -CH₂Br and -CH₂OH. Shi[4] suggested a new method to clarify the category of the director based on the oxidation numbers of the atoms or groups attached to the benzene ring. If the oxidation number of the atom attached to the aromatic ring is less than zero, the substituent is of the first category of director; otherwise it should be of the second category of director. This method still would not help in understanding the deactivating capability of -CHCl₂, -CHBr₂ to the aromatic ring.

Hereby, based on the inductive effects theory, we developed a new half-qualitative method to classify the category of aromatic substitution by means of calculating the addition of electronegativity difference.

II. Methodology

It is well known that the electronegativity is one of the important characteristics of element. It can be used to describe the polarity of covalent bond in a molecule and reveals the distribution of chemical charges in the covalent molecule. When a covalent bond is formed between two identical atoms, the electrons are equally shared and symmetrically distributed between two atoms since each atom holds the electrons of the bonding pair to the same extent. Their electronegativity difference is zero. In the other case of bond formation between two different atoms, each atom holds the electrons of the bonding pair differently. If the electronegativity of A atom, Xₐ, is smaller than that of W atom, X₇, the electron density lies closer to W atom in the covalent bond.

In substituted benzene structure, either σ-π or π-π conjugated system may influence the distribution of electron density of [W] in the benzene ring. Suppose the structure of the substituted aromatic ring is:

![Substituted Benzene Ring Diagram]

W is designated as the atom which attaches to benzene directly; A, B, C could be an atom or group. While the W atom forms a covalent bond with A, B, and C, the total electronegativity of the group is the addition of the electronegativity differences between atom W and A, B and C, respectively. If the addition of their difference is zero or negative, it means the electron density around W atom is higher than other atoms; W atom in the molecule is able to donate electrons to the aromatic ring; and this substituent is assigned to the first category of director. Otherwise, when the addition of their electronegativity difference is positive, the substituent is assigned to the second category of director. It is described mathematically as:

\[ \Delta X_w = \Sigma (X_i - X_w), \quad (i=A,B,C) \]

\[ \Delta X_w^* : \text{the addition value of the electronegativity difference between W atom and A, B, and C atoms (groups) respectively.} \]

The electronegativity value is from Pauling’s
standard [5, 6]. It is calculated based on the following assumptions:

Assume \( \Delta X_w \) is zero if the substituted halogen atom in benzene ring bonds to no other atom.

Table 1: The addition values of electronegativity difference of some substituents

<table>
<thead>
<tr>
<th>Substituent</th>
<th>Difference of electronegativity, ( \Delta X_w )</th>
<th>Director category</th>
</tr>
</thead>
<tbody>
<tr>
<td>-OH</td>
<td>-1.24</td>
<td>1</td>
</tr>
<tr>
<td>-OCH₃</td>
<td>-0.89</td>
<td>1</td>
</tr>
<tr>
<td>-OCOCH₃</td>
<td>-0.70</td>
<td>1</td>
</tr>
<tr>
<td>-CH₂CO₂CH₃</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>-O⁻</td>
<td>-3.44</td>
<td>1</td>
</tr>
<tr>
<td>-CH₃Br</td>
<td>-0.59</td>
<td>1</td>
</tr>
<tr>
<td>-CH₂Cl</td>
<td>-0.09</td>
<td>1</td>
</tr>
<tr>
<td>-C≡CH</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>-CH₃</td>
<td>-1.05</td>
<td>1</td>
</tr>
<tr>
<td>-CH₂CH₃</td>
<td>-0.70</td>
<td>1</td>
</tr>
<tr>
<td>-CH₂CO₂CH₃</td>
<td>-0.70</td>
<td>1</td>
</tr>
<tr>
<td>-CH₂CH₂NH₂</td>
<td>-0.70</td>
<td>1</td>
</tr>
<tr>
<td>-NH₂</td>
<td>-1.68</td>
<td>1</td>
</tr>
<tr>
<td>-NHCH₃</td>
<td>-1.63</td>
<td>1</td>
</tr>
<tr>
<td>-N(CH₃)₂</td>
<td>-1.58</td>
<td>1</td>
</tr>
<tr>
<td>-NHCO₂H</td>
<td>-1.63</td>
<td>1</td>
</tr>
<tr>
<td>-NHC₃</td>
<td>-1.38</td>
<td>1</td>
</tr>
<tr>
<td>-SH</td>
<td>-0.38</td>
<td>1</td>
</tr>
<tr>
<td>-SCH₃</td>
<td>-0.08</td>
<td>1</td>
</tr>
<tr>
<td>-CH₂F</td>
<td>0.73</td>
<td>2</td>
</tr>
<tr>
<td>-CHCl₂</td>
<td>0.87</td>
<td>2</td>
</tr>
<tr>
<td>-CCI₃</td>
<td>1.83</td>
<td>2</td>
</tr>
<tr>
<td>-CF₂</td>
<td>4.29</td>
<td>2</td>
</tr>
<tr>
<td>-CHO</td>
<td>1.43</td>
<td>2</td>
</tr>
<tr>
<td>-COCl₂</td>
<td>1.78</td>
<td>2</td>
</tr>
<tr>
<td>-CONH₂</td>
<td>2.17</td>
<td>2</td>
</tr>
<tr>
<td>-COOH</td>
<td>2.67</td>
<td>2</td>
</tr>
<tr>
<td>-CO₂H</td>
<td>2.45</td>
<td>2</td>
</tr>
<tr>
<td>-CN</td>
<td>1.78</td>
<td>2</td>
</tr>
<tr>
<td>-CNH=NO₂</td>
<td>1.47</td>
<td>2</td>
</tr>
<tr>
<td>-SO₂H</td>
<td>4.30</td>
<td>2</td>
</tr>
<tr>
<td>-SO₂CH₃</td>
<td>3.36</td>
<td>2</td>
</tr>
<tr>
<td>-Si(OH)₄</td>
<td>2.80</td>
<td>2</td>
</tr>
<tr>
<td>-CO₂H</td>
<td>0.19</td>
<td>2</td>
</tr>
<tr>
<td>-NO₂</td>
<td>1.60</td>
<td>2</td>
</tr>
<tr>
<td>-NO</td>
<td>0.80</td>
<td>2</td>
</tr>
<tr>
<td>-N(CH₃)₂</td>
<td>3.04</td>
<td>2</td>
</tr>
<tr>
<td>-P(CH₃)₃</td>
<td>2.19</td>
<td>2</td>
</tr>
</tbody>
</table>

If there is a multiple bond between W and A (B, C), it is calculated as \( n \) times single electronegativity difference of W and A (B, C). For example: \(-NO₂\), \( \Delta X_w = 4 \) \( (X_C-X_N) = 4 (3.44-3.04) = 1.60 \).

If A, B, C in the substituent W-A (B, C) are the atomic groups, we take the electronegativity value of the atom which directly connects to W atom, such as -C(CH₃)₃, the electronegativity difference is \( \Delta X_C = 3 (X_C-X_N) = 0 \).

If W atom is negatively charged or positively charged, \( \Delta X_w \) is taken as the negative or positive value of its electronegativity value, respectively. For example, the \( \Delta X_w \) of -O⁻ is 3.44 and the \( \Delta X_w \) of -N⁺ (CH₃)₃ are 3.04. Table 1 lists the \( \Delta X_w \) of some common substituents.

III. Practicability

When a halogen atom forms covalent bond with an aromatic ring alone, we may say its electronegativity remains unchanged, \( \Delta X_w = 0 \). It is similar to the atoms of O, S, and N in groups -OH, -NH₂, -SH. The halogen atoms have un-bonded electron pair, which may be described as the conjugated structure:

\[
\begin{array}{cccc}
\text{X} & \text{X} & \ldots & \text{X} \\
\text{O} & \text{O} & \ldots & \text{O} \\
\text{S} & \text{S} & \ldots & \text{S} \\
\text{N} & \text{N} & \ldots & \text{N} \\
\end{array}
\]

The conjugation structure indicates that the electron density is higher at the ortho-, para- position of atom X. As a result, it makes the ortho-, para-position of the X atom on the benzene ring be the target of electrophilic agent and the halogen atoms exhibit the property of the first category of director. However, another question might be asked: Why halogen atoms deactivate the aromatic ring, but -OH, -NH₂ activate the aromatic ring? It is known that the radius of halogen atom (except for F) is larger than that of oxygen or nitrogen atoms; its p orbit has bigger spreading radius than that of oxygen and nitrogen atoms; the resonance effect of halogen with the aromatic ring is less than oxygen or nitrogen, while the inductive effect of halogen is stronger than that of its resonance effect and causes net electron withdrawal and hence deactivation-for attack at all positions. As a result, halogen atoms deactivate the aromatic ring. Since O, N and C are in the same period in the element periodic table, the p orbit of O, N atoms can conjugate with the benzene ring effectively, and they could activate the benzene ring.

While the covalent bonds are formed between two different atoms, the bond electron density is surely close to the atom with more electronegativity. In the case of -CN, the bond electrons appear mostly around nitrogen atom and the electron density around carbon atom decreases. The electronegativity difference of -CN group \( \Delta X_w \) is +1.47, so it deactivates the aromatic ring. Its conjugated structure can be described as:

\[
\begin{array}{cccc}
\text{C} & \text{N} & \ldots & \text{C} \\
\text{N} & \text{N} & \ldots & \text{N} \\
\text{C} & \text{C} & \ldots & \text{C} \\
\text{C} & \text{C} & \ldots & \text{C} \\
\end{array}
\]

The conjugated structures apparently show that the electron density at the ortho, para-position of -CN substituent on the aromatic ring decreases a lot, and -CN exhibits the property of the second category of director.
By using $\Delta X_w$ value we can quantitatively estimate the directing capability of some directors with similar structure. As different substituents in electrophilic substitution reaction, their relative directing capability can be previewed via reaction rate constant; vice versa, the relative reaction rate of electrophilic substitution reaction of the substituted benzene with similar substituents can be predicted using the $\Delta X_w$ value. Table 2 lists the $\Delta X_w$ value of some substituents and the relative reaction rate in the bromonation of the substituted benzene. In Table 2, the relative bromonation reaction rate of the substituted benzene with different substitutes is: $-\text{CH}_3 > -\text{C}_2\text{H}_5 > -\text{CH}(_3)\text{CH}_3 > -\text{C}_2\text{H}_6$. It shows that the more negative the $\Delta X_w$ is the stronger ortho (para) directing capability the group has. Consequently, the $\Delta X_w$ of $-\text{CH}_3$, $-\text{CH}_2\text{Cl}$, $-\text{CHCl}_2$, $-\text{CCl}_3$ is $-1.05$, $-0.99$, $+0.87$, $+1.83$, respectively. As the relative bromination reaction rate is plotted as the function of $\Delta X_w$ value, as shown in Figure 1, a good linear relationship between the relative reaction rate and $\Delta X_w$ value is observed.

Table 2: The bromonation reaction rate of substituted benzene with different substituents and their electronegativity difference ($\Delta X_w$)

<table>
<thead>
<tr>
<th>Substituent</th>
<th>Relative reaction rate</th>
<th>$\Delta X_w$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$-\text{CH}_3$</td>
<td>$3.4\times10^2$</td>
<td>$-1.05$</td>
</tr>
<tr>
<td>$-\text{C}_2\text{H}_5$</td>
<td>$2.9\times10^2$</td>
<td>$-0.70$</td>
</tr>
<tr>
<td>$-\text{CH}(_3)\text{CH}_3$</td>
<td>$1.8\times10^2$</td>
<td>$-0.35$</td>
</tr>
<tr>
<td>$-\text{CH}(_3)\text{CH}_2$</td>
<td>$2.1\times10^2$</td>
<td>$0$</td>
</tr>
<tr>
<td>$-\text{H}$</td>
<td>$1.0\times10^3$</td>
<td>$0$</td>
</tr>
<tr>
<td>$-\text{OH}$</td>
<td>$1.1\times10^1$</td>
<td>$-1.24$</td>
</tr>
<tr>
<td>$-\text{OCH}_3$</td>
<td>$1.2\times10^3$</td>
<td>$-0.89$</td>
</tr>
</tbody>
</table>

When the hydrogen in a methyl group is substituted by a more electronegative chloride atom, the first category of director turns into the second category gradually; and it helps to produce more meta-substituted compound, as seen in Table 3. For the carboxylic groups, the order of the meta-directing capability is: $-\text{COOH} > -\text{CONH}_2 > -\text{COCH}_3$, and their $\Delta X_w$ are $+2.67$, $+2.17$, and $+1.78$, respectively, which is in good agreement with the yields of meta-substitution of $80.2\%$, $70.0\%$ and $68\%$.
Advanced Oxidation Process in general chemistry laboratory. ZnO catalysed degradation of acridine orange dye in visible light

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Abstract
In this article, we describe an experiment for undergraduate general chemistry in which students investigate the chemistry involved in photocatalytic degradation of a dye. This useful technique is widely used to treat coloured waste water. Students investigate the details of the degradation process by determining the effect of various parameters on the pseudo first-order rate constant for the degadation of acridine orange. The experiment can be performed in two or 3-hour lab periods, depending on how much time and freedom the students are given to design and carry out their own projects. The material covered in the laboratory includes advanced oxidation process, pseudo first order kinetics, heterogeneous catalysis, spectrophotometry and the application of chemistry toward solving environmental problems.

Background
Academic initiatives are vital for the evolution of advanced oxidation processes. It is required to update undergraduate science programs by incorporating more subject material on wastewater treatment methods. Recently there has been considerable interest in the utilization of advanced oxidation processes for the destruction of organic compounds. Moreover, introduction of photocatalysis is an important issue that needs to be addressed effectively. Among the advanced oxidation processes, heterogeneous photocatalysis appears as an emerging technology leading to the total mineralization of most of the organic pollutants. It is important to incorporate more subject matter on wastewater treatment processes in undergraduate chemistry and environmental chemistry courses (1).

If a fine suspension of suitable semiconductors are irradiated with visible or UV light electron excitation from the valence band to the conduction band results and a vacancy or hole is left in the valence bond. Such holes have the effect of a positive charge. This, in turn, generates the formation of “holes” on the surface of the semiconductor, which can react with oxygen, water, and hydroxide ion to form hydroxyl radicals (2).

Furthermore, superoxide radicals are formed from the reaction of excited electrons with oxygen molecules. The highly reactive active oxygen species so formed then react with the organic pollutants resulting in their oxidation. In many cases, carbon dioxide, water and inorganic ions are the final products.

The kinetics of photocatalytic degradation of many organic compounds in ZnO dispersions under visible irradiation has often been modeled to the simple Langmuir-Hinshelwood equation [3,4] (Eq. 1):

\[ r = \frac{-dC}{dt} = \frac{k'KC}{1 + KC} \]  

(1)

where \( r \) is the rate of disappearance of the reagent, \( C \) is the initial concentration of the dye, \( k' \) is the rate constant and \( K \) is the apparent equilibrium constant. The integrated form of the eq.1 is:

\[ t = \frac{1}{Kk'} \ln(C_o/C) + \frac{1}{k'C_o - C} \]  

(2)

Where \( t \) is the time in min required for the initial concentration of dye \( C_o \) to become \( C \). At low concentration of the dye the second term in equation (2) is negligible compared to the first term. On neglecting the second term the final form of the equation is:

\[ \ln(C_o/C) = k'Kt = - k_{o}^{'}t \]  

(3)

Where \( k'_{o} \) is the apparent rate constant of the photocatalytic degradation. The plot of \( \ln(C_o/C) \) vs \( t \) is a straight line and its slope is - \( k_{o}^{'} \).

Zinc oxide as photocatalyst
ZnO is a potential semiconductor used in photocatalytic reactions. Semiconductors have an energy gap (band gap) between the valence band i.e. highest occupied molecular orbital and the conduction band i.e. lowest unoccupied molecular orbital of moderate order. As a result of this they can behave as a conductor under certain conditions, i.e on exposure to light. Generally semiconductors having large band gaps are good photocatalysts. ZnO and TiO2 with band gaps larger than 3eV show strong activity. Band gap: ZnO = 3.2, TiO2 = 3.2, CdS = 2.3 and \( \alpha \)-Fe2O3 = 2.3 (eV). Diagrammatically representation of band gapes can be shown as:

Diagram:

- Conduction Band
- Valence Band
- Insulator
- Semiconductor
- E > 5 eV
- E ≤ 1 eV
- E = 1.5 - 3 eV
Chemistry of photochemical reaction:

Photocatalytic reaction involves an electron transfer process from semiconductor to substrate or vice versa. The visible light mechanism can be depicted as follows: The dye molecules have the ability to absorb visible light and the electron transfer from the dye to photocatalyst have been found to be very effective. The degradation mechanism in the presence of visible light can be shown as [5,6]:

\[
\begin{align*}
\text{Dye} + h\nu & \rightarrow \text{Dye*} \\
\text{Dye*} + \text{ZnO} & \rightarrow \text{Dye}^{-} + \text{ZnO(e)} \\
\text{ZnO(e)} + \text{O}_2 & \rightarrow \text{ZnO} + \text{O}_2^{-} \\
\text{O}_2^{-} + \text{H}^+ & \rightarrow \text{HO}_2 \\
\text{HO}_2 + \text{HO}_2 & \rightarrow \text{H}_2\text{O}_2 + \text{O}_2 \\
\text{O}_2^{-} + \text{HO}_2 & \rightarrow \text{HO}_2^{-} + \text{O}_2 \\
\text{HO}_2 + \text{H}^+ & \rightarrow \text{H}_2\text{O}_2 \\
\text{H}_2\text{O}_2 + \text{e}^- & \rightarrow \text{OH} + \text{OH}^- \\
\text{Dye}^{-} + \text{O}_2 \text{ (or O}_2^{-} \text{ or OH)} & \rightarrow \text{peroxide or hydroxyl intermediate} \\
& \rightarrow \text{Degraded or mineralized products}
\end{align*}
\]

The equation (4) depicts the absorption of light by the dye molecule (Dye*). This excited dye (Dye*) injects an electron to the conduction band of ZnO where it is scavenged by O₂ to form active oxygen molecule as shown in equation (5) and (6). Further active oxygen molecule formed in equation (6) subsequently reacts generate OH radicals as well. This active radicals drive the photodegradation or of the dye molecule.

Experimental set up

Chemicals: All chemicals used are of AR grade. Acridine orange (Aldrich) was used. The photocatalyst used was ZnO. The basic structure of acridine orange is:

\[\text{\includegraphics[width=0.5\textwidth]{acridine_orange_structure.png}}\]

Apparatus: The photocatalytic reaction was carried out in a batch reactor with dimension of 7.5 x 6 cm (height x diameter) provided with a water circulation arrangement in order to maintain the temperature in the range of 25-30°C. The irradiation was carried out using 500W halogen lamp. In all cases during the photolysis experiments, the slurry composed of dye solution and catalyst was placed in the reactor and stirred with a magnetic stirrer while exposed to visible light. Samples were withdrawn at periodic intervals from the reactor to assess the extent of decolourisation and degradation. The intensity of light was measured by a luxmeter (Lutron LX-101). The pH was constantly monitored using a pH meter. A systronics UV-VIS spectrophotometer (Systronic 106) was used for measuring absorbance at different time intervals. The photographs are given below:

At the start of the experiment
After 90 min

Procedure

Chemistry students investigated the kinetics of the photocatalytic disappearance of acridine orange dye solution in a batch reactor in the presence of ZnO. The absorbance maximum was determined by varying the wavelength and recording the absorbance. A graph between of wavelength on abscissa and absorbance on the ordinate gives \(\lambda_{max}\). The spectrophotometer was set on 490 nm. The suspension is stirred in the dark for 60 minutes before irradiation to achieve an equilibrated adsorption as deduced from the steady-state concentrations. Then 50 ml of dye solution with 250 mg ZnO was taken in a batch reactor and placed on a stirrer. The small amount of solution was taken out periodically and centrifuged to measure the absorbance on spectrophotometer. The Effect of irradiation time in absence of ZnO, in absence of light and in presence of light and ZnO were studied (Figure 1). If a rotator is not available, students can shake the reaction mixture by hand.

Results and Discussion

Students obtain the pseudo first order rate constant, \(k'_0\), for the degradation of the dye by plotting In (Co/C), (where C is the absorbance of the dye at time t and Co is the initial solution absorbance), versus time; they should obtain a straight line with a slope equal to - \(k'_0\) (Figure 2). In
this case the representative data are given in the Table 1, which gives the rate constant value as $k_0' = 3.45 \times 10^{-2}$ min$^{-1}$. Table 1 has a column describing the % degradation with time. This has been calculated by simple determination of percentage and then subtracting it from 100.

**Table 1:** Effect of irradiation period  
Amount of ZnO=250 mg  
PpH=6.4  
Dye concentration=2.0 $\times$ 10$^{-4}$M  

<table>
<thead>
<tr>
<th>Irradiation Period (min)</th>
<th>absorbance</th>
<th>% decolourisation</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.916</td>
<td>0</td>
</tr>
<tr>
<td>15</td>
<td>0.448</td>
<td>51.09</td>
</tr>
<tr>
<td>30</td>
<td>0.328</td>
<td>64.19</td>
</tr>
<tr>
<td>45</td>
<td>0.171</td>
<td>81.33</td>
</tr>
<tr>
<td>60</td>
<td>0.101</td>
<td>88.97</td>
</tr>
<tr>
<td>75</td>
<td>0.071</td>
<td>92.24</td>
</tr>
<tr>
<td>90</td>
<td>0.039</td>
<td>100</td>
</tr>
</tbody>
</table>

$k'=3.45\times10^{-2}$min$^{-1}$

Once the students have mastered the experiment using standard conditions, they can plan their own experiments to probe the details of the degradation process. Students should be encouraged to develop their own ideas as well.

**Figure 2** Representation of pseudo first order kinetics  
Amount of ZnO = 250 mg, pH = 6.4  
dye concentration=$2.0 \times 10^{-4}$M

**Dye concentration**

The students should determine how the variation of dye concentration affects the observed degradation rate constant. In the range chosen (Table 2) the rate decrease with the increase in the dye concentration from 9.0 $\times$ 10$^{-4}$ mol dm$^{-3}$ to 2.0 $\times$ 10$^{-5}$ mol dm$^{-3}$. The decrease in $k_0'$ values with increase in initial concentration of the dye can be ascribed to the following reasons:

**Table 2:** Effect of dye concentration  
Amount of ZnO = 250 mg, Light intensity = 350,00lux

<table>
<thead>
<tr>
<th>[Acridine Orange]/mol/L</th>
<th>$k_0'/10^{-2}$ min$^{-1}$*</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.0$\times$10$^{-4}$</td>
<td>8.98</td>
</tr>
<tr>
<td>9.5$\times$10$^{-4}$</td>
<td>7.36</td>
</tr>
<tr>
<td>1.0$\times$10$^{-3}$</td>
<td>6.90</td>
</tr>
<tr>
<td>1.5$\times$10$^{-3}$</td>
<td>6.12</td>
</tr>
<tr>
<td>2.0$\times$10$^{-3}$</td>
<td>3.45</td>
</tr>
</tbody>
</table>

(1) Decrease in the path length of photons entering the solution due to impermeability of the dye solution. At low concentration the reverse effect observed, thereby increasing the number of photon absorption by the catalyst [7]. (2) The increase in requirement of catalyst surface for the increased concentration of the dye. The irradiation time and amount of catalyst has been kept constant. Hence the relative number of O$_2^\cdot$ and OH$^\cdot$ radicals formed on the surface of ZnO are also constant. As a result the relative number of O$_2^\cdot$ & OH$^\cdot$ attacking the dye molecule decrease with increasing initial concentration of the dye. Hence, the rate degradation decrease considerably with increase in concentration of the dye.

**Effect of ZnO loading**

Students should observe the effect of mass ZnO used. It was found that with 250 mg/50ml ZnO, $k_0'$ is $3.45 \times 10^2$ min$^{-1}$ while increasing the mass to 350 mg/50 ml $k_0'$ becomes 7.43 $x10^2$ min$^{-1}$ (Table 3). But this trend does not follow on further increase in the ZnO mass. 400 mg/ 50 ml ZnO will give $k_0'$ values to 5.30 $x10^2$ min$^{-1}$. These observations can be rationalised in terms of availability of active sites on ZnO surface and the penetration of photo activating light into the suspension. Owing to an increase in the number of ZnO particles as the concentration of catalyst increased the availability of active sites increase but the light penetration and hence the photoactivated volume of the suspension shrinks. The trade off between these two effects is that at low solute concentration, when there are excess active sites, the balance between the opposing effects is evenly poised. At high solute concentration availability of excess active sites outweighs the diminishing photoactivated volume and substantially high $k'$ value is obtained at increase in ZnO concentration. The increased amount of ZnO increase the quantity of photons absorbed as well. Further increase in catalyst concentration beyond 250mg/50ml may result in deactivation of activated molecules due to collision with ground state molecules as shown below.

ZnO$^\ast$ + ZnO $\rightarrow$ ZnO#$^\ast$ + ZnO  
ZnO$^\ast$ : ZnO with active species adsorbed on its surface  
ZnO#$^\ast$ : deactivated form of ZnO$^\ast$ shielding by ZnO may also take place

**Table 3:** Effect of ZnO loading  
Initial concentration of dye = 2.0$\times$10$^{-5}$ mol/L,  
Intensity of light=350,00lux

<table>
<thead>
<tr>
<th>Amount of ZnO(mg)</th>
<th>$k_0'/10^2$ s$^{-1}$*</th>
</tr>
</thead>
<tbody>
<tr>
<td>250</td>
<td>3.73</td>
</tr>
<tr>
<td>300</td>
<td>5.40</td>
</tr>
<tr>
<td>350</td>
<td>7.40</td>
</tr>
<tr>
<td>400</td>
<td>5.30</td>
</tr>
<tr>
<td>450</td>
<td>4.21</td>
</tr>
</tbody>
</table>

**Conclusions**

This manuscript describes pseudo first order kinetics, spectrophotometry and the kinetics of heterogeneous catalytic reaction applied to wastewater treatment. Students get insight into the mechanism of the photocatalytic degradation and how a photocatalyst works. It’s a novel experiment for the determination of the kinetics of photocatalytic degradation of pollutants in aqueous solution. It further substantiate the importance of AOPs in clean technologies. The experiment is safe and simple. In addition to this, the experimental is also suitable to stimulate the student to further research activities.

To be continued on page 38
Richard Watson, Bishop of Llandaff (1737-1816):
A chemist of the chemical revolution.

Dr Bill Palmer
Charles Darwin University,

Abstract
The chemical revolution is usually placed in the latter part of the eighteenth century, during which time there was a rapid advance in the understanding of chemistry. Behind the very famous and well-known names with their myriad discoveries are many other chemists who made important contributions to the progress of chemistry. One of these, Richard Watson, will be the subject of this essay. I came across one of his five books of essays when doing historical research for my doctoral thesis and was impressed by the simplicity and elegance of his writing. Since then I have managed to collect his five volumes of Chemical essays, his two volume autobiography called Anecdotes of the life of Richard Watson, Bishop of Llandaff, his Apology for the Bible and one volume of his Collection of theological tracts in six volumes. His is an interesting personality that illustrates a number of paradoxes both about the nature of chemistry at that time and the use of scientific expertise as a means of social advancement.

Introduction
I have been fortunate in being able to assemble a variety of materials for this study. It is my intention to harmonise newer and older sources to explore the character, social relations, political and religious views and chemical innovations of Richard Watson more thoroughly than has been possible so far. Watson was unpopular and the reasons for his unpopularity will become evident.

My major sources are Watson’s own writings, Gregory (1798), Anon (1917), Partington (1937, 1960, 1965), Bartow (1938), Coleby (1953), Clow & Clow (1952), Brain, (1882) Musson, & Robinson (1989), Laidler (1995) and various biographies of scientists and others that mention Watson. Brain’s (1982) doctoral thesis is very thorough dealing with the whole of Watson’s life and Brain’s overall assessment of this enigmatic character is that he will be remembered by history as an Anglican bishop who supported the cause of increased civil rights for both Catholics and Protestant dissenters.

Lineage and early years
Richard Watson was born in August 1737 in Heversham, Westmoreland (Magnusson, 1990). His father was headmaster of the grammar school (appointed in 1698) and a clergyman. Richard says of him that ‘he taught with great reputation for nearly forty years’ (Watson, 1818a, p. 5). The father died in November 1753, leaving Richard, his younger son, the sum of £300 to provide for his education. Richard’s mother was Agnes Watson (née Newton) and he had an elder brother, Antony and sister, Agnes (Brain, 1982). Watson does not mention the Christian names of his mother or siblings in his autobiography, but mentions his mother ‘imbuing his young mind with the principles of religion’ and his own ‘filial piety’ (Watson, 1818a, p. 11). In 1754, Richard started his university studies at Cambridge as a sizar, feeling very much alone (Watson, 1818a, p. 10). A sizar is a position for less wealthy students where they are required to undertake some menial tasks. He endured this and worked extremely hard by his own account and obtained a scholarship on 2nd May 1757 (Watson, 1818a, p. 14). In his autobiography, Watson is quite open about his ambition, his continual seeking of personal advancement and his ever-present need for more money: he thus appears to modern eyes as selfish and self-seeking, but perhaps judgement should be reserved. He studied languages, Latin, Greek and Hebrew, and also mathematics and natural philosophy.

To achieve his ambition he needed to have a wide range of social contacts and initially these were largely confined to his university peers and lecturers. For example, he knew and to some extent befriended, by his account (Watson, 1818a, pp. 30-32), William Paley, later to become Arch-Deacon of Carlisle, author of Natural theology and the editor of the Bridgewater treatises. Clarke in his biography of Paley writes ‘…and Paley, who was generally well-disposed to his fellow men, maintained a hearty dislike of Watson, a feeling which those who know the latter’s autobiography may be disposed to share’(Clarke, 1974, p. 8).

Watson continued to climb the hierarchy. In 1759 he obtained his BA, was made second wrangler and was made a fellow next year working as an assistant tutor to Mr Backhouse (Watson, 1818a, p. 33). He then obtained his MA and became a moderator two years later (Bartow, 1938, p. 104). Unexpectedly the Professor of Chemistry, Dr Hadley, died in 1764. Watson was unanimously elected to the vacant position, in spite of his lack of knowledge of chemistry. Watson himself said that ‘At the time that this honour was conferred upon me, I knew nothing at all of Chemistry, had never read a syllable on the subject; nor seen a single experiment in it; but I was tired of mathematics and natural philosophy’ (Watson, 1818a, p. 46). Watson immediately sent to Paris for an ‘operator’ and buried himself in his laboratory. After 14 months’ preparation he ‘read a course of chemical lectures to a very full audience’ (Watson, 1818a, p. 46). During this time, Watson learnt chemistry from Baumé’s Manuel de chymie (Partington, III, 1965, p. 90), which he rated highly.

In 1766 he applied to the Prime Minister, Lord Rockingham, through the Duke of Newcastle, to obtain
Edward Gibbon wrote the twelve volumes of himself open to the charge of treason. Seeking independence, when at war with Britain, he left to take moral courage as in arguing for the Americans “left of centre”, a moderate, or a “wet”. His stance did not equal rights for the various religious dissenters (non-Anglicans). In today’s political terms he would be called “atheistic/deistic movements of the time; and he sought staunchly argued for Christianity against the popular Christianity (The Internet Encyclopedia of Philosophy, An address to the people of Great Britain). Watson destroyed all his chemical papers (see preface, Chemical Essays, Volume 4) and although his essays were reprinted many times after 1786, this can be taken as the finale of his scientific career, though there is other evidence that he long retained some interest in chemistry (Mills, p. 426). In 1786 his old friend, John Luther, died leaving him an estate in Sussex, so he used the funds on selling this to purchase a property, Calgarth, in Westmoreland. His health deteriorated and in 1786 and arranged to pay a deputy, Thomas Kipling, to carry out his duties at Cambridge.

On recovering, he built a new grandiose mansion for himself and his family at Calgarth, now that he had an income that more than covered his needs. He was able to attend the House of Lords as Bishop of Llandaff, but evidently only visited Llandaff triennially. He had no further duties in Cambridge and he ceased his scientific work.

In 1794, Thomas Paine wrote The age of reason, an attack on the bible (see-The Internet Encyclopedia of Philosophy) to which in 1795, Watson responded with a well-reasoned counter-attack (An Apology for the Bible in a Series of Letters Addressed to Thomas Paine), which was one of the most popular of his writings (Watson, 1828). Then Watson (1798) wrote An address to the people of Great Britain.

He continued writing religious pamphlets and busied himself with writing his autobiography and improving his estate. In fact, the improvement of the estate at Calgarth can be seen as his last scientific endeavour. Brain (1982, p. 38) writes ‘Watson mastered the skills of agriculture in the same way as he had taught himself chemistry and divinity’. Knight (1998, p. 96) goes as far to ascribe to him an underlying motive for teaching his Cambridge students applied chemistry, which he sees as a course in cameralistics (how to manage large estates).

Watson is credited with draining and improving the pasture around Calgarth and with planting large numbers of larch trees for financial return (Brain, 1982, p.38), which could be said to be putting his ideas on cameralistics into practice. In 1806 and 1808, he wrote papers on agriculture and was mocked by the poet Wordsworth for his ‘vegetable manufactory’ (Anon, 1917, p. 937).

He did not attend the House of Lords after 1807. In 1809 he had a stroke, followed by another in 1811. His eldest son, Charles Luther, died in 1814. In his later years Watson

Watson’s political religious and wider scientific connections
Watson busied himself with the study of chemistry and evidently gave lectures personally with well-prepared and competently demonstrated experiments. This was an unusual practice at that time as many university positions were treated as sinecures. In 1771 there was another death, this time of the Professor of Divinity, Professor Thomas Rutherford (Anon, 1917, p. 935; Watson, 1818a, p. 56). Watson admits that he had hoped for the position after he had obtained a degree in divinity, but he did not have this qualification and a degree in divinity was an essential qualification for the chair of divinity. There were only seven days to gain the qualification, so he traveled to Windsor (Coleby, 1953) and obtained the King’s mandate for a Doctor’s degree. He was appointed as Professor of Divinity at Cambridge at the age of 34 (Partington, 1937, p. 820). Again he had taken a huge risk in seeking a position of authority when he really had insufficient scholarship to undertake the task. Yet by exceptionally hard work and a concentration on the New Testament, he wrote extensively both books and pamphlets on the issues of the day, combining his religious and political ideals. Gradually he strengthened his position and his income. Income was dependent on being appointed to benefices, which had income attached to them. By the end of his life he had obtained fourteen such livings (Manchester Literary and Philosophical Society, 2003).

Watson married Dorothy Wilson, daughter of Edward Wilson of Dallam Tower in Westmoreland on 21 December 1773. Watson’s politics were Whig (liberal): he was against slavery; he supported the American colonists in their war of independence against Britain; he had sympathy for the ideals of the French Revolution; he staunchly argued for Christianity against the popular atheistic/deistic movements of the time; and he sought equal rights for the various religious dissenters (non-Anglicans). In today’s political terms he would be called “left of centre”, a moderate, or a “wet”. His stance did not take moral courage as in arguing for the Americans seeking independence, when at war with Britain, he left himself open to the charge of treason.

Edward Gibbon wrote the twelve volumes of The history of the decline and fall of the Roman Empire and some of his chapters caused offence to Christians. In 1776 Watson responded to Gibbon in a pamphlet, Apology for Christianity (The Internet Encyclopedia of Philosophy, 2003), with reasoned argument rather than biting criticism and thereafter Gibbon treated him with respect. Also in 1776 he offended the court by a preaching a Whig sermon, which later contributed to his lack of promotion to higher ranking bishoprics. In 1779, Watson was appointed Archdeacon of Ely. In July 1781, he became very ill and his wife probably saved his life by giving him an emetic against medical advice (Brain, 1982, p. 25). In 1782, the Bishop of Salisbury died, leaving a vacancy, Watson was able to move to the rank of bishop; he was appointed Bishop of Llandaff, the most junior bishopric. ‘In this manner did I acquire a Bishopric. But I have no great reason to be proud of the promotion...’(Watson, 1818a, p. 153).

In 1785, he wrote his six volume work called Theological tracts (Watson, 1791), which were heavily criticised by many in the Church of England as being too liberal.

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was unable to complete his memoirs and he died on 4 July 1816. He was buried at the church of St Martin’s, Bowness. His son Richard completed and published his memoirs. His wife Dorothy passed away in 1831.

**The influence of Watson’s scientific writings**

Watson is probably best known scientifically for writing the five volumes entitled *Chemical essays*. These writings inspired others and his research in chemistry laid foundations on which others built. This was his most important scientific achievement.

Watson’s *Chemical essays* were widely thought to have been a useful introduction to chemistry. These essays popularised chemistry as a distinct mode of study and helped future chemists. Several famous persons were influenced by reading Watson’s *Chemical essays*, for example The engineer, Thomas Telford, took notes from the *Essays* and always carried these notes with him (Musson & Robinson, p. 75). Musson & Robinson, (1989, p. 168, Note 4) and De Quincey, (1970, p. 83) state that the *Chemical essays* were still highly regarded by chemists such as Davy and Thomson. Thackray (1972, p. 64) states that John Dalton thought the essays were a good introduction to chemistry. Knight, (1989, p.86) points out that Edward Gibbon also thought the essays were the best standard textbook of the time. Rev George Gregory (1798) dedicated his encyclopaedia *The economy of nature explained* to the Lord Bishop of Llandaff. Cooper (1991) states that even Samuel Johnson when travelling by carriage was ‘chiefly occupied in reading Dr Watson’s second volume of *Chemical essays*, which he liked very well’.

**Watson’s scientific progress and relationships.**

Richard Watson was said to have discovered platinum Lockemann (1959, p. 90), but this is untrue, though it is a furphy of long-standing.. However Anon (1917) states ‘The introduction of platinum wrongly ascribed to him (Watson) belongs to William Brownrigg’. It is interesting to reflect that a misconception, evidently known in 1917, was still uncorrected in 1959. Watson’s inter-relationships with some other well-known English scientists of his time was investigated through their biographies. William Paley had a hearty dislike of Watson; Joseph Priestley believed Watson to be insincere. Fay’s biography of Benjamin Franklin mentions a Dr Watson as a close associate several times (Fay, 1929, p. 73: p. 271). Schofield (1966) mentioned that Joseph Priestley, Benjamin Franklin and a Dr Watson are indeed close personal acquaintances, but the ‘Dr Watson’ referred to is Dr William Watson, not Richard Watson.

Thus there seems to have been little personal contact between these contemporary scientists and Richard Watson on the evidence available. However Watson was ‘active in the English scientific community’ (Brain, 1982, p. 38) and was a member of a number of scientific societies. He was elected to the Royal Society in February 1769 as a result of his paper on the solution of salts which was published in *Philosophical Transactions* and later in his chemical essays. He became one of the Vice-Presidents of the Royal Society and would have met Cavendish, Franklin and Priestley at Royal Society meetings. When Sir Joseph Banks (President of the Royal Society) was under attack by a group of Royal Society members (The Malcontents), Watson spoke to calm the situation and defend Banks. A picture of the embattled state of the Royal Society in the late 18th century is available (Gascoigne, 1999, pp. 171-184) but Watson is not mentioned.

Watson was also a member of the Manchester Literary and Philosophical Society (see MLPS), which would have provided other contacts. For example, Priestley, Franklin, Lavoisier and Banks are all listed as honorary members. Musson & Robinson (1989, pp. 23-24) quote a Mrs Montagu as telling her friends that she had entertained ‘Sir Joseph Banks, the Bishop of Llandaff and all our distinguished philosophers’, so there were occasions when these scientists met together.

Watson was a founder member in 1788 of ‘The Association for promoting the discovery of the inland districts of Africa’ (Brain, 1982, p. 60). Sir Joseph Banks also became a member, so the two men had some common interests. The aim of the society was to sponsor expeditions with a scientific purpose to explore unknown regions of Africa (Africa, 2003).

Here are some of the views of a variety of writers about different aspects of Watson’s character. In Thomas De Quincey’s *Recollections of the lakes and the lake poets* there is a lengthy character description of him, studded with numerous little stories about him. As shown in the brief quotations below, De Quincey (1970, pp. 82-91) provides both favourable and unfavourable opinions, but in the end there is the impression that Watson’s culture and knowledge is only a thin veneer, that he is a buffoon, but a likeable one, and that he is not sincere about either his chemistry or his religion. For example:

He talked openly at his own table, as a Socratinian; ridiculed the miracles of the New Testament, which he professed to explain as so many chemical tricks or cases of politic legerdemain; and certainly had as little devotional feeling as any man that ever lived. (1970, pp. 86-87)

Yet De Quincey is quoted (The 1911 Encyclopedia, 2003) as expressing an opposite view where he states that ‘His lordship was a joyous, jovial, and cordial host’. The Reverend Gregory in his encyclopaedia quoted Richard Watson’s experiments on evaporation (Gregory, 1798, Vol II, p. 75) and on boiling (Gregory, 1798, Vol I, p. 128.) and on the origin of pit coal (Gregory, 1798, Vol II, p. 269). Knox, (1996 p. 176), when explaining Sayer’s contemporary cartoon (1787) of Watson, observes that Watson wishes to sever his chemical reputation from his political views; that is he wished to dissociate himself from an image of a ‘chymical empirick’. Sherwood Taylor, (1948) is critical of Watson, accusing him of making ‘little or no attempt to grapple with the unsolved problems of the subject…’

Further, De Quincey (1970) recalled the opinions of
the poets Coleridge and Wordsworth. Evidently Watson was referred to using the ‘contemptuous sobriquet “Dull Daniel”’ (p. 85), perhaps because he did not seek the company of others interested in science who lived within twenty miles radius of him. These included Coleridge (Levere, 1993a), Wordsworth (Space with Wordsworth, 2003) and John Gough who helped the young Dalton (Thackray, 1972, p. 15; Greenaway, 1966, p. 63). In telling the story of a meeting between Coleridge and Watson, De Quincey (1970) implies that Watson was a dullard, whilst Coleridge had great ‘scholastic erudition’ (p.89). Wordsworth did write to Watson, though his letter was never sent; it is largely a political attack on Watson’s lack of support for republicanism (Butler, 1986, pp. 224-226). In view of Watson’s considerable achievements, it is difficult to accept De Quincey’s contemporary account as unbiased.

Watson had a good general knowledge of science. He had studied natural philosophy and mathematics, improved his chemistry to a stage where he was at the frontiers of research for the period, been interested in anatomy and dissection, and finally worked at scientific agriculture. His interest in anatomy and the syllabus then taught at Cambridge as evidenced in Chemical Essays, Volume V, indicate that he was one of the early chemists interested in what in the 21st century would be called biochemistry. His essays show him to have been involved in practical rather than theoretical concerns, though his thoughts on phlogiston are widely quoted and indicate a slightly sceptical down-to-earth approach (Hall, 1954. p. 327: Toulmin & Goodfield, 1968, pp. 239-240: Crosland, 1971, p. 135).

Watson was particularly concerned with accurate measurement and is credited with the invention of a black bulb thermometer (Watson, Essays 5, pp. 193-194).

Watson was one of the first chemists to observe a variety of manufacturing industries for himself and to advise the owners on chemical problems relating to that industry. He would find a place in modern chemistry as a consulting industrial chemist.

Watson’s contributions to science
Overall Watson wrote forty-three essays so the comments below relate to those essays that appear most significant.

1. Perhaps one of the most important separation/purification processes in modern mining is froth flotation, the removal of particulate matter by foaming (IUPAC, 2003). The Australian historian, Geoffrey Blainey (2003, pp. 255-256), who, in the following account of flotation of lead–zinc sulfide ores at Broken Hill in Australia, credits Bishop Watson with the first recorded idea about flotation as a means of separating mixtures.

The flotation process resembled bubbles rising in a glass of beer and in fact brewers were amongst its first architects. The strength of the rising bubble had been observed as early as 1789 by Richard Watson, Welsh bishop and Fellow of the Royal Society, who put powdered lead ore into an ale-glass of water and added nitric acid. He observed that the acid generated air bubbles, and the bubbles attracted the mineral to the surface (pp. 255-256).

Blainey goes on to say that no commercial use was made of the observation at the time. However, if Watson is credited with even a small part in the idea of froth flotation, then his significance as a chemist is greatly increased as this process is of fundamental industrial importance.

2. Because of the huge breadth of the subject matter of his essays, different commentators give emphasis to different essays. Essay 10 (volume 1) deals directly with the subject of gunpowder and there are other essays on some of the constituents of gunpowder, for example Essays 7, 8, & 9 (volume 1) and essay 1 (volume 2). Almost all biographies mention his government work on gunpowder, which is estimated to have saved the government over £100,000 a year for several years (Coleby, 1953, p. 119; Partington, 1960, pp. 338-339, note 289; Russell (in press). Watson’s suggestion was to obtain the charcoal for the gunpowder by heating the wood in closed vessels. In this case Watson’s suggestions were implemented and Russell, (2000, p. 208) states that the manufacture of charcoal for gunpowder was introduced in Britain at Hythe in 1787. This meant that the government did very well on its investment of £100 a year towards the endowment of the Cambridge chemistry professorship.

3. When beginning his experiments in chemistry, Watson repeated the experiments of other scientists to check their results for himself. Watson checked earlier work by a chemist called Eller, who experimented to see if the volume of a solution varied with the amount of solute added. Eller concluded that there was no increase in volume when solutes dissolved in solvents. Watson repeated this work meticulously and showed that Eller’s earlier work was incorrect and wrote a paper Experiments and observations on various phenomena attending the solution of salts which was a model of scientific method (Chemical essays 5).

4. Blagden’s Law, if remembered at all, is now probably more commonly found within Raoult’s Law which covers the change in boiling point or freezing point due to the addition of solute (see Encyclopedia, 2003). Blagden’s Law states that the lowering of the freezing point is proportional to the amount of solute added for dilute solutions only (Walker, 1991, p. 95). However the credit for this law should really have gone to Watson who made the discovery in 1770 rather than Blagden who discovered the law in 1787. The work of both Blagden and Watson was forgotten, but the depression of the freezing point became an important physical method for determining the molecular weights of substances, so Blagden’s paper was brought into prominence and his name was associated with the law. Blagden should have been familiar with Watson’s earlier work but did not refer to it (Laidler, 1995, pp. 123-124). In 1921 Partington (1937, p. 820) pointed out that Watson should have had precedence. It is worth remarking that the quantitative work by both Watson and Blagden was extremely accurate for their period with their
emphasis moving from speculation to measurement.

5. On visiting various industrial enterprises Watson made numerous suggestions. When visiting the salt manufacturers at Northwich he made careful meteorological measurements. On the basis of his results he suggested that atmospheric evaporation of brine dispersed over suspended cloths would be the best method of obtaining salt (Clow & Clow, 1952, p.54: Essay 2, volume 2).

6. Several articles (for example, Mills, p.426; Partington, 1937, p. 820) point out that Watson recommended the recovery of volatile products from heating metal ores and from coke ovens in several of his essays. There were major problems with the effects of the effluents of these pollutants on people and on the countryside. In the period in which Watson was working these effects were starting to become apparent: ‘The only alternative was to have flues of an immense length, suggested by Bishop Watson in the late 1770s’ (Russell & Wilmott, 2000, p.305). The same authors go on to explain that some extended flues were built soon after and that one such flue built in about 1845 zigzagged its way uphill for five miles.

7. It was difficult to convert cast iron into malleable iron using coal, and charcoal from wood was evidently expensive. In 1782, Watson noted that coke could be substituted, but this produced an inferior product and it took much further work to solve the problem (Clow & Clow, 1952, p. 347: Essay 8, volume 2).

8. Sometimes processes were just described as in the case of manufacturing white lead from sheets of lead metal. The process was to treat the lead sheets in earthenware pots with vinegar, with dung added from time to time over several months. White scales were produced and these were ground up and used to make paint which clever and dishonest vendors adulterated with chalk. The process of manufacture was painfully slow. (Clow & Clow, 1952, pp. 381-382: Essay 10, volume 3).

Watson makes some witty remarks about ladies using lead based cosmetics, not using them in Harrogate or similar spas as ‘they were suddenly despoiled of all their beauty and changed quite black by the sulphureous waters’. (Clow & Clow, 1952, p. 385: Essay 10, volume 2, p.365). This is a reference to the fact that most lead salts are turned black by hydrogen sulfide as lead II sulfide is formed.

Conclusion
Watson was hardworking, driven by an ambition to succeed and to become rich, though he did act with more compassion and concern for the poor and for slaves than many of his contemporaries. He had success in the chemical, religious, political and agrarian fields, though he had failures in all these areas too. His single-minded determination to succeed did bring him many enemies and some of the harsh comments about his character may be due to personal jealousy. His life and achievements should be remembered by all chemists, but particularly by physical and industrial chemists. In Australia, where froth flotation is important perhaps there is an especially good reason to remember his contribution to chemistry.

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