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Computer Software to prepare students for Laboratory work

Software para preparar estudiantes en trabajo de laboratorio

Jane Tomlinson, Peter O'Brien and John Garratt,

Department of Chemistry, University of York ,York YO10 5DD, UK

Email cjg2@york.ac.uk

Abstract

The eLABorate project has prepared a range of computer-based packages which is designed to complement laboratory work by allowing students to practice different skills associated with experimental science. As part of this project we have designed a software package to prepare students for an organic synthesis using the Horner-Wittig reaction. The software is in four parts which can be accessed independently and at will from a menu. Part 1 simulates the weighing of the required amounts of starting material. Part 2 deals with the calculation of the appropriate volume of the two other reagents. Part 3 uses animated line-drawings to demonstrate the equipment and procedure to be used. Part 4 shows how the chromatographic procedure for purifying the product is rationalised. Observation of and discussion with students during and after the laboratory session provides evidence that the use of the software provides an effective learning experience.

Key Words computer simulations, pre-labs, organic synthesis.

Resumen

En el proyecto eLABorate se han preparado diferente programas para complementar el trabajo del laboratorio y permitir a los estudiantes practicar las diferentes habilidades en la ciencia experimental. Como parte de este proyecto se ha diseñado un paquete de software para preparar a los estudiantes para una síntesis orgánica que usa la reacción de Horner-Wittig. El software tiene cuatro partes que pueden utilizarse independientemente de un menú. La parte 1 simula la operación de pesar las cantidades requeridas de sustancia; en la parte 2 se calcula el volumen apropiado de los otros dos reactivos. En la parte 3 se utilizan las animaciones para demostrar el equipo y procedimiento para la reacción. La parte 4 muestra cómo utilizar la cromatografía para purificar el producto . Las observaciones y discusiones con los estudiantes durante y después de la sesión del laboratorio proporcionan la evidencia de que el uso del

software permite lograr el mejor aprendizaje .

Palabras clave: simulaciones por el computador, pre-laboratorios, síntesis orgánica.

Introduction

Chemistry is a discipline which is built on observation and experiment. Laboratory work is therefore an essential part of all undergraduate chemistry courses. However, in most courses most laboratory work involves the students in following recipes (Meester and Maskill, 1994). We have argued elsewhere (Garratt, 1997) that there are good reasons for this; in particular it *simulates* what most experimental scientists actually **do** when they work at the bench. A key difference is that professional scientists define their own objectives and design their own recipe to achieve these objectives, and so understand the recipe which they are following. This illustrates our point that, with a simulation, some aspects of reality are sacrificed in order to focus attention on others; in most laboratory work the aspects of reality which are sacrificed are the **choice** of the system to experiment on and the **choice** of the method to use, and yet these are key skills for an experimental scientist. Thus laboratory work is only one of the skills which a professional scientist needs to develop; others include planning investigations, selecting appropriate methods, devising experimental procedures or protocols, interpreting results and presenting these results to the scientific community. The scope of *practical work* must be broadened to take account of this, if it is to prepare students adequately for work as experimental scientists.

A further problem with laboratory work is that students frequently do not spend time trying to understand the theoretical background to their laboratory work, nor do they reflect retrospectively on the lessons they may have learned. This means that the intellectual return from time spent is rather small. Johnstone is one of the chief advocates of the benefits of pre-labs and post-labs to improve the learning experience of laboratory work (Johnstone, 1997 a,b).

The *eLABorate* project (Garratt et al, 1997) was established in order to generate computer-based resources which would complement laboratory work and provide contexts in which students could develop more of the skills of experimental work.

The first *eLABorate* resources were simulations based on accurate models of the relevant system. Thus *enzymeLAB* generates rates of reactions catalysed by an enzyme which obeys Michaelis Menten kinetics ($v = (V_{max} * S)/ (K_m + S)$). The simulated rate depends realistically on the pH and on the concentrations of enzyme and of substrate in the assay system, and the displayed value of rate (v) has an 'experimental error' with a standard deviation of 5% of the correct calculated value. Another example is *equilibLAB*, which simulates the pH of a solution of a diprotic acid when it is titrated with NaOH. Appendix A lists available *eLABorate* products and provides references to previously published descriptions of this work. The brief descriptions of each package illustrate the flexibility and range of products, and may stimulate suggestions for other similar packages.

In our experience, *eLABorate* simulations can be an effective way of

- Preparing students for prescriptive laboratory work, by providing them with an opportunity to explore the consequences of varying the recipe;
- Providing students with the opportunity to explore theory beyond what can be done in the laboratory;
- Providing students with the opportunity to manipulate the controls of expensive equipment;
- Removing the constraint of time from the process of data collection, so that students can be allowed to make their own choices of parameter settings, and collect sufficient data for in-depth analysis and interpretation, and sufficient to justify the requirement that they write a report on a 'virtual investigation' in the style of a scientific paper.

Because the first *eLABorate* products relied on the availability of an accurate mathematical model of the system being simulated, it appeared inappropriate to use them to support laboratory classes dealing with synthetic organic chemistry. Nevertheless, students need to learn the skills of the synthetic organic chemist, and observation of students at the bench shows that they frequently lack understanding or appreciation of the procedures they carry out. We decided that careful observation of students in the laboratory would reveal misunderstandings and misconceptions which would indicate the kind of exercises which would be an effective preparation for a laboratory synthesis.

Here we describe the software we created, and our analysis of its value as a learning experience for our students.

The Laboratory Work

The laboratory exercise we chose was a Horner-Wittig reaction to synthesise a mixture of diastereoisomeric β–hydroxy phosphine oxides from an alkyl phosphine oxide. This is one of three experiments in the third year Advanced Training Module, first introduced into the course in 1997. In this example of the synthesis, pentyldiphenylphosphine oxide is the starting material. It is deprotonated using n-butyllithium and the resulting anion is reacted with p-anisaldehyde. The mixture of products, which includes the two diastereoisomers and excess p-anisaldehyde, is then separated using flash column chromatography. This exercise is included in the course because it gives experience and training in a number of important techniques which are essential for research in organic synthesis and which the students have not previously experienced. Of particular interest here is the deprotonation of the pentyldiphenylphosphine oxide using n-butyllithium which requires rigorously dry reaction conditions and must be carried out under an atmosphere of nitrogen. In addition, the deprotonation is carried out at –78^{o} C and the manipulations involve syringe/septa techniques.

Observation by one of us (PO'B) of the students carrying out this synthesis demonstrated four major deficiencies in their approach.

They lacked experience to make judgments about the amounts of reagents to use. Thus, in this synthesis the protocol specifies that approximately 500 mg of pentyldiphenylphosphine oxide should be used, and that 1.1 molar equivalents of n-butyllithium and 1.15 molar equivalents of p-anisaldehyde will be required. The laboratory script therefore instructs the students to weigh accurately approximately 500 mg of pentyldiphenylphosphine oxide and to calculate the required amounts of n-butyllithium and p-anisaldehyde. Many students perceived the instruction 'accurately approximately' as a contradictory statement, and went to great lengths to weigh out **exactly** 500 mg. In contrast to their concern to weigh exactly 500 mg, we noted that the students did not appear at all concerned that the equipment available provided insufficient precision to ensure accurate delivery of the required 1.1 or 1.15 molar ratios of the other reagents.

Many students lacked confidence in their ability to calculate the required amounts of n-butyllithium and p-anisaldehyde. Although the calculations are simple, they are not trivial because they involve more than the simple conversion of a mass into a relative molar mass. One involves calculating the volume of a solution of known molarity which contains the required amount of reagent, and the other involves using the density of the liquid p-anisaldehyde to calculate the volume which contains the required amount.

The students were inevitably unfamiliar with the technique of creating an inert atmosphere in the reaction vessel (since the synthesis was included in the course exactly to give them this experience for the first time). However, this unfamiliarity slowed down their work rate considerably.

They did not reflect on the strategy used either to detect or to separate the three components of the reaction mixture. They therefore fail to recognize that the principles of the chromatographic procedure can be applied much more widely, and also that results from analytical thin-layer chromatography can be used to devise a separation procedure using column chromatography.

The Software

The software we developed has four main sections, each dealing with one of the problems outlined above. These sections are titled "*Weighing*", "*Calculations*", "*Reaction*" and "*Column*". These are described below. We also included two other sections "*Introduction*" which introduces the user to the purpose of the package (i.e. to help them in the laboratory) and briefly describes how to use the software, and "*Overview*", which gives a brief summary of the other sections in the software. All sections can be accessed through a menu of icons which is always on screen. Students can move at will between the different sections. This means that the time taken to use the software is variable. We estimate that most students spend 30 – 60 mins at the computer.

Weighing

Figure 1 shows a screen-dump from the main screen of the Weighing section.

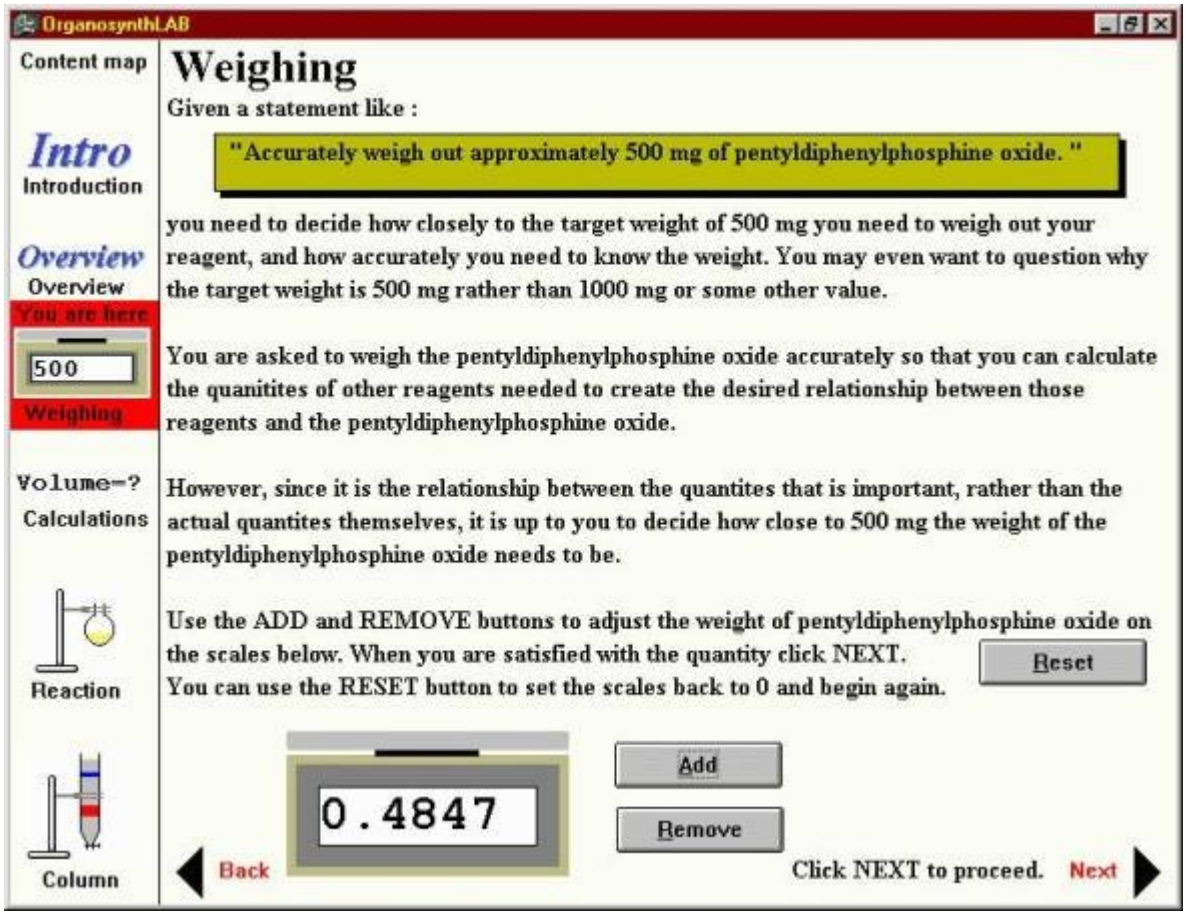

Fig. 1

The first part of the screen focuses on the task of interpreting the meaning of weighing 'accurately approximately'.

The box at the bottom of the screen allows students to add and subtract amounts of substance to the four-figure balance. The initial amount added is 300 mg with a standard deviation of 10% of the value. Subsequent additions (or subtractions) are calculated by taking the difference between the amount present and the 500 mg target and adding an 'error' value with a standard deviation which increases as the target is approached. This is not intended to simulate the experience of the weighing process; its value lies in the feedback it provides to the user. This is provided when the student indicates satisfaction with the amount weighed out. Most of the feedback message is the same for all students. It reminds them of the number of additions they made, and of the weight with which they were satisfied. It goes on to state our view of a reasonable range within which their weight should fall. This standard part of the feedback message reads

as follows;

Weighing Feedback

You made a total of ? additions or subtractions to the quantity of pentyldiphenylphosphine oxide you were weighing.

You were satisfied with a weight of 0.??? g of pentyldiphenylphosphine oxide.

Note that the number of additions and the weight obtained are the student's own values.

When this synthesis was designed it was assumed that the weight of pentyldiphenylphosphine oxide used would be 500 mg.

Although you do not need exactly 500 mg of pentyldiphenylphosphine oxide, you do need to be quite close to that weight, so that the whole protocol is appropriate.

In our judgement you should aim for a weight within 5% of 500 mg.

A student satisfied with a weight of 474 mg or less will get the following additional message indicating that this falls outside the range of +/- 5% of the target of 500 mg.

Your value is not within this range.

A student who is satisfied with a value within the range 475 – 525 mg, but who has made a large number of additions (or subtractions) to achieve this gets the following message (in which the weight and the

number of attempts is taken from the student actions)

You had weighed out 0.4823 g, which is also within the acceptable range, on attempt 4.

Accepting this value would have saved you time, without compromising your experiment.

When the student is satisfied with an amount between 475 and 525 mg on the first occasion that this is obtained, the additional feedback message is

The weight you were satisfied with is within this range.

You were right not to spend extra time getting closer to the target weight of 500 mg.

Calculations

Figure 2 shows a screen dump of one of the key screens from this section. The students first calculate the number of moles of the starting material (pentyldiphenylphosphine oxide) they have weighed out. This forms the basis of calculations of the volume of n-butyllithium giving a 1.1 molar equivalent, and the volume of p-anisaldehyde giving a 1.15 molar equivalent. Students who are unsure how to do the calculations, or who get incorrect answers, have the option of being taken step-by-step through the calculations. This runs alongside example calculations. The student's calculations are checked by the software at each stage, so that any mistakes are highlighted immediately. They can repeat this calculation as often as they like to give themselves practice; if they choose to do this, the program will provide them with a random value for the weight of the starting material from within the acceptable range.

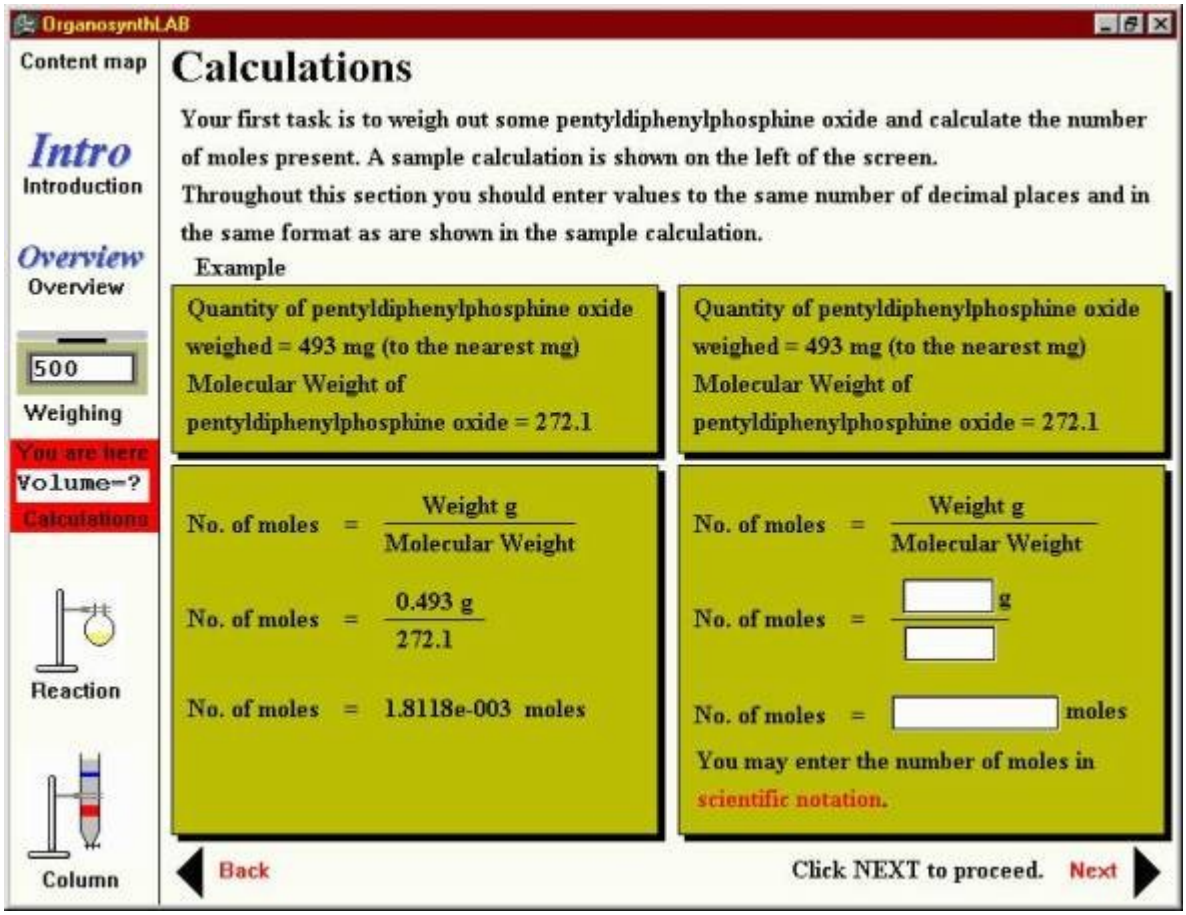

Fig. 2

Reaction

This section of the program contains two simple animations. One deals with the reaction itself and the other with the work-up of the product. Each animation consists of a number of short stages with its own accompanying text. Figure 3 shows, as an example, a single screen from one stage of the animation of the reaction. In the previous stage a balloon filled with nitrogen was used to replace the air in the flask. As the text commentary on the left of figure 3 implies, this stage shows moving sequences of

- the removal from the septum of a needle used to allow the air to escape from the flask;
- the removal of the septum and addition of reagent (shown at the bottom of the flask below the stirring bead);
- the replacement of the balloon.

The student controls the time at which the next piece of text commentary appears and the next stage of the animation is shown, so that the demonstration is paced to suit the individual.

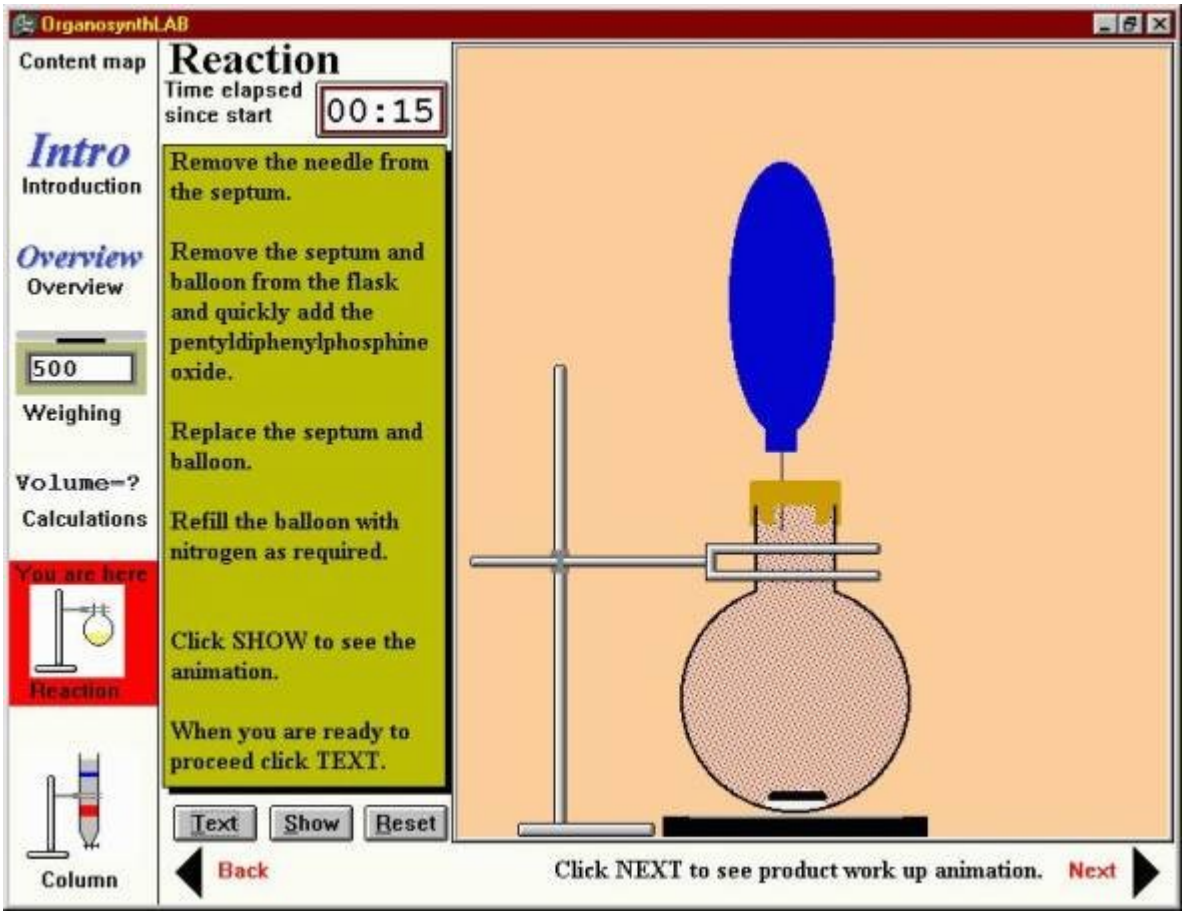

Fig. 3

Column

This section engages the student in selecting a suitable solvent system to apply to a column in order to separate the three components of the reaction mixture. The first stage uses a simple animated sequence of line drawings to demonstrate how the Rf of a compound measured on a thin-layer chromatography (TLC) plate can be used to predict the number of column volumes needed to wash that compound off a column. Figure 4 shows one of the screens from this sequence. The screen then displays six thin layer plates showing the Rf values of the three constituents of the reaction mixture which were obtained in the laboratory using pure light petroleum, pure ethyl acetate, and four mixtures of the two solvents. The students are first asked which of the solvents they would use for analytical purposes (ie using TLC to determine which of the constituents was present in a sample). Then they are asked to use the same data to

choose a suitable solvent to use on a column for preparative purposes, bearing in mind the need to compromise between maximum separation of the constituents and minimum volume of solvent needed to remove all the constituents from the column. The final stage of this section explains the strategy of changing the solvent in order to wash off the slowest running constituent as quickly as possible once the two fastest running constituents have been washed off.

Results and Discussion

During the Advanced Training Module, students complete three different experiments, one in each of three separate weeks during which the laboratory is open for 8 hours per day every Thursday and Friday. They are expected to spend a total of 11 hours per week on their laboratory work, including their preparation and write-up. In any given week, about 12 students, working on their own, are carrying out the Horner-Wittig synthesis. We introduced *organosynthLAB* as part of this exercise in 1999-00 as a result of perceived deficiencies in the students' preparedness for this work. The software was made available on the university's networked computers. This means that it is available on the 24-terminal classroom in the chemistry department, and in three other classrooms on the campus which are available to students 24 hours a day.

For the 1999-00 course, the laboratory manual included information about the availability of *organosynthLAB* on the university networked computers, and explained the benefits of using the software. In addition, one of us (PO'B) gave an oral introduction to the course in which he stressed the importance of using the software. No sanctions were applied to ensure that all the students actually used it, and no formal record was made of those who did. Observation of the students and informal discussion with them suggests that more than half of them took advantage of the availability of the software.

Weighing

Comparison of the laboratory reports with those from previous years showed a marked increase in the number of students who were satisfied with a weight of starting material which differed from the specified 500 mg. This is good evidence for the effectiveness of this section of the software. Nevertheless, the number of students who recorded 500 mg as the weight of their starting material was significantly greater than could be expected if these students had been satisfied on the first occasion when they recorded a weight within 5% of 500 mg. At first sight this is discouraging. However there are two explanations. The first is that these students may not have used the software, and therefore were in the same position as students in previous years. The second is that the concept of following instructions thoughtlessly and to the letter may be so deeply ingrained in some students that exposure to a single computer-based exercise is insufficient to remedy the situation. We believe that this latter is a serious possibility and we regard it as evidence in favour of introducing this (and similar) pre-lab exercises much earlier in the course.

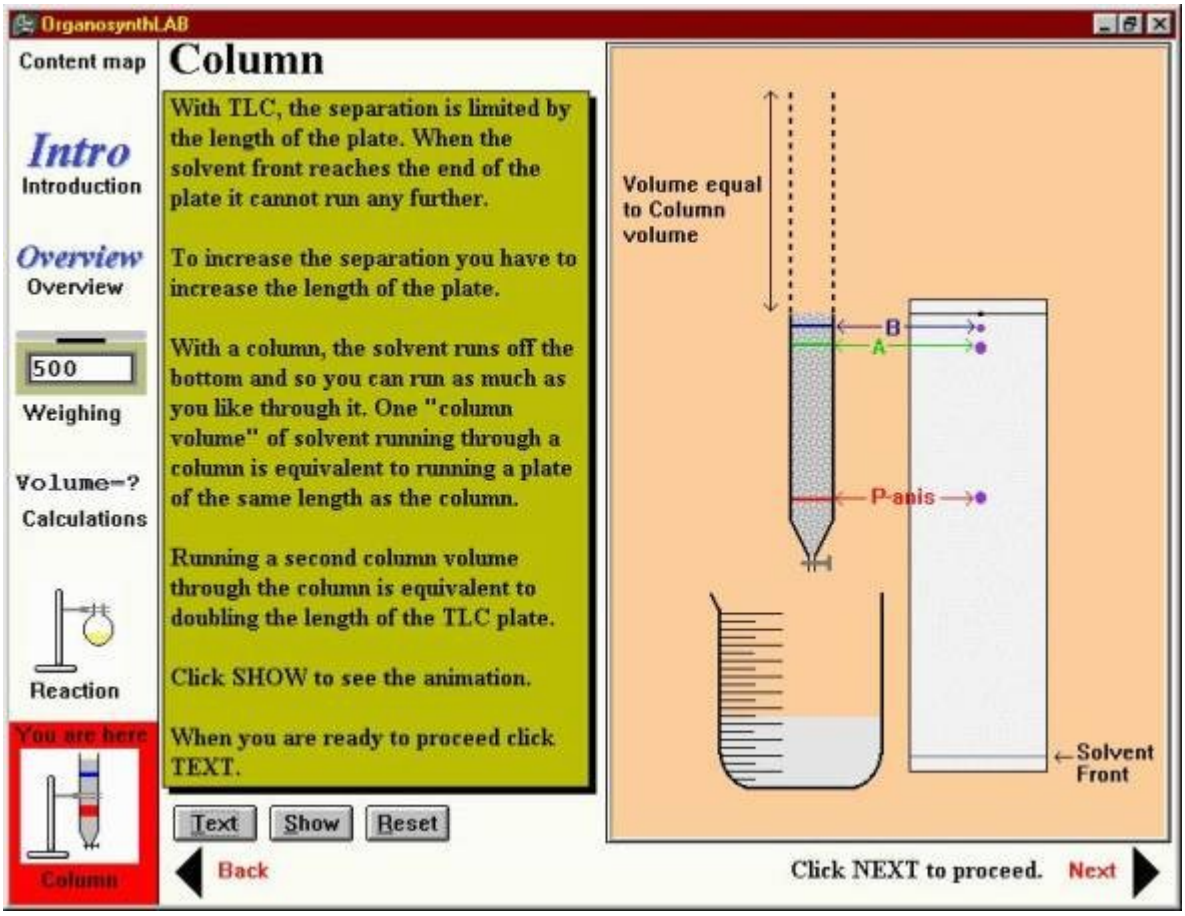

Fig.4

Calculations

This section of the software deals with straightforward calculations of a kind that third year students are generally assumed to be familiar with. It was included in the program specification because in previous years so many students sought help from laboratory demonstrators when doing these calculations. After the introduction of *organosynthLAB* the demonstrators confirm that there was a marked drop in the amount of time spent helping students with these calculations. This is consistent with the experience of Walker (Walker, 1999) who has shown the effectiveness of computer-based practice with numeric calculations.

Analysis of laboratory reports provides no information about the value of this section of the software because it gives no indication of how much difficulty the students had in completing the calculation correctly.

Reaction

We evaluated the effectiveness of this section of the software by observing the general attitude of the students in the laboratory and assessing the efficiency with which they set about their work. In comparison with previous years, these students showed greater confidence with procedure. Informal discussion with selected students confirmed that they felt comfortable with the technique even though aspects of it were completely new to them. It is not easy to evaluate whether this improvement in confidence led to increased effectiveness in the laboratory. However, even without any such evidence, increased confidence adds to the quality of the learning environment in that it allows a higher quality of student-demonstrator interaction.

Our observation of the students using the computers and our informal discussions with them during and

after the laboratory work have convinced us that animations have many advantages over video clips for the demonstration of certain techniques. It is not easy to demonstrate these advantages with screen dumps from the software because the essence of good animation is that (like a video clip of a real procedure) it is a moving sequence. The chief advantage of the animation is the same as that of any other *simulation*; it can omit distracting aspects of the real world (as represented by a video) in order that all of the student's attention is focused on carefully selected aspects of the procedure. The simple line drawings (illustrated by figure 3) give a clear representation of essential features which, our observation of the students convinces us, they can usefully carry with them into the laboratory. In many cases the sheer richness of a video clip provides too much information, and overloads the working memory space, so that the key lessons are not taken in (Johnstone 1984).

Column

This section of the software leads itself least well to evaluation because it is designed to provide additional background information, to broaden the students experience, and to encourage them to reflect on their laboratory work. In order to assess whether the students learned some principles of planning a strategy for chromatographic separation of a mixture, we would have to set specific tasks (preferably before and after carrying out the computer-based exercise) and use the results to evaluate whether they showed any improvement which could be attributed to their use of the software. On this occasion, we could not justify requiring the students to spend extra time in order to provide data for us.

We nevertheless regard the development of this section of the software as a worthwhile activity for ourselves. Drawing up the specification for the section has helped us to appreciate the steps involved in applying information from one experience (like thin layer chromatography) to a related problem (like devising a procedure for column chromatography). It is easy to see how this section of the software could be used in a tailored context to teach specific lessons of this kind. Furthermore, it has demonstrated that there is a range of new possibilities for the use of computer animations in the teaching of chemistry

Appendix A

eLABorate packages produced at the University of York (Garratt et al, 1999)

Elaborate packages are available for downloading from the web free of charge to academic institutions : http:// www.york.ac.uk/depts/chem/staff/elaborate

nmrLAB

a simulation of a high field FT NMR spectrometer

The aim of ***nmrLAB*** is to improve students' understanding of the process of FT NMR spectroscopy and to give them first hand experience of how (some of) the parameters chosen when an NMR spectrum is collected affect that spectrum. The program stores information about compounds as ideal spectra. The student enters values for such parameters as the duration of the pulse and the number of repeat scans, and the program simulates the FID which would be generated by a real spectrometer with those parameters. The students can then manipulate the FID before Fourier-Transforming to obtain a spectrum. The program can simulate simple ^{1}H, proton-decoupled ^{13}C, and ^{13}C DEPT experiments.

nmrLAB allows students to investigate the process of obtaining and manipulating an NMR spectrum for themselves, without needing access to a real spectrometer. It can also be used for spectral interpretation exercises.

enzymeLAB

Characterising an enzyme through kinetic studies (Clow and Garratt, 1998)

This program simulates experimental measurements of the rate of a reaction catalysed by an enzyme which obeys Michaelis Menten kinetics. The values of V_{max} and K_M, and their sensitivity to pH are selected at random from a realistic range of values stored by the program. Furthermore, the program determines whether a reversible inhibitor acts competitively, non-competitively or uncompetitively. Each user is allocated an enzyme with different characteristics.

The program is designed to allow students to plan their own strategy for carrying out a study as directed by the tutor. This may be a full characterisation of the enzyme (pH optimum, V_{max} and K_M at optimum pH, effect of pH on V_{max} and K_M, type of inhibition), or it may be a shorter study (for example to determine whether the optimum pH is affected by substrate concentration). When using the software, the student chooses experimental variables (amount of enzyme, concentration of substrate, pH, and concentration of inhibitors). ***enzymeLAB*** can be used flexibly, for example:

- to give practice with the planning of experimental strategy;
- to consolidate basic theory;
- to give practice with data interpretation and presentation.

statsLAB

empirical investigation of basic statistical procedures

statsLAB allows the student to study empirically the strengths and limitations of statistical procedures dealing with samples from a Gaussian population and with regression analysis. The student can set parameters for the parent population: for example the parent mean for a Gaussian population or the values of the independent and dependent variables in the relationships:

$$y = mx + c$$

and

$$y = x \exp(-kt)$$

The student can ask the computer to draw samples from the Gaussian population or use either of the two given relationships to calculate *y* values with a specified random error. Thus they can observe characteristics such as how the calculated standard deviation or standard error of the mean varies with the sample size, and how estimates of constant terms vary with the range of the independent variable.

electrochemLAB

an investigation of equilibrium electrochemistry

This program allows students to investigate basic principles of equilibrium electrochemistry in a way which is difficult in a real laboratory. There are six modules; each module simulates an electrochemical system where the student can set the conditions and see the results. Because the systems are simulated, a much wider range of conditions are possible than in the laboratory. For instance, in the laboratory, there is only a smallish number of different possible metal ion redox couples which can sensibly be used, which limits the number of standard electrode potentials which the student could examine. ***electrochemLAB*** can simulate a metal ion redox couple with any standard electrode potential within a sensible range.

The six modules in the program cover:

- redox equilibria
- qualitative electrochemistry
- quantitative electrochemistry
- activity
- temperature dependence
- potentiometric curves

The program is designed to allow students to investigate the systems for themselves. The package includes student notes with suggested investigations.

tracerLAB

A virtual investigation of the relationship between RNA and protein synthesis in E. coli

tracerLAB is a virtual investigation of the relationship between RNA and protein synthesis in various strains of *E. coli*. It allows the user to discover what happens to protein/RNA synthesis when RNA/protein synthesis is inhibited. The processes of protein/RNA synthesis are followed by tracing the incorporation of radioactive precursors, ^{14}C adenine and ^{3}H lysine into RNA and protein respectively, during exponential growth and after either protein or RNA synthesis has been inhibited. It is possible to follow the incorporation of the labelled precursors by using long-term labelling or pulse labelling.

tracerLAB allows the student to practise the skills involved in planning experiments and interpreting data. It can be used in

two different modes. In the default mode the student can select which bacterial strain to use in their investigation. In the alternative mode the student investigates the characteristics of an unknown strain of bacteria.

tracerLAB can be used flexibly, for example:

- to give practice with the planning of experimental strategy, data interpretation and presentation;
- to learn something of the nature of bacterial growth;
- to gain an insight into the power and limitations of using radioactive isotopes to monitor biological processes;
- to determine the differences (in terms of RNA and protein production) between strains of bacteria;
- to compare the information obtainable from the study of cumulative or pulse labelling.

equilibLAB

Titration of a diprotic acid

This program simulates the titration curve of a diprotic acid. It can be used in two different modes. In the default mode the pKs of the acid are selected by the students; in the alternative mode they are chosen at random from the two sets of the stored values. The student selects the size of the beaker and burette, the volume and concentration of the acid, and the concentration of the base together with the volume added. The program can be used as a pre-lab to allow students to devise a protocol to carry out a real titration in the laboratory, to explore theory (for example to test how much the difference between two pK values affects the precision with which the two values can be determined), or as a virtual investigation in which the task is to find the pKs which have been randomly assigned.

Acknowledgements

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