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A Systems Based Approach To The Teaching of Instrumental Chemical Analysis

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Introduction

Instruments are taking an increasingly important role in chemistry and other sciences. Modern instruments are designed to optimise performance. As a consequence the operation of them is highly automated and there is little indication of how they are working.

In addition the rapid developments in modern technology mean that design of instruments are constantly changing and new instruments are continuously evolving. This places a considerable burden on university science departments. The cost of instruments is enormous and this is amplified by the cost of replacing instruments in order to keep them up to date.

The increase in instrumentation has altered the skill requirements of industry. In the past there was an emphasis on wet chemistry and the use of quantitative apparatus. Industry now requires individuals that are competent with highly automated equipment and the production of very large numbers of results in short time spans.

Conversely and understanding of how an instrument works is necessary in order to optimise operation and to be aware of the quality of results that are produced.

Chemistry departments generally have large numbers of obsolete but still working instruments. These instruments operate on the same basic principles as modern instruments. However the technique that they use to process the results is cumbersome and slow.

Finally there is an hitherto unsatisfied demand to provide graduates with appropriate skills in information technology. This demand has appeared against a background where there is pressure to maximise the use of resources.

Outline of Project.

The project made use of low cost analogue-to-digital computer interface cards. These cards fit into an ISA slot of an IBM compatible computer. The cards chosen can process up to 8 analogue inputs and can read ± 5 volts with a precision of 1 in 65535 (16 bit). Software is available which allows the data to be processed by commonly used computer languages. In our project all programs are written using Visual Basic 3.

The philosophy of our approach was to use the output of the cards as a means of data processing. (Although it would be possible to write programs which did sophisticated processing of data from instruments and to simply produce results we feel that this approach would not contribute to the students' understanding of the basic processes.) Once the data was collected, students' processed the data using principally a spreadsheet package. Experiments were designed which illustrated basic principles of chemistry that a student had already dealt with. The emphasis was to demonstrate the student a means of gathering data and processing results in a manner that could not be done by manual means. This illustrates a number of important principles

Some results

It is not possible in a short article to cover all of the work that we have done. We have included some of the exercises that we consider the most successful.

The Visible Absorption Spectrum of Iodine Vapour

A Unicam Sp1800 Spectrophotometer, which is at least 20 years old was used to measure the spectrum. This instrument was chosen because its optical specifications are excellent. The spectrum of iodine vapour is very complex and has over 50 maxima. The position of these maxima can be used to determine the dissociation energy of the iodine molecule. However the measurement of the spectrum and determination of the maxima normally requires a very sophisticated and expensive instrument or many hours of tedious measurements.

In the experiment, the spectrophotometer was interfaced with a computer. Two channels of the computer interface card were used. 14,000 pairs of data points were recorded. As well as determining the dissociation energy of the iodine molecule, the experiment has been used to demonstrate the principles involved in numerical methods of noise reduction. The principles used in the technique of noise reduction form part of many courses in analytical chemistry. In the past these could only be taught theoretically. The use of this data has provided a valuable pedagogic tool.

Kinetics of the reaction of Chromium(III) with EDTA

Kinetics forms the basis of the study of any chemical reaction mechanisms, and forms a considerable part of physical chemistry syllabuses. There have always been problems in illustrating the principles of kinetics with practical exercises. The complex analytical procedures involved mean that students find the practical exercises confusing. Further the amount of analysis required can mean that meaningful practical exercises do not fit into a laboratory period.

We have developed an exercise whereby the formation of a chromium complex is monitored. Each kinetic run takes 25 minutes and 1500 data points are collected. Data is processed using the solver option in Excel. This approach would not be possible without the ability to collect and store data and the numerical processing. The experiment measured the change of the rate of reaction with temperature. Thus we have a new experiment which reinforces physical chemistry theory and integrates information technology in a completely transparent manner.

A redox titration

Commercial instruments are available which perform titrations automatically. However such instruments merely produce the results of the titrations and are not suitable to illustrate the principles behind the technique. The results of titrations can also be used to illustrate fundamental chemical principles.

We have developed such an automatic titrator. The liquid delivery system is a second hand automated syringe pump, purchased from a local hospital. The pump delivers titrant and a constant rate. The computer program stores the time, which is proportional to volume, and the electrode potential generated by the detection system.

Students are able to investigate the various means of determining end points. They can also use the data to determining the redox potentials of the system, and to compare the experimental results with titration curves generated from theoretical data.

Student feedback

Students enjoy doing the experiments. They particularly like the fact that all data is processed with a computer and little writing using old fashioned pens and paper is required. There is some indication of improved student understanding in important areas, however the exercise has not been running long enough, and parallel control groups have not been sufficiently alike to provide any conclusive results.

We do not feel that we have the balance of experiments correct yet. We are investigating the use of macros in Excel in order to cut down the amount of time that the students spend processing their data. However it is vital to ensure that the system does not become too automatic leaving the students with no idea of what they have done.

Applications to other areas.

We have been very impressed with the performance of the interface cards and how easy it has been to interface with existing instruments. It is clear that the basic system can be applied to practically any measurement problem. This means that the technique is applicable across all those disciplines of the university which rely on the input of quantitative data.