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Flow injection analysis (FIA) is a very suitable tool for use in the educational chemistry laboratory as a wide variety of techniques including titrations,[1] colorimetry[2] and fluorescence[3] can be illustrated in its use as an analytical method. The small volumes of reagent consumed make FIA suitable even when using expensive reagents. In this time of economic restraint, any procedures which reduce student costs but still maintain sufficient academic content should be appreciated by most educational establishments. The major obstacle to the widespread use of FIA in schools and colleges is the cost of commercial equipment. The present work describes the construction of low cost FIA equipment for spectrophotometry.

Experimental

The first part of the research concerned the reagent transport system. This originally consisted of a windscreen washer pump and the injector valve and tubes of 1 mm diameter (Omnifit Ltd). The Omnifit system was chosen because although each joint is more expensive than that of other manufacturer's there is no need for expensive flanging tools. The injection valve used was of the six-port rotary type with an external loop. Variation of the sample size was achieved by changing the loop size. Initially, a commercial flow cell (Starna Ltd.) was used.

It soon became apparent that windscreen washer pumps are not very suitable for flow injection analysis because, in spite of various modifications, the pumps overheated, which resulted in unacceptable variations in pump speed and hence flow-rate. These pumps were therefore replaced by peristaltic ones, originally of the 10 rev min^{-1} mains voltage type, which were later changed to 12 V adjustable voltage pumps with a maximum speed of 70 rev min^{-1}. These were found to give a much reduced pulsing effect for a given flow-rate. An uncoiled length (1 m) of thin walled silicone rubber tubing of 2-mm bore also reduced this effect.

With a simple one-reagent system and with the expensive commercial flow cell and spectrophotometer an acceptable calibration curve could be obtained. The next requirement was to construct a low-cost detector. A new style of flow cell was designed by using fibre optic light guides and stainless-steel tubes for flow introduction. The rest of the detector consisted of a light source (a light-emitting diode) and a photodiode based light detector.

The flow cell was designed using chemical kinetic considerations[4] and dispersion coefficient calculations.[5] Fig. 1 shows the straight light path of 10 mm and flow ducts set into a perspex block at a shallow angle to the light path. This shallow angle, together with the constant bore of the system from injector to

waste, minimised the dispersion. Several refinements of the original design were made, including variation of the angles of the flow tubes and light-guide ends. The original design had light guides with highly polished perpendicular ends, which caused a small eddy to be formed in the flow and small bubbles to be trapped at the end of the light guide. These bubbles distorted with the pulsing of the pump and caused a very poor signal to noise ratio. The trapping of the bubbles was removed by cutting the end of the light guide to the angle of the inlet tube. These new light guides had to be left unpolished in order to allow light to be scattered out of the end and not internally reflected. Both the light guides and the inlet tubes were carefully aligned to eliminate distortions in flow and set into position with silicone rubber cement.

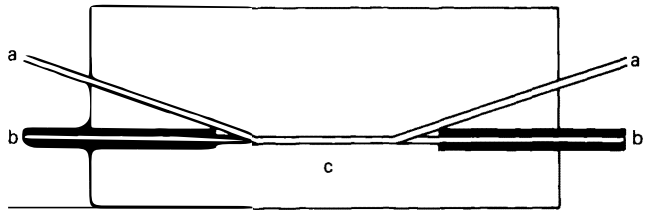

Fig. 1. New design of flow cell using fibre optics. a, Stainless steel tubes for flow inlet; b, fibre optic light guides; c, perspex block

The electronic part of the detector consisted of a photodiode and a simple amplifier. This system was built of a prefabricated printed circuit board, a single chip optoamplifier and four variable resistors. The light source was a single light emitting diode. All of these components were obtained from RS Components Ltd.

The detector components were housed in a light-tight metal box to eliminate stray capacitance effects. The PTFE tubes used to transport the reagents were shielded from the light for 25 cm from the detector to eliminate the transfer of light to the cell by these tubes. It was also beneficial, when using non-ionic reagents, to earth the flow between the pump and the injector. This was accomplished by passing the flow through a stainless-steel tube, which was earthed to the same point as the detector.

Programs were written for a BBC master microcomputer to control the pumps and monitor the output of the detector. Data treatment was by means of a home written program, together with a program made available to educational establishments by Manchester Polytechnic.[6] Using these together it was possible to display and evaluate peak heights and to produce hard copies of peak shape, peak height values and calibration curves.

Results and Discussion

Several series of experiments were performed to examine the performance of the detector for various coloured reagents. One of these series involved the investigation of the determination of iron(III) by three different reagents, ferron (8-hydoxy-7-iodoquinoline-5-sulphonic acid), variamine blue (C.I. 37240) and potassium thiocyanate. For each of these reagents experiments were performed to investigate the effects of variation of concentration and flow-rate of the particular reagent. Plots of detector outputs (mV) *versus* concentration were obtained under optimum conditions. These experiments were repeated for each of the three available colours of light-emitting diode and ferron was found to be the most suitable reagent. For each of the LED colours, the regression line (see Fig. 2) was calculated giving correlation coefficients of 0.997, 0.998 and 0.998 for yellow, green and red LEDs, respectively. A series of replicate injections of a given concentration of iron(III) gave peak heights with a relative standard deviation of 0.68%.

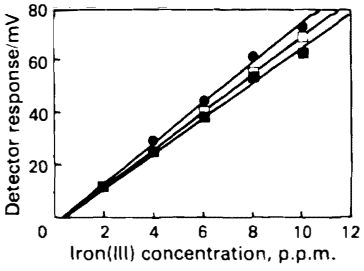

Fig. 2. Determination of iron(III) by ferron using various coloured light emitting diodes. □, Yellow; ●, green; ■, red

Other low cost detectors[7] have used phototransistors rather than photodiodes because of the improved sensitivity of the former. This effect has been partially offset by the use of a longer path-length flow cell. The construction of a small photodidode array with a white light source detector is currently being investigated. This should enable histogram spectra to be produced so that selection of analytical wavelengths can easily be made. Kinetic studies should also be possible.

Conclusion

For the expenditure of approximately £50 a detector can be constructed, the sensitivity and reproducibility of which are adequate for the requirements of a teaching laboratory.

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