A NEW AMPEROMETRIC DETECTOR FOR FLOW INJECTION ANALYSIS

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INTRODUCTION

Recent advances in flow analysis (FIA) detection systems are very important for good performance in the analysis. The reduction of the volume of the detector is desirable [1]. In the amperometric detection case, the construction of an adequate cell is not a trivial process, because three electrodes are usually requires [2]. Some systems are described in literature [3], but considerable work is necessary, besides requiring sophisticated apparatus. However, the flow of the sample does not remain the same as that in the tube entering into the cell, principally due to the dead volume of the cell [4]. The necessity for obtaining a smaller and more sensitive cell led us to develop an easy -to-build cell with a very small internal volume.

The cell described here in consists of a small platinum foil, a platinum wire, a Vycor Fritz pointer and polyester resin. The Cell has a volume of about 25 μL and does not present the dead volume problem. The flow into the cell is the same as that in the tube. The precision and sensitivity of the measurements was calculated by injecting standard solutions of H_2O_2.

MATERIAL

To construct such a cell we need essentially a small platinum foil, two pieces of platinum wire of adequate diameter, Vycor Fritz resin with a polyethylene tube and polyester resin. Figure 1 describes the cell.

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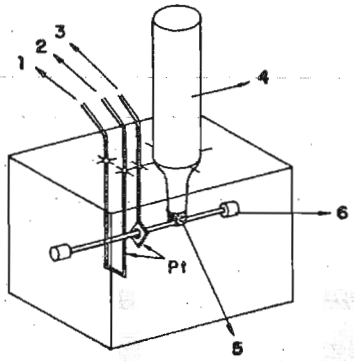

Fig.1 Cell design. 1. Ground, 2. working electrode, 3. counter electrode, 4. polyethylene tube for reference electrode, 5. Vycor Fritz pointer and 6. orifice with 1 mm internal diameter.

PROCEDURE

1. Construct an acrylic box of desirable size.
2. Solder the platinum foil to the platinum wire and it to the Ni-Cr wire.
3. Take a platinum wire of 1 cm in length and solder to its extremities pieces of Ni-Cr. wire.
4. Put the platinum wire and foil, and the Vycor Fritz pointer with polyethylene tube in the same line into the acrylic box.
5. Fill the box with polyester resin and let it dry, without bubble formation.
6. Make an orifice passing through the foil, wire and the Vycor Fritz pointer.
7. Polish the orifice with cotton string soaked with alumina emulsion, then wash with 0.5 mol dm^{-3} H_2SO_4 solution.
8. Fill the pointer tube with saturated KCl solution and put it in the reference electrode.
9. Test the flow of the cell with water stream. The water introduced to one extremity of the orifice must flow out only through the other.
10. The connections of the electrodes can be made with alligator clips.
11. When a thermostated bath is required, a small glycerine or water bath can be used.

CELL EVALUATION

The cell was evaluated injection 0.1 mol dm^{-3} KCl solution and performing the voltammetry experiment. The obtained voltammogram with the KCl solution containing 1.0×10^{-3} mol dm^{-3} hexacyanoferrate (II) was similar to those obtained with a normal cell.

Amperometry experiments shows that the cell has a high stability and sensitivity as can be seen in Fig.2 (curve A). The current dependence on hydrogen peroxide concentration is presented in curve B. As can be verified from the curve the system showed a linear range between 1.0×10^{-4} and 8.0×10^{-4} mol dm^{-3}. The reproducibility was excellent as can be observed from the results shown in curve C, indicating that this system can be used with a success in flow injection analysis. The systems was easy to wash, allowing the determination of about 60 samples per hour.

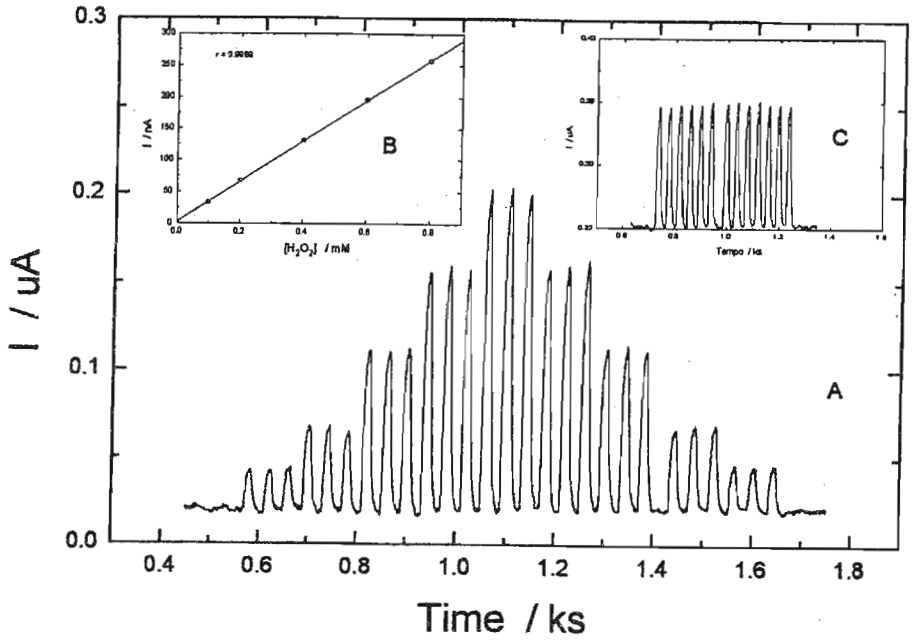

Fig.2 Fiagram obtained by injection of 25 μL of H_2O_2 solution in different concentrations(A), curve of the current dependence on concentration of H_2O_2 (B) and reproducibility of several injections of 25 μL of 3.0×10^{-4} mol dm^{-3} H_2O_2 solution (C). Applied potential 700 mV vs SCE.

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