Use of a Sintered Glass Crucible for Easy Construction of Liquid-Membrane Ion-Selective Electrodes

Theodore K. Christopoulos and Eleftherios P. Diamandis[1]
University of Athens, Laboratory of Analytical Chemistry, 104 Solonos Street, Athens 106 80, Greece

We are presenting a new means of preparing liquid-membrane ion-selective electrodes using silanized sintered glass crucibles as electrode bodies. These devices are suitable for educational purposes because they combine the following advantages: (a) The construction of the electrodes is fast and simple. (b) Only inexpensive sintered glass crucibles and standard laboratory equipment are needed. (c) The ion-selective electrode is open to visual inspection by the students who actually see the membrane and the inner reference electrode. This arrangement is very useful because it provides students a better understanding of exactly how ion-selective electrodes function.

The preparation procedure is schematically demonstrated in Figure 1. A G4 sintered glass crucible is cut as shown using the appropriate cutting tool. After cutting the whole glass ring under the glass filter, any sharp edges are smoothed using emery paper. The resulting crucible is shown in Figure 1(II). The following operations should be done in the fume hood. The crucible is washed under vacuum, with chromic acid, water, and finally with acetone, and it is allowed to dry for 3 min. The vacuum is released from the bottle, and 3 mL of trimethylchlorosilane is added to the crucible. After waiting for about 3 min, the vacuum is applied again and the silylating reagent is left to pass through the filter (Fig. 1(III)). The filter is washed with water (3 × 5 mL) and acetone (3 × 5 mL), and it is allowed to dry for another 3 min. A few drops of the liquid ion-exchanger are added to the porous hydrophobic glass (Fig. 1(IV)) so as to cover the whole surface of the filter. The exchanger is left for about 1 min to wet the sintered glass; any excess is removed with a pipet and finally with absorbent paper. The internal reference solution (10^{-2} M solution of the ion of interest) is added into the crucible up to a level of about 1–2 cm from the bottom. One of the reference electrodes is dipped into the internal reference solution, as shown in Figure 2, for the complete electrochemical cell.

The electrode is ready to use immediately after preparation. One crucible can be used for the construction of several liquid-membrane electrodes since the liquid ion-exchanger can be readily washed out with acetone and replaced by a new one in minutes.

We have used our approach, to construct ion-selective electrodes for Ca^{2+} and perchlorate (liquid ion-exchangers from Orion Research, Inc.) and propantheline (liquid ion-exchanger described by Mitsana-Papazoglou et al.[2]).

We have used the electrodes to construct calibration curves. The curves did not differ from those obtained with conventional dip-type electrodes. We have also applied the electrodes for the potentiometric titration of perchlorate with tetraphenylarsonium chloride, of calcium with EDTA, and of propantheline with sodium tethaphenylborate. In all cases, the titration curves were the same with those obtained with conventional dip-type electrodes.

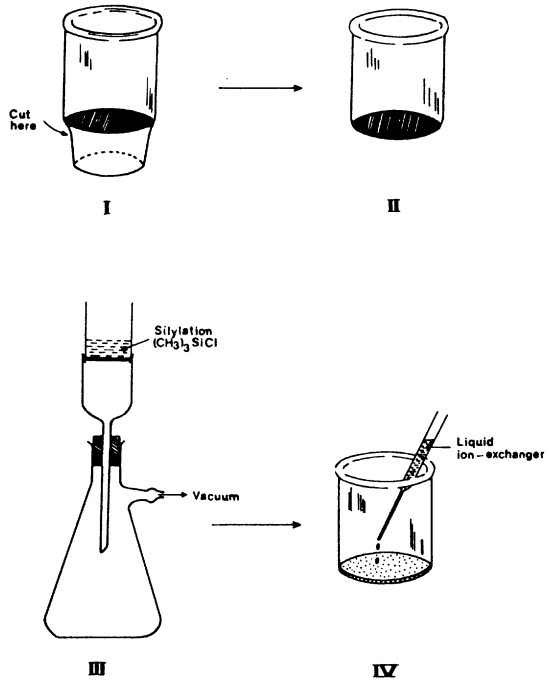

Figure 1. Procedure for the preparation of the electrode body by using a G4 sintered glass crucible. For more details see text.

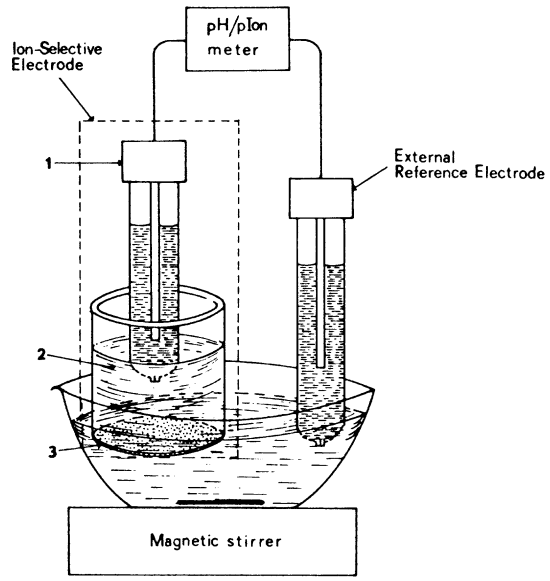

Figure 2. The complete electrochemical cell. (1) Internal reference electrode; (2) internal reference solution; (3) hydrophobic porous glass supporting the liquid ion-exchanger.

[1] Author to whom correspondence should be addressed; mailing address: Cyberfluor, Inc., 179 John Street, Suite 400, Toronto, ON, Canada M5T 1X4.

[2] Mitsana-Papazoglou, A.; Christopoulos, T. K.; Diamandis, E. P.; Hadjiioannou, T. P. *Analyst* **1985**, *110*, 1091.