



IE Series

Liquid Ion Exchangers

INSTRUCTION MANUAL

Serial No. _____

CAUTION: The toxicological properties of this LIX have not been fully determined. Ingestion or contact with the human body may be harmful. Exercise due care! LIXes should be stored in a cool place out of direct sunlight. For laboratory use only. Not for food, drug, household or other uses.

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Fig. 1–Liquid Ion Exchanger

WPI's Liquid Ion Exchangers (LIX), for use with the FD223 Electrometer, allow intracellular measurements to be made for cations (hydrogen, potassium and calcium) using the procedures described in this manual.

CATIONS

| ION | H+ | K+ | Ca++ |
|------------|-----------|-----------|-------------|
| ORDER NO. | IE010 | IE190 | IE200 |

SELECTIVITY COEFFICIENTS*

| | | | |
|-----------------|---------|--------|---------|
| Na+ | 12.7 | 1.97 | 5.5 |
| Mg++ | - | 2.95 | 4.9 |
| K+ | - | - | 5.4 |
| Ca++ | - | 2.7 | - |
| USEFUL pH RANGE | 2-10 | 4-10 | 4-10 |
| SLOPE | 56 mV | 58 mV | 28 mV |
| LINEAR RANGE | pH 4-12 | pK 0-3 | pCa 1-7 |
| REFERENCES | 12 | 3, 4 | 16 |

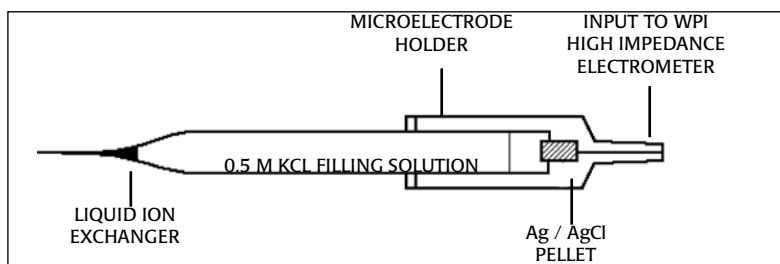
*Selectivity Coefficients are expressed here as $-\log K_{ij}$ or pK_{ij} .

FABRICATION OF ION-SELECTIVE MICROELECTRODES

Ion selective microelectrodes have gained wide use in biology as a means for examining intracellular ion activities (1, 3, 4, 9). The construction of electrodes with tip diameters of one micron or less using liquid ion exchanger resins is, in principle, straightforward and follows a few simple steps (2, 5, 6, 7). The general procedure is to:

1. Pull the microelectrode from a suitable glass capillary.
2. Apply a hydrophobic coating to the interior of the electrode tip.
3. Fill the tip with the ion exchanger.

In practice, the specific method employed will depend upon the individual requirements and laboratory conditions (e.g., the resin used, glass type, electrode shape and tip size, and ambient humidity). Variations in this procedure most often involve the method for producing the hydrophobic coating. The following note presents a general method for constructing ion selective microelectrodes using liquid ion exchangers and also illustrates variations in the method which may be relevant to specific applications.



Pulling the Microelectrode

Conventional methods are used to pull the micro-electrode. The method chosen is more dependent upon the use of the electrode than the fact that it is to be used to construct an ion selective electrode. Tip diameters can be one micron or less.

Electrodes can be pulled from a variety of capillary glasses. Pyrex glass with or without an internal fiber has been most often used. Multi-barrel glass can also be used, but caution must be taken to avoid coupling between barrels at the tip of the resulting microelectrode.

The most important consideration in this initial step is that the glass must be thoroughly cleaned before use. A recommended procedure is to soak the capillaries in a commercial glass cleaning solution (sulfuric and chromic acids) for several hours followed by thorough rinsing – first in distilled water, then in acetone, and finally drying in an oven.

Silanizing the Tip

Ion exchanger resin will not stick well to a hydrophilic clean glass surface. It is necessary, therefore, to make the inner surface of the electrode hydrophobic in order to stabilize the resin in the tip and to form a good seal with glass surface. This is a critical step and can significantly influence both the sensitivity and selectivity of the resulting electrode (8). The general procedure is to coat the electrode with a chloro- or alkoxy silane dissolved in an organic solvent, and to then heat the electrode to fix the hydrophobic layer to the glass surface. Too much silane will plug the tip, while too little will not hold the resin.

A recommended procedure uses dimethyldi-chlorosilane dissolved as a 5% solution in xylene (5). Immediately after pulling, the shaft of the electrode is exposed to this solution vapor for 1-2 minutes. The electrodes are then baked for one hour at 100 °C.

Because of the critical nature of this step no single method has proven best in all cases. Several other silanizing solutions and curing methods have been reported. A sampling includes: exposure to non-diluted dimethyldi-chlorosilane vapor (6); a 1% solution of Siliclad in 1-chloronaphthalene (7, 8); or a 1.25% solution of Dow Corning 1107 fluid in trichloroethylene (2). Baking times and temperatures are also variable, but do not seem to be critical for a successful electrode. While this list is not complete, it illustrates possible variations which may be used in an attempt for an improved electrode.

Filling the Tip

Following silanization, the ion selective membrane is formed by filling the tip of the electrode with a 200-500 micron length of ion exchanger. Several methods are again possible. A recommended method is to construct electrodes using glass capillaries containing an internal glass fiber (6). When a small drop of resin is placed in the electrode shank, it will rapidly migrate and fill the tip. A distinct advantage in this method is that it is rapid and requires only a small amount of the ion exchanger.

If capillaries without an internal fiber are used, the electrodes may also be filled from the inside by working the resin to the tip with a thin glass fiber inserted into the back of the electrode. Positive pressure applied to the electrode can also be used. Alternatively, electrodes may be filled from the front by dipping the tip in the ion exchanger until the required column is formed. The time required for filling will depend on the tip diameter. This method has the advantage that failure to fill indicates a plugged tip most often resulting from particles in the resin or from the silanizing procedure.

Whichever method is chosen for forming the ion selective membrane, the tip should be visually checked under the microscope to verify the completeness of filling and to confirm the absence of air bubbles in the exchanger column. The electrode can then be filled with a reference electrolyte (0.5 M KCl or NaCl are recommended for potassium and sodium exchangers) and stored with tip immersed in the identical solution. Although electrodes may be stored for extended periods, a loss of both selectivity and sensitivity usually occurs with time. It is recommended that fresh electrodes be made on the same day as usage if possible.

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