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August 1970

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FUMED SILICA SALT-BRIDGES

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August 1970

This investigation was prompted by our need for rugged, highly conducting salt-bridges of various geometries with low leakage which could be fabricated rapidly and at low cost. Although unfired Vycor tubes (1) have gained wide acceptance for use as salt-bridges in coulometry, they shatter if allowed to dry with a salt solution inside, and strongly adsorb dye material. Also, they have a considerable chemical memory which necessitates a long wash-out operation when one wishes to change the filling solution. These drawbacks, in addition to the fact that the tubes are quite expensive and must be custom fabricated, thus involving a long time delay, forced us to consider alternative materials. Ion exchange membranes (1) were briefly considered, however, they too were rejected owing to difficulties in making the bridges solution tight. These problems were overcome very simply and inexpensively by the use of a supporting electrolyte gel, prepared from fumed silica, contained in a tube having a fritted lower section.

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EXPERIMENTAL

Apparatus and Materials. An ESI, Model 250 conductance bridge was used, in conjunction with a conductivity cell fabricated by this laboratory's glass shop, to make conductivity measurements. The coarse quartz frit blank, having a diameter of 100 mm and a thickness of 7 mm, was obtained from the Amersil Quartz Division of Engelhard Industries. The fumed silica, having an average particle size of 70 Å and tradenamed Cab-O-Sil, Grade HS-5, by its manufacturer, the Cabot Corporation, was used in about a 7% concentration by weight to gel the reagent grade, 1.00 M KCl solution. All water used was distilled. The coulometry cell employed was similar to the one described by Harrar (2).

Frit Fabrication. The blank quartz frit was cut into strips 7 mm wide, employing a circular, water-cooled, carbide saw. These strips were then cut to 10 mm lengths and a 3 mm diameter bit was used to drill a hole in the center, 7 mm deep, in the long axis of the block. Molten parafin was poured into the holes and 3 mm glass canes were inserted, after cooling, the mounted blocks were turned against the side of the carbide saw blade. The 6 mm O.D. frit cans which resulted from this operation were warmed, the glass canes removed, and the cans were then butt sealed, with aid of a glass lathe, to 55 mm lengths of 6 mm O.D. quartz tubing. Coincidental to this operation, the parafin was removed by burning.

Salt-Bridge Preparation. The quartz frits are washed in aqua regia, rinsed several times in water, followed by an ethanol rinse and finally dried by aspirating air through them. The gelled 1.00 M KCl is injected into the dry frit to a depth of 2 cm using a polystyrene spitzer. The tube is then filled with 1.00 M KCl and is now ready for use.

It is essential that the gelled KCl be injected into a dry tube, otherwise, the gel will separate from the fritted section when the tube is immersed in the sample solution.

Leak Rate Determination. A 1.00 M KCl gel bridge was filled with 1.00 M KCl and taped up right in a small beaker containing sufficient 1.00 M KCl to cover the fritted section. This beaker was placed in a large glass desiccator, and to prevent evaporative losses, 1.00 M KCl was poured in, to a depth of 1 cm, then the desiccator was closed. After 30 hours, the solution necessary to restore the original volume was added from a microliter-buret. An average leak rate of 13.2 μl per hour was calculated.

RESULTS AND DISCUSSION

These salt-bridges have been in use for about one year and have met all our requirements. Fumed silica gels can be formed with a wide variety of electrolytes, the only limitation being that the pH must not exceed 8, if the gel is to be stable. A salt-bridge can be converted from one supporting electrolyte to another within minutes and no harm is done to the frit if the silica gel is allowed to dry. The leak rate also is quite acceptable.

The addition of fumed silica to a supporting electrolyte does not significantly affect its conductivity. The resistance of 1.00 M KCl in our conductivity cell was 4.14 ohms. The resistance increased insignificantly (4.16 ohms) after gelling the KCl with 7% fumed silica. The resistance between the mercury pool and the analyte salt-bridge in the coulometry cell, commonly used in this laboratory, was 100.0 ohms--again employing a 1.00 M KCl solution and gel. Currents as high as 100 mA have been passed through the cell without deleterious heating effects.

The authors wish to thank Mr. William A. Jeunger of the Cabot Corporation, Burlingame, California office, for providing the samples of Cab-0-Sil fumed silica.

LITERATURE CITED

* Work performed under the auspices of the U. S. Atomic Energy Commission.

- (1) W. N. Carson, C. E. Michelson, K. Koyama, Anal. Chem. 27, 472 (1955).
- (2) J. E. Harrar, U. S. Atomic Energy Commission Report UCLRL-50335 (1967).

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