CF_2Cl_2 in one hour at temperatures up to 500° or between CH_3F and HF up to 400° in one hour. Higher temperatures could not be used because of chemical decomposition or reaction with the wall. No radioactivity was ever observed on the reactor wall, confirming the absence of exchange between HF and NiF_2 reported by Rogers and Katz.[3] Our results emphasize the remarkable stability of the carbon–fluorine bond.

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[3] M. T. Rogers and J. J. Katz, THIS JOURNAL, **74**, 1375 (1952).

[4] Operated by Carbide & Carbon Chemicals Corp. under Contract W-7405 for the U. S. Atomic Energy Commission.

Concentration Profiles in Packed-bed Ion-exchange Systems

BY E. B. BYRNE AND L. LAPIDUS

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In recent years, considerable theoretical effort has been expended to describe the behavior of fixed bed percolation columns. Implicit in all of this work was the assumption of a flat velocity or concentration profile as the solute moves down the bed. Experimental verification or rejection of this assumption has not been considered in any detail. Baddour, *et al.*,[1] and Glueckauf and Coates[2] have briefly mentioned the problem in their ion-exchange studies, while Stewart[3] has shown that adsorption on alumina oxide may produce considerable spreading of the concentration band at any column cross-section. Klinkenberg[4] has also mentioned data collected indicating large variations in fluid velocity in a number of percolation processes.

In this note, the present writers would like to describe briefly a series of experiments in which concentration profiles between acidic and basic forms of an anion-exchange resin, Amberlite IRA-400, were observed.

The main piece of equipment was 10 inches long, 1 inch ID stainless steel column packed with exchange resin to a depth of 8 inches. A 1 inch layer of $^1/_2$ mm. glass beads was placed on top of the resin to act as flow distributor, and glass wall plugs were placed at both ends to retain the beads and the resin. The column was constructed of two milled, semicylindrical sections, held together with two pressure clamps. By removing the clamps, the column could be split into two halves. Means were provided for feeding solution at a constant rate to the top of the column as well as for regeneration of the resin and backwashing of the bed proper. Various acids and bases were used both as regenerants and as solutes. These included acetic acid, sulfuric acid and sodium hydroxide. After the concentration profile had progressed to the desired point in the column, the solution was stopped, and 35 ml. of phenolphthalein solution added. The pressure clamps were removed; and the column, including the resin. was slit in a longitudinal direction. As a result, a cross-section of the entire column was available for observation with the acidic and basic portions being colored either a bright red or a neutral color.

The results indicate two distinct types of concentration profiles. The first, occurring when an acid solution percolated through the resin in the basic form, was characterized by a flat profile. This profile maintained its shape throughout the length of the bed. The second, occurring when a basic solution flowed through the acid form resin, resembled sections of an inverted parabaloid or a frustrum of a parabaloid. In the second case, the concentrated wave at the wall was as much as 4 inches ahead of the center section. These two radically different profiles are intimately connected with the swelling and shrinking of the resin as the exchange process takes place. The uniform band occurs when the resin expands during reaction and the non-uniform band when shrinkage occurs. Expansion of the resin tends to flatten the concentration profiles since, as the flow in a localized area advances ahead of the main flow, expansion of the particles reduces the size of interstices between the particles and increases resistance to flow. Shrinkage, on the other hand, tends to accelerate the flow in areas where local flow has advanced ahead of the main flow. This was particularly true near the walls of the column where the resin mass tended to shrink away from the walls, thus producing channels where the flow could advance farther and farther ahead of the main flow. Because of this accelerated flow near the walls of the column, it is possible for basic solution to pass through the entire column almost unreacted, even though the full exchange capacity of the column has not been used.

Thus, the assumption of a flat concentration profile in an ion exchange column would seem to be valid only if the exchange reaction is such that swelling of the resin takes place.

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[1] R. F. Baddour, D. J. Goldstein and P. Epstein, *Ind. Eng. Chem.*, **46**, 2192 (1954).

[2] E. Glueckauf and J. I. Coates, *J. Chem. Soc.*, 1315 (1947).

[3] A. Stewart, *Disc. Faraday Soc.*, **7**, 65 (1949).

[4] A. Klinkenberg, *ibid.*, **7**, 151 (1949).

Equilibrium Constants for the Formation of Complexes between 1,3-Diamino-2-propanol and Various Metal Ions[1]

BY ELY GONICK, W. CONARD FERNELIUS AND BODIE E. DOUGLAS

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Compounds in which 1,3-diamino-2-propanol is present in the form of complexes with metal ions have been isolated.[2,3] However, no data are available to permit comparison of the stability of these complexes with those containing no hydroxyl group. Such data are presented here.

Experimental

1,3-Diamino-2-propanol as obtained from Eastman Kodak Company was refluxed over barium oxide for four hours and distilled at 5 mm. The fraction boiling at 114° melted 40–42° and was found to possess the theoretical equivalent weight on titration.

Details of the experimental procedure are the same as those previously described.[4]

The terms used here and the method of calculation of con-

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[1] Abstracted from a portion of a thesis presented by Ely Gonick in partial fulfillment of the requirements for the degree of Doctor of Philosophy, August, 1951.

[2] F. G. Mann, *J. Chem. Soc.*, 2904 (1927).

[3] J. G. Breckenridge and J. W. R. Hodgins, *Can. J. Research*, **17**, 331 (1939).

[4] E. Gonick, W. C. Fernelius and B. E. Douglas, THIS JOURNAL, **76**, 4671 (1954).