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Electrical Conductance Cell Assembly for Use with Aqueous Solutions up to 800°C and 4000 Bars*

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An electrical conductance cell assembly, designed for use with aqueous solutions at extremes of temperature and pressure, is described. Its utility was demonstrated by measurements of the conductance of 0.010 molal KCl solution at 800°C and at pressures up to 4000 bars.

INTRODUCTION

AQUEOUS solutions at high temperatures, especially under supercritical conditions, have received considerable renewed interest in the past several years. A general review of investigations in this field has been published elsewhere.¹ The interest originates in part from geochemical problems,² from the efforts to synthesize crystals hydrothermally,³ and from the long range consideration of homogeneous nuclear reactors.⁴ Information about the formation and mobility of ions in such solutions is still very limited. Determination of electrolytic conductance can furnish such information.

Electrical conductance data on a large number of aqueous solutions were obtained by A. A. Noyes and co-workers,⁵ using cells capable of operating at temperatures up to 306°C and at pressures up to about 100 bars. Some investigators have determined solubilities and conductances of solutes in nonaqueous solvent such as sulfur dioxide and methanol at moderately high temperatures.^{6,7} Fogo, Copeland, and Benson⁸ constructed an all platinum cell and made very accurate conductance measurements of NaCl in water at pressures up to 300 bars and at temperatures between 378 and 393°C, i.e., slightly above 374°C, the critical temperature of water. More recently, Corwin, Bayless, and Owen⁹ have determined the conductance

of NaCl in H₂O at 390°C and at densities up to 0.50 g/cm³.

At temperatures several hundred degrees above the critical temperature, in order to reach solvent densities approaching those of the liquid state at low temperature, the conductance cell must be operated at pressures up to several thousand bars. Such a cell has to be made from a material of very high tensile strength at high temperatures. Walls and insulation must resist the corrosive action of supercritical solutions. One type of cell to meet these requirements has been used and was described previously.¹⁰ Based on the experience with this earlier cell, an improved conductance cell has been designed and used for preliminary measurements. It is described below.

DESIGN

The main problem in constructing a high temperature, high pressure conductance cell is to find an electrode insulation which withstands the combined action of temperature, pressure, and corrosion. In this cell, as well as in the previous model, one electrode is the wall of the cylindrical cell and the other one is a small platinum cylinder at the end of a platinum rod. This rod is insulated by a tube of sintered, pure and nonporous aluminum oxide.¹¹ The insulated electrode rod leads out of the cell proper through a thick-walled, metal tube into a cold closure where plastic can be used for insulation and packing. Thus, there are no concentrated mechanical forces acting on the brittle insulation material in the high temperature region. The construction of the cell and details are shown in Fig. 1.

The main body (1), machined from a forged billet of a nickel-based alloy, Udimet 700 (0.1% C, 15% Cr, 19% Co, 5% Mo, 3.5% Ti, 4.5% Al, 1% Fe, 0.03% B, and remainder Ni),¹² has a cylindrical hole honed to a mirror-like finish to receive the similarly machined lining tube of 75% platinum-25% iridium alloy¹³ with a 0.040-in. wall. Flanges on the ends of the tube were machine spun over the internal shoulder within the hole to retain the lining in position. Two screwed caps (2) of Udimet alloy force the tungsten

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¹² Utica Drop Forge and Tool Division of Kelsey-Hayes Company, Utica, New York.

¹³ J. Bishop & Company, Malvern, Pennsylvania.