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A Solid Electrolyte Oxide Ion Electrode For Molten Nitrates

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A SOLID ELECTROLYTE OXIDE ION ELECTRODE FOR MOLTEN NITRATES

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ABSTRACT

An oxide ion sensitive electrode of the type Pb, $PbO/ZrO_2(Y_2O_3)//$ was constructed and its performance tested in the binary, equimolar molten salt $NaNO_3$-KNO_3 over the temperature range 336-350°C. The response of this electrode to oxide ion concentrations over the range 10^{-6}-10^{-10} moles/kg is linearly dependent upon log $[O^=]$, and $dE/dlog[O^=]$ corresponds to a two-electron process.

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Introduction

Oxide ion conducting ceramics such as stabilized zirconia have assumed increased scientific interest and technical significance in recent years as solid electrolytes. Indicator electrodes employing zirconia stabilized with CaO, MgO, or Y_2O_3 have been used in the metallurgical industry to measure oxygen activity in gases and molten metals. They have also been used in laboratory research to determine the dissociation pressure of solid oxides, the solubility of oxygen in liquid metals, and the oxide ion activity in molten salts and glasses.

The introduction of 10-20 at. percent of the oxides of lower valent cations such as Ca^{+2} or Y^{+3}, stabilizes the cubic fluorite structure of ZrO_2 and leads to the formation of anion vacancies in order to maintain charge neutrality. Oxide ion conduction then occurs through this one-dimensional structure[1].

Stabilized zirconia can function as a semi-permeable membrane in molten salts, and is able to transport oxide ions into and out of these liquids[2]. The reversibility of the process

$$0^= (ZrO_2) \rightleftharpoons 0^= (\ell) \tag{1}$$

has been demonstrated under potentiometric conditions, i.e. when very small currents are passed as, for example, when stabilized zirconia forms part of an indicator electrode[1,2]. Electrodes employing stabilized zirconia, and which are sensitive to the concentration of oxide ions, have been described[3,4].

The oxide ion sensitive electrode used in this study is one where the oxygen partial pressure is fixed by a metal-metal oxide couple. The electrode may be represented as

$$Pb, PbO/ZrO_2 (Y_2O_3)// \tag{a}$$

where the double line represents the zirconia membrane that separates the electrode from the bulk melt. In this case the electrode reaction is

$$2e^- + 1/2\ O_2 \rightleftharpoons 0^= \tag{2}$$

$$E = E^0 - \frac{RT}{2F} \ln \frac{a_{O^=}}{P_{O_2}} \qquad (3)$$

Since the oxygen partial pressure is fixed by the Pb, PbO equilibrium the constant terms can be incorporated into E_0 and equation 3 reduces to

$$E = E^0 - \frac{RT}{2F} \ln a_{O^=} \qquad (4)$$

As a consequence of the gradient in oxide ion activity across the zirconia membrane a potential is developed which is given by

$$E = \frac{-RT}{2F} \ln \frac{(a_{O^=})_1}{(a_{O^=})_2} \qquad (5)$$

where 1 and 2 correspond to the inside and outside of the zirconia membrane, respectively. It is assumed in equation (5) that the zirconia membrane functions in a manner analogous to a liquid junction and that the transport number of the oxide ion equals one.[5,8] Therefore, the potential of electrode (a), with respect to the reference electrode, is given by

$$E = E^0 - \frac{RT}{2F} \ln (a_{O^=})_1 - \frac{RT}{2F} \ln \frac{(a_{O^=})_1}{(a_{O^=})_2} \qquad (6)$$

Since the oxide ion activity inside the zirconia membrane is fixed, these terms may be incorporated with $E°$ to give

$$E = E^{0'} + \frac{RT}{2F} \ln (a_{O=})_2 \qquad (7)$$

Thus, the potential of electrode (a) can be directly related to the activity of the oxide ion in the $NaNO_3 - KNO_3$ melt.

While the electrode potential is a measure of the activity of the oxide ion, equation (7), it is not possible to associate a particular cell emf with a specific oxide ion activity unless an independently determined value of $E°$, the standard electrode potential, as well as phase boundary potentials are available. At the present state of knowledge in molten salts these data are generally not available. However, in this study we are primarily concerned with the response of the Pb, PbO/ZrO_2 electrode to changes in the oxide ion concentration rather than its absolute value. Thus we shall, as a first approximation, assume that the Pb, PbO/ZrO_2 electrode potential is a measure of the oxide ion concentration.

Metal-metal oxide electrodes like (a) have been used quite successfully in both research and industrial applications. Generally, the most frequently used metal-metal oxide couples are Cu-Cu_2O, Fe-FeO, Ni-NiO, or Pb-PbO (other couples are also possible)[4]. For our experiments we chose the Pb-PbO couple because this particular couple has been investigated extensively. The electrode process is unambiguous and reversible and the mutual solubility of the

reproducibility of the electrode at low temperature (350°C) should be improved by the presence of a liquid phase (Pb, m.p. 327°C).

In order to evaluate the response of the Pb, PbO/ZrO$_2$ electrode it was necessary to vary the oxide ion concentration of the NaNO$_3$-KNO$_3$ melt in a systematic manner. Typically, this is done by adding weighed amounts of Na$_2$O. However, this approach was not taken for several reasons: Na$_2$O is very difficult to obtain commercially in high purity (typically it is 95-97 percent pure), even Na$_2$O synthesized in the laboratory under carefully controlled conditions contains several percent Na$_2$O$_2$ [7]; Na$_2$O is hydroscopic and difficult to maintain unreacted in other than inert atmospheres. Evidence has recently appeared indicating that even in the absence of molecular oxygen oxide ions may react with molten alkali nitrates (at temperatures below 350°C) to form peroxide and superoxide ions[5,8,9]

$$O^= + NO_3^- \rightleftharpoons O_2^= + NO_2^- \tag{8}$$

$$O_2^= + 2NO_3^- \rightleftharpoons 2O_2^- + 2NO_2^- \tag{9}$$

Therefore, the approach taken here was to establish a known oxide ion concentration by means of a Lux-Flood acid-base reaction

$$A + O^= \rightleftharpoons B \tag{10}$$

where A and B are the conjugate acid-base pair, e.g.

$$CO_2 + O^= \rightleftharpoons CO_3^= \tag{11}$$

$$SiO_2 + O^= \rightleftharpoons SiO_3^= \tag{12}$$

In the Lux-Flood acid-base concept an acid is an oxide ion acceptor and a base is an oxide ion donor[10,11]. In these experiments the oxide ion concentration was determined by the chromate-dichromate equilibrium

$$Cr_2O_7^= + O^= \rightleftharpoons 2 CrO_4^= \tag{13}$$

That is,

$$[O^=] = \frac{[CrO_4^=]^2}{[Cr_2O_7^=]K} \tag{14}$$

where K is the equilibrium constant for reaction (13) in the NaNO$_3$-KNO$_3$

melt. Thus, the oxide ion concentration is uniquely determined by the ratio of chromate to dichromate, equation (14). Strictly speaking, eqn. (14) should be written in terms of the activities of its components. However, the equilibrium constants reported by Kust[12] for reaction (13) were calculated from concentrations. It is entirely self-consistent therefore, to use eqn. (14) to calculate oxide ion concentrations. It is only necessary to assume that the activity coefficients are independent of concentration which seems reasonable in solutions this dilute ($<10^{-1}$ mole/kg). Reaction (13) has been well characterized in $NaNO_3$-KNO_3 and there appear to be no side reactions of any consequence below 350°C[12].

Experimental Procedure

The cell used to measure changes in oxide ion concentration of the $NaNO_3$-KNO_3 may be represented as

$$Pb, PbO/ZrO_2 (Y_2O_3)//NaNO_3\text{-}KNO_3/AgNO_3 (0.1 \text{ molal}), NaNO_3\text{-}KNO_3, Ag \qquad (b)$$
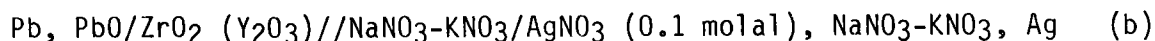
where the double line represents the zirconia membrane. The Ag, $AgNO_3$ reference electrode, used to monitor the potential of the Pb, PbO electrode, was contained in a thin Pyrex membrane. Because it exhibits ideal Nernst behavior and the metal has no observable tendency to dissolve in molten silver and is highly reversible to its ions, the Ag, $AgNO_3$ electrode has been used extensively in molten nitrates as a reference electrode.[13,14] The Pb, PbO electrode was contained in a closed end stabilized zirconia tube (Corning Glass Works, Solon, Ohio) one-half inch in diameter and 12 inches long. Three different compositions were tried: ZrO_2-8 wt. percent Y_2O_3, and ZrO_2-3 wt. percent MgO and ThO_2-15 wt. percent Y_2O_3. No difference in electrochemical behavior was detected. All these compositions exhibited excellent thermal shock resistance and appeared visually to be unattacked by immersion in the nitrate melt at 350°C for periods of up to 14 days.

The Pb, PbO electrode was made by mixing together approximately equal volumes of 99.9% pure Pb shavings and the tetragonal form of PbO, litharge. Connection to the Pb, PbO electrode was made by a 304 stainless steel rod. Visually the rod appeared to be unattacked by the Pb, PbO mixture and the constancy and reproducibility of the EMF measurements seemed to confirm this. The stainless steel rod was held in place by an O-ring compression seal that also served to maintain atmospheric integrity.

Figure 1 is an illustration of the experimental apparatus and the Pb, PbO electrode. In order to avoid any reaction between the nitrate melt and the container that might affect the results, the melts were held in a gold crucible. The chromel-alumel thermocouple used to monitor the melt temperature was sheathed in gold.
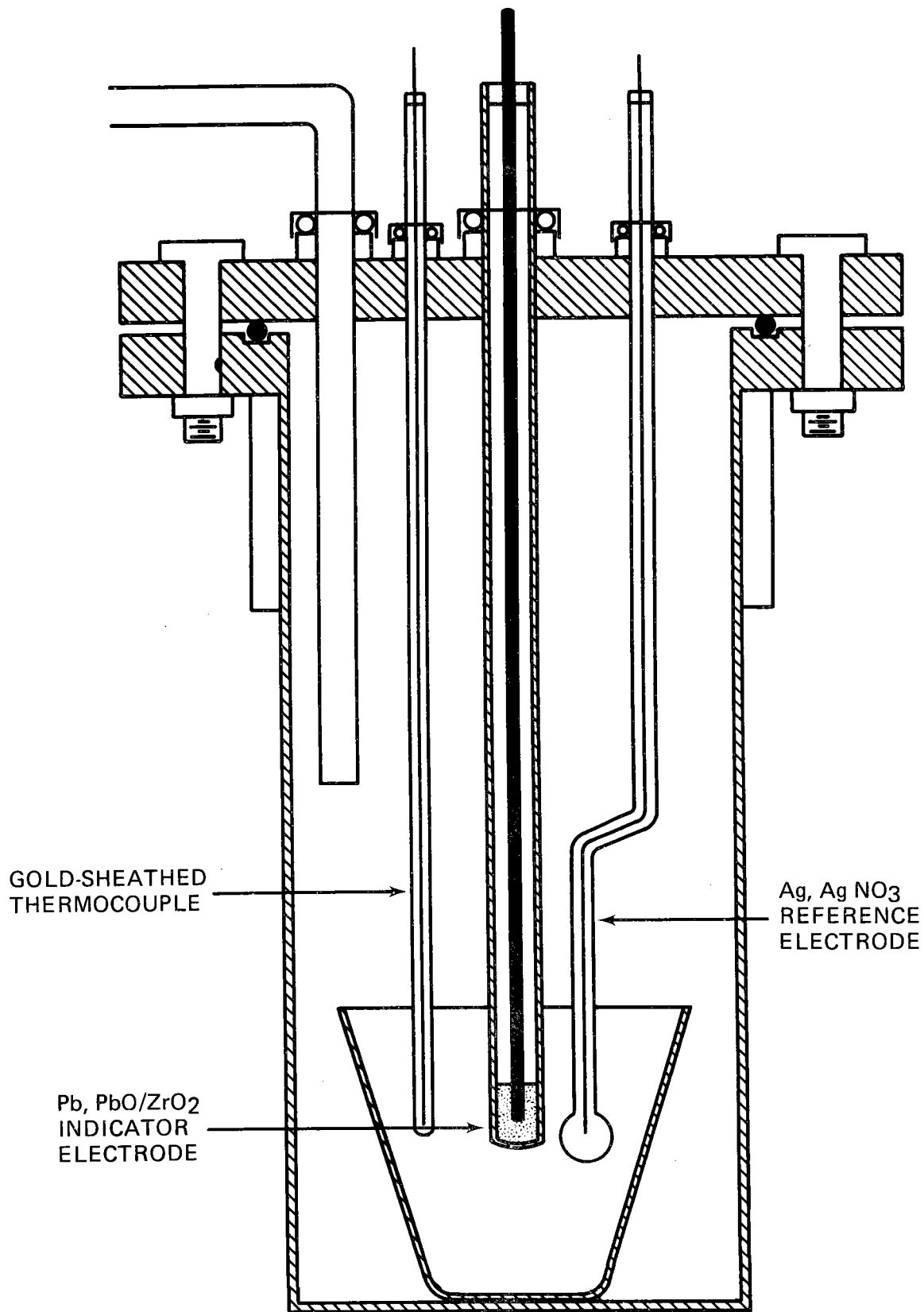
GOLD-SHEATHED
THERMOCOUPLE

Ag, Ag NO_3
REFERENCE
ELECTRODE

Pb, PbO/ZrO_2
INDICATOR
ELECTRODE

Figure 1. Experimental Cell and Reference Electrode

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Argon was used in this study to provide an inert, controlled atmosphere over the melt. It was carefully purified by passage through an 800°C titanium bed to remove oxygen and water and then through an Ascarite column to remove any carbon dioxide. To prevent back-diffusion of atmospheric gases, the effluent gas was also passed through a sulfuric acid bubbler. In addition to careful purification of the gases, all the salts used in this program were routinely purified by recrystallization from distilled water. Following the recrystallization operation the salts were stored in a vacuum oven at 150°C.

The potential between the Pb, PbO indicator electrode and the Ag, $AgNO_3$ reference electrode was measured with a high impedance electrometer (Keithley Model 616). The output from the electrometer was recorded on a Hewlett-Packard Model 7100B strip chart recorder.

In order to establish a particular oxide ion concentration the appropriate weighed quantities of vacuum-dried K_2CrO_4 and $K_2Cr_2O_7$ were added to the melt and argon was bubbled through the melt to help establish a uniform concentration. Changes in the oxide ion concentration could be made by adding weighed amounts of either K_2CrO_4 or $K_2Cr_2O_7$ depending upon the change desired. The solutions were kept relatively dilute with the $CrO_4^=$ and $Cr_2O_7^=$ concentrations varying between $5 \times 10^{-1} - 3 \times 10^{-5}$ moles/kg.

Except for the lowest oxide ion concentration ($< 10^{-12}$ moles/kg) the response of the Pb, PbO/ZrO_2 electrode to changes in the oxide ion concentration was very rapid. Once the equilibrium potential had been reached ($\sim$ 5-10 min) it remained very stable ($\pm$ 0.0005V) for as long as 48 hours. At the lower oxide ion concentrations about an hour was required for the potential to stabilize.

Results and Discussion

The results of 60 experiments in which the response of the Pb, PbO/ZrO_2 electrode to variations in the oxide ion concentration of a $NaNO_3$-KNO_3 melt, as determined by the $CrO_4^=/Cr_2O_7^=$ ratio, are shown in Figure 2. Also included are the relevant oxide ion concentrations calculated from equation (14). To aid in interpretation of the data presented in this Figure all the potential values for a given concentration have been averaged. The spread is $\pm$ 0.005V.

Over the oxide ion concentration range 10^{-10} to 10^{-6} moles/kg the Pb, PbO/ZrO_2 electrode potential is a linear function of the logarithm of the oxide ion concentration with a slope equal 2.3 RT/2F. As the oxide ion concentration falls below 10^{-10} moles/kg the indicator electrode potential begins to show a pronounced departure from this relationship. At oxide ion concentrations below 10^{-12} moles/kg the Pb, PbO/ZrO_2 electrode potential appears to be invariant with concentration. While lines of differing slope may be drawn through the data shown in Figure 2 to include more of the data points, the precision of the measurements is such that this treatment of the data is unjustified.
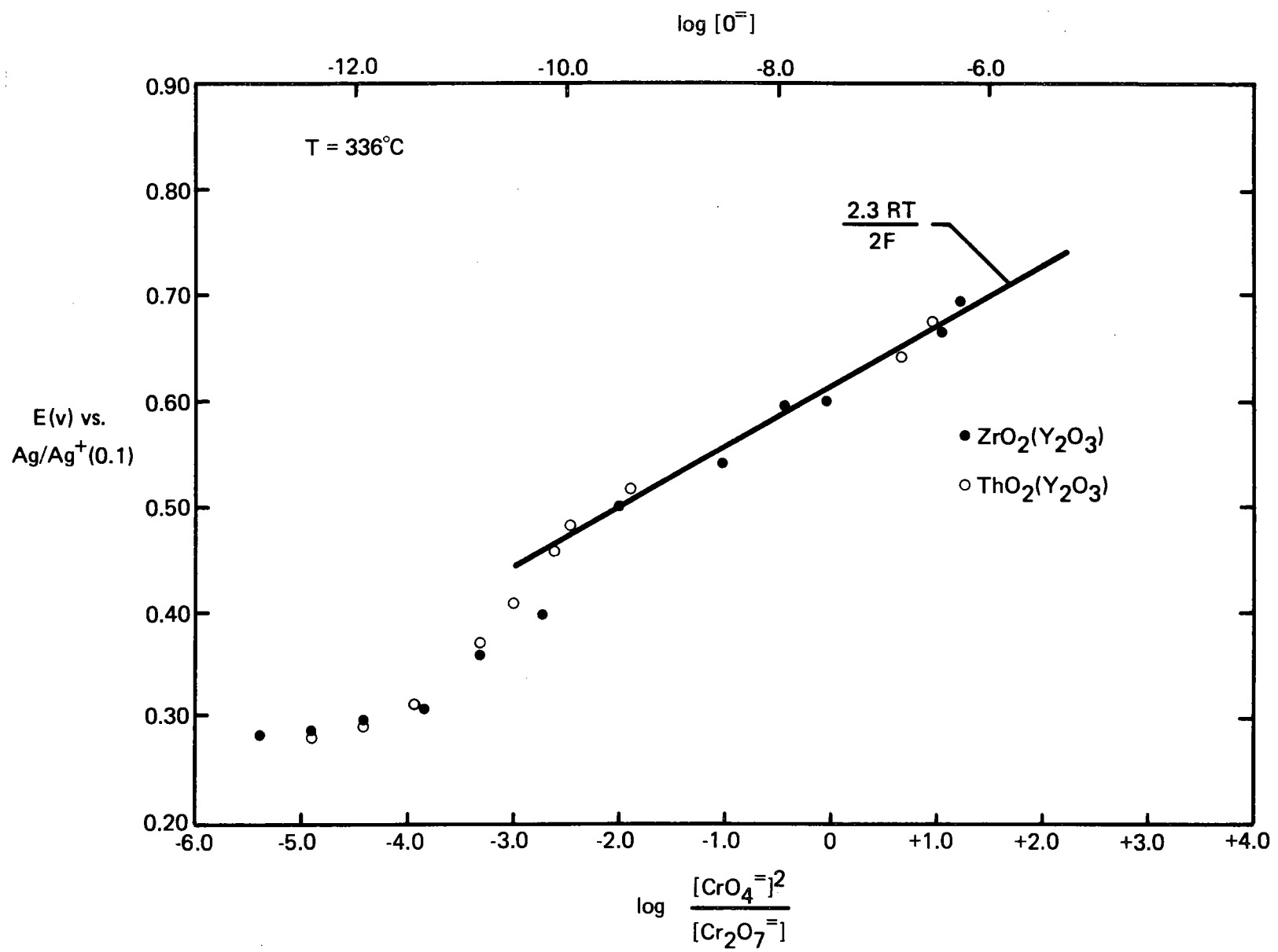
Figure 2. Pb, PbO Indicator Electrode Potential vs.log $\frac{(CrO_4^=)^2}{(Cr_2O_7^=)}$

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If equation (2) represents the potential determining reaction that takes place at the Pb, PbO/ZrO$_2$ electrode a slope of 2.3 RT/2F would be expected. The question then is, why does the concentration dependence of the Pb, PbO electrode potential change when the oxide ion concentration falls below 10^{-10} moles/kg? Since the dichromate concentration at this oxide ion concentration is ~10^{-3} moles/kg significant changes in its activity coefficient are considered unlikely. It is possible that at very low oxide ion concentrations (<10^{-10} moles/kg) reactions which cause a decrease in the oxide ion concentrations and whose products are undetectable by the Pb, PbO/ZrO$_2$ electrode, begin to become significant. As a consequence, the oxide ion concentration is actually lower than would be predicted from equation (7). As the nominal oxide ion concentration falls below 10^{-12} moles/kg essentially all of it is converted to a species undetectable by the Pb,PbO/ZrO$_2$ electrode and consequently the electrode potential becomes invariant with changes in the nominal oxide ion concentration. Reactions such as

$$O^= + 1/2O_2 \rightleftharpoons O_2^= \tag{15}$$

$$O^= + 3/2O_2 \rightleftharpoons 2O_2^- \tag{16}$$

in which the oxide ion reacts with small amounts of oxygen in the melt may be responsible. These reactions are particularly credible in light of Stern's observation that zirconia-based electrodes are apparently only responsive to changes in the oxide ion concentration.[5,15] The activity coefficient of the oxide ion in this melt, while unknown, appears to be reasonably constant over the concentration range 10^{-10} - 10^{-6} mole/kg because of the linear variation of potential with concentration.

As a measure of the response of the Pb, PbO/ZrO$_2$ electrode a series of potentiometric acid-base titrations were performed. The reaction that was followed was

$$Cr_2O_7^= + CO_3^= \rightleftharpoons 2CrO_4^= + CO_2 \tag{17}$$

which is, in effect, a titration of the Lux-Flood acid Cr$_2$O$_7^=$ by the base CO$_3^=$. This reaction was first studied by Shams el Din who showed it to be uncomplicated[16]. The presence of chromate in the melt appears to have no effect on the titration[17]. In these experiments weighed quantities of vacuum-dried reagent grade Na$_2$CO$_3$ were added to various ratios of Cr$_2$O$_7^=$/CrO$_4^=$ in NaNO$_3$-KNO$_3$ at temperatures between 335-350°C. The response of the Pb, PbO/ZrO$_2$ electrode for a typical titration at 336°C is shown in Figure 3. The equivalence point is quite sharp and corresponds to a change in potential of about 500 mV. The results of three different titration at 336 ± 2°C are given in Table I.

The agreement between the concentration of Cr$_2$O$_7^=$ in the melt and that determined analytically is reasonably good. These experiments indicate that even at temperatures below 350°C the Pb, PbO/ZrO$_2$ electrode responds rapidly and reproducibly to changes in the oxide ion concentration and that the charge-transfer is reversible.
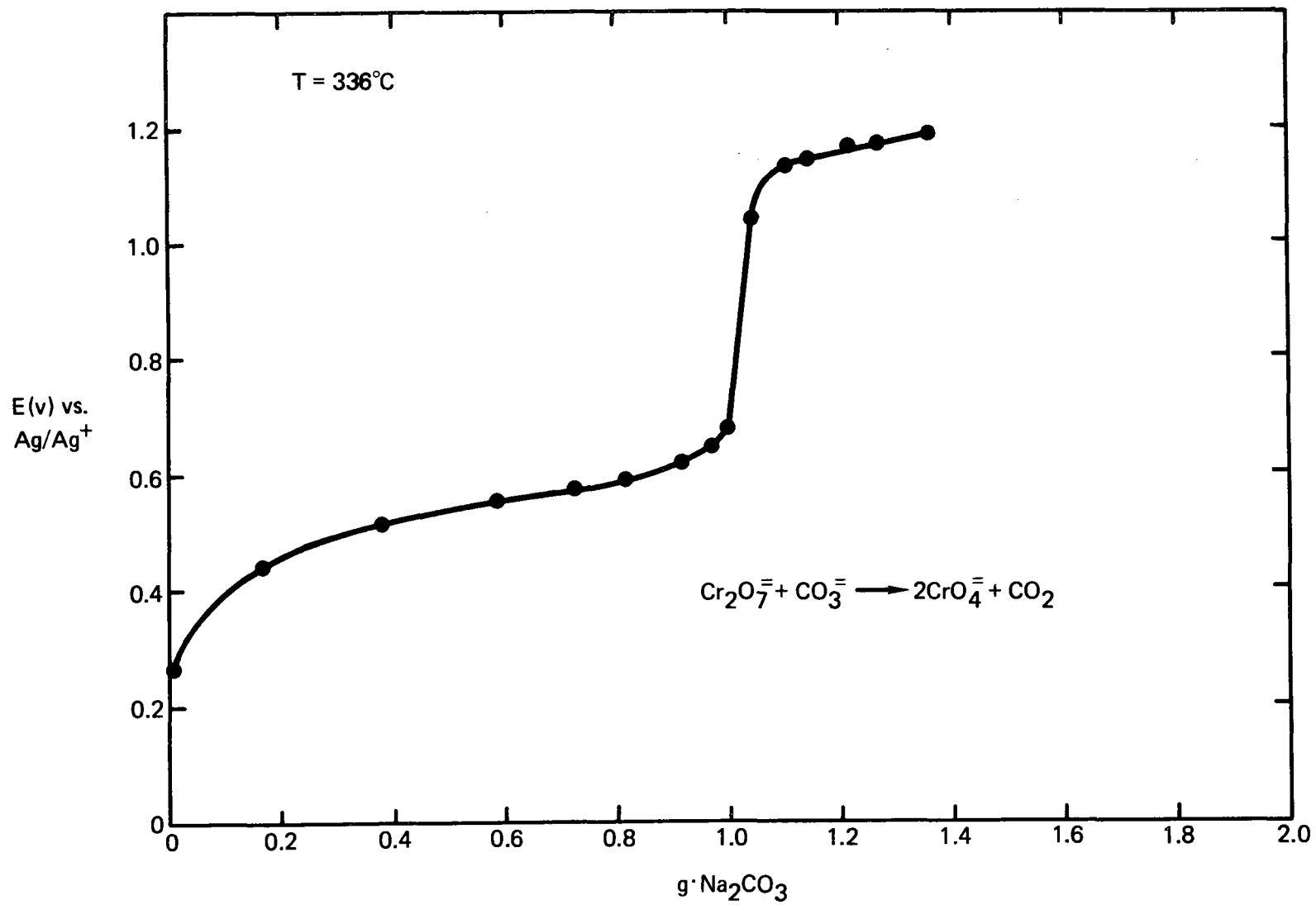
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Figure 3. Titration Curve of Dichromate With Carbonate

TABLE I

TITRATION OF DICHROMATE WITH CARBONATE IN $NaNO_3$-KNO_3

T = 336 $\pm$ 2°C

Exp't.	$[Cr_2O_7^=]$ added (moles)	$[Cr_2O_7^=]$ found (moles)	Percent Recovered
DN-1	9.91 x 10^{-3}	9.86 x 10^{-3}	99.5
DN-2	1.02 x 10^{-3}	1.00 x 10^{-3}	98
DN-3	1.04 x 10^{-2}	1.07 x 10^{-2}	103

Conclusion

The electrode Pb, PbO/ZrO_2 (Y_2O_3) has been demonstrated to be sensitive to oxide ion concentrations over the range 10^{-10} to 10^{-6} moles/kg in molten $NaNO_3$-KNO_3 between 336 and 350°C. Over this concentration range the concentration dependence of the electrode potential is linear with a slope corresponding to a two-electron process. The response of the Pb, PbO/ZrO_2 electrode to changes in the oxide ion concentration is very rapid, and once the equilibrium potential has been reached (~5-10 min) it remains very stable ($\pm$ 0.0005 V) for as long as 48 hours.

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