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EVALUATION OF AN ELECTROCHEMICAL
OXYGEN METER

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

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Gene H. Palmer

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OXYGEN METER

By

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ABSTRACT

Two electrochemical oxygen meter probes have been operated in the Systems Quality Sodium Loop. Both electrodes were responsive to oxygen injections; however, due both to the interaction of stainless steel with oxygen and to the imprecision of analysis for oxygen below about 5 ppm in sodium, only qualitative results were obtained.

The emf of the first electrode appeared to have a long term downward drift. The second electrode has been quite stable during five months of operation. The temperature coefficient of the second electrode is approximately zero. The apparent temperature coefficient of 0.5 mv/°F is a result of a shift in the equilibrium of the stainless steel-oxygen-sodium system.

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I. INTRODUCTION

As the technology of sodium as a coolant for nuclear power reactors has advanced, one of the areas which has received considerable study is instrumentation for monitoring impurities in circulating sodium systems.

One of the impurities of major importance is oxygen. The earliest in-line device for measuring relative amounts of oxygen was the plugging meter. More recently, the resistivity meter and the electrochemical oxygen meter have been developed. A recent Los Alamos report describes the comparison of the resistivity meter, the electrochemical meter, and vacuum distillation as methods for determining oxygen in sodium.⁽¹⁾ The authors concluded that the resistivity meter is of limited value for determining oxygen in sodium while the electrochemical meter is capable of measuring oxygen to $\pm 10\%$ in the 1 to 30 ppm range. They state that vacuum distillation should be capable of measuring oxygen to ± 0.5 ppm.

The sodium supply loop⁽²⁾ was built as a part of the Liquid Metal Fast Breeder Reactor Cladding Program (LMFBR). The principal purpose of this loop is to supply sodium to the mechanical properties tests.⁽³⁾ Because this loop operates at essentially constant conditions, it was believed that testing of an electrochemical oxygen meter in this facility would provide useful steady-condition operating data. Therefore, two electrodes manufactured by United Nuclear Corporation (UNC) have been operated in the supply loop during the past year. The results of the investigation are presented in this report.

II. THEORETICAL

The oxygen meter is composed of a ceramic electrolyte tube prepared from a slip-cast mixture of $\text{ThO}_2\text{-Y}_2\text{O}_3$. Inside the electrolyte tube is a mixture of $\text{Cu}_2\text{O-Cu}$ which serves as one half-cell. The appropriate electrical connections are brought out of the end of the tube through seals. The tube is inserted in sodium so that the other half-cell is $\text{Na}_2\text{O-Na}$. The two half-cell reactions are:

and

and the net cell reaction is,

The ideal emf of this cell is:

$$E = E_o - \frac{RT}{nf} \ln \frac{(A_{\text{Cu}})^2 (A_{\text{Na}_2\text{O}})}{(A_{\text{Na}})^2 (A_{\text{Cu}_2\text{O}})} \quad \dots(4)$$

where $E_o = \Delta F^o / nf$, and ΔF^o is the standard free energy change for reaction (3), R is the gas constant, T is the temperature in $^{\circ}\text{K}$, n is the number of electrons involved in the electrode reaction, f is the Faraday constant, and the A -values are the activities of the indicated species. In the oxygen meter cell, all components except oxygen, or Na_2O , are at unit activity, hence $(A_{\text{Na}_2\text{O}})$ is the only variable term in the equilibrium constant. Because the quantity $(A_{\text{Na}_2\text{O}})$ is proportional to (A_{O}) , Equation 4 can be rewritten,

$$E = E_o - \frac{RT}{nf} \ln (k A_{\text{O}}) = E_o - \frac{RT}{nf} \ln (k \gamma C_{\text{O}}) \quad \dots(5)$$

At saturation,

$$(kA_{O=})_o = 1 = k\gamma(C_{O=})_o \quad \dots(6)$$

so that,

$$k\gamma = \frac{1}{(C_{O=})_o} \quad \dots(7)$$

Therefore,

$$E = E_o - \frac{RT}{nf} \ln \frac{(C_{O=})}{(C_{O=})_o} \quad \dots(8)$$

where $(C_{O=})$ is the observed concentration and $(C_{O=})_o$ is the saturation concentration of oxygen at T.

The supply loop normally operates at 500°F, but the oxygen meter operates at $515 \pm 5^\circ\text{F}$ so the theoretical emf vs oxygen concentrations have been calculated at 500°F. Free energy values taken from Bureau of Mines Bulletin 605⁽⁴⁾ give a ΔF° (500°F) of -50,680 cal. Therefore, E_o is + 1.098 volts.

The most reasonable solubility data for oxygen in sodium appears to be that obtained at Los Alamos by vacuum distillation. The method was developed by Bergstresser and co-workers,⁽⁵⁾ and was utilized by Rutkauskas^(6,6a) to obtain additional values for the solubility of oxygen in sodium. Equation 9 has been fitted to the LASL data,

$$\log (C_{O=})_o = a - \frac{b}{T} = 6.79 - \frac{2740}{T} \quad \dots(9)$$

Using the value of E_o calculated above and Equation 9 for $(C_{O=})_o$, the final expression for the emf of the cell at 500°F as a function of oxygen concentration is,

$$E = 1.185 - 0.0528 \log (C_{O=}) \quad \dots(10)$$

A plot of this equation is given in Figure 1. Figure 1 also shows an estimated 500°F calibration curve based on the curves for 550, 600, and 650°F. The equation for the 500°F curve is,

$$E = 1.160 - 0.025 \log (C_{O=}) \quad \dots(11)$$

Figure 1. Theoretical emf of Na₂O-Cu₂O Cell at 500°F

The reason for the large difference in the coefficient of $\log (C_{O=})$ in Equations 10 and 11 is not understood.

The equation for the temperature coefficient of the cell can be developed by differentiating Equation 5 at constant activity of Na₂O,

$$\left(\frac{\partial E}{\partial T}\right)_A = \left(\frac{\partial E_o}{\partial T}\right)_A - \frac{R}{nf} \ln (A_{Na_2O}) - \frac{RT}{nf} \left(\frac{\partial \ln (A_{Na_2O})}{\partial T}\right)_A \quad \dots(12)$$

The last term is zero because of the restriction of constant activity. Substituting $(C_{O=})/(C_{O=})$ for (γ_{Na_2O}) , as before, gives,

$$\left(\frac{\partial E}{\partial T}\right)_A = \left(\frac{\partial E_o}{\partial T}\right)_A - \frac{2.303R}{nf} \left[\log (C_{O=}) - \left(a - \frac{b}{T}\right) \right] \quad \dots(13)$$

where a and b have the values of Equation 9.

An alternate development is to substitute for $\ln (C_{O=})$ in Equation 8 giving,

$$E = E_o - \frac{2.303RT}{nf} \left[\log (C_{O=}) - \left(a - \frac{b}{T}\right) \right] \quad \dots(14)$$

Again, taking the partial derivatives at constant activity,

$$\left(\frac{\partial E}{\partial T}\right)_A = \left(\frac{\partial E_o}{\partial T}\right)_A - \frac{2.303R}{nf} \left[\log (C_{O=}) - a \right] - \frac{2.303RT}{nf} \left[\frac{\partial \log (C_{O=})}{\partial T} \right]_A \quad \dots(15)$$

In this case, the last term is not zero because of the temperature dependence of the solubility. Taking the derivative of the temperature dependence,

$$\frac{\partial \left(a - \frac{b}{T}\right)}{\partial T} = + \frac{b}{T^2} \quad \dots(16)$$

Substituting this result in Equation 15 gives Equation 13, so that each development gives the same result.

7-016-251-3

Figure 2. Oxygen Meter Results

III. EXPERIMENTAL RESULTS

A. OXYGEN ADDITIONS

The original oxygen meter installation was made in January 1967. The supply loop was shut down and an existing "ell" was removed for mounting of the electrode "tee" housing. After the housing was welded in place, the electrode was installed according to the manufacturer's recommended procedure.⁽⁷⁾ The loop was returned to normal operation.

The principal purpose of the loop is to furnish constant purity sodium for other users. However, during periods during which sodium dispensing was not required, it was possible to add oxygen to the loop. Three such experiments were conducted during the six-months operation of the first electrode. The response of this electrode over its period of service is shown in Figure 2. All voltages recorded have been corrected to 500°F by using a temperature coefficient of 0.5 mv per °F to correct for ambient temperature fluctuations. Also shown is the oxygen content of the loop as determined by the mercury amalgamation procedure.

The initial operation (until February 8) represents stabilization of the electrode, and perhaps of the loop surfaces as well. The output of the electrode was fairly constant from February 8 until February 23, when the first addition of oxygen was carried out. The loop operated at a nominal temperature of 500°F with the cold trap operating at 260°F and the hot trap operating at 1200°F.

The first experiment was carried out from February 23 to March 2. A bulb was attached to a sample station and pressurized with an amount of oxygen calculated to add 6 ppm to the loop sodium. The cold and hot traps were valved out of the loop, circulation was established through the sample station, and oxygen was admitted by briefly cracking the valve on the bulb. Some oxygen was introduced by this technique, but the tubing from the valve to the flowing sodium plugged with oxide before the bulb was empty. The loop was sampled one day after the addition, but the chemical analysis failed to show any increase in oxygen. The loop is normally cold-trapped at 260°F, which is equivalent to an equilibrium solubility of ~ 1 ppm,⁽⁶⁾ and not all the planned 6 ppm was injected into the sodium. Therefore, it is not too surprising that no change was noted.

The 28 mv decrease in emf is equivalent to C_2/C_1 of 3, so if the loop were operated at ~ 1 ppm, the increment of added oxygen would be equivalent to 2 ppm.

At the completion of this experiment, the cold trap was returned to service. It can be noted that there was a rapid rise in emf followed by a slow increase over several days.

The second experiment was carried out from March 21 to April 14. In this experiment, oxygen was added in the form of sodium peroxide which was placed in a bulb and attached to a sample station so that sodium circulated through the bulb. Two incremental additions of peroxide were made, each calculated to increase the oxygen level in the sodium by 10 ppm.

An immediate drop in emf of 20 mv resulted, as shown in Figure 2. No sharp decrease was obtained at the second addition, only a gradual decrease. It would seem that the reaction of molten sodium with sodium peroxide should be rapid. The flow rate through the sample station is approximately 0.5 gpm and, assuming the sodium is saturated with oxygen as it leaves the sample station, the peroxide should have reacted and dissolved in 30 minutes.

The oxygen levels analytically determined during this experiment were slightly higher than before the addition. The decrease in emf is equivalent to a factor of 2-3 increase in oxygen concentration over the course of the experiment.

The third experiment was carried out from May 4 to May 11. Initially, an amount of oxygen calculated to add 25 ppm to the sodium was added through the cover gas on the surge tank. Indications were that the oxygen was rapidly absorbed by the sodium. By the following morning, the emf had decreased 48 mv. A sample was taken which analyzed 15 ppm oxygen. The 48 mv decrease in emf is equivalent to a factor of 6.7 increase in oxygen.

The emf held constant for 4 days at which time the emf increased 18 mv. At this time, a second oxygen addition equivalent to 20 ppm was made. The emf decreased slightly below the earlier level. Another sodium sample was taken which again analyzed 15 ppm oxygen. The next day, the emf had increased 12 mv. Unfortunately, the loop had to be cold-trapped for sodium dispensing, so the experiment was terminated.

In July of 1967, the loop was shut down for replacement of the first electrode with one which was precalibrated by the manufacturer. The ceramic tube on the electrode broke during the removal operation so that the broken ceramic and some sodium had to be dug out of the housing. This proved to be a tedious undertaking but was finally satisfactorily completed. The calibrated electrode was inserted and the loop returned to operation. No voltage output was obtained! The loop was again shut down and the electrode removed. The tip of the ceramic electrolyte tube had broken, rendering the electrode inoperative. According to the UNC trouble-shooting guide, an indication of a short circuit should have shown on the readout instrument. This was not observed.

A section of each of the broken electrolyte tubes was sectioned and examined metallographically in an effort to determine the cause of breakage. Macro-photographs and photomicrographs of the broken tubes are shown in Figures 3 and 4. Examination of the electrode used 6 months indicates there was some interaction with the sodium, perhaps with impurities in the ceramic. The consensus of the ceramists who examined the broken electrodes was that mechanical stress was the most likely cause of failure. It should be observed, however, that thoria-base ceramics are known to be susceptible to failure by thermal shock.

A second calibrated electrode was installed and was operative upon startup. The operational results for this electrode are shown in Figure 5. It can be seen that the output of this electrode was quite stable after the initial "wearing in" period was completed. The reason for the day to day fluctuation in emf is not known. In the operation of the first electrode, it was observed that ambient temperature in the building could cause a 10-mv change in output. An air thermostat was installed around the second electrode so that a constant external temperature is maintained.

On December 5, oxygen equivalent to 25 ppm was added through the cover gas on the surge tank. As can be seen in Figure 5, a decrease in emf of 42 mv resulted. Chemical analysis of a sample taken December 6 did not show an increase in oxygen content, but a plugging temperature of 325°F was found. Normally, the plugging meter does not show plugging above the freezing point of sodium. The 42 mv decrease in emf is equivalent to an increase in oxygen of a factor of six.

a. Broken Electrode

b. Polished Section

7706-5502

Figure 3. Broken Oxygen Meter Electrode Used Six Months

a. Broken Electrode

b. Polished Section

7706-5501

Figure 4. Calibrated Oxygen Meter Electrode Which Broke During Installation

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Figure 5. Results on Second Electrode

B. TEMPERATURE COEFFICIENT STUDIES

Earlier work has indicated that the oxygen meter was quite sensitive to temperature fluctuations. To further evaluate this effect, a series of runs was performed in which the loop temperature and the oxygen meter electrode temperature were varied. The loop was operated for several days at each temperature in order to insure that all readings were stabilized.

The results of the experiment are shown in Table 1. The four temperatures listed are as follows: "T" is at the tee which is part of the electrode housing, "Fins" is near the top of the finned part of the electrode housing, "Pump" is about five feet upstream from the electrode and "Tank" is about five feet downstream from the electrode on the return pipe to the surge tank.

TABLE 1
OXYGEN METER READING vs TEMPERATURES
AROUND THE LOOP

Run No.	Temperature (°F)				emf (v)
	"T"	Tank	Pump	Fins	
1	520	497	490	169	1.117
2	571	530	524	169	1.134
3	551	522	520	169	1.132
4	575	530	521	169	1.135
5	575	569	568	169	1.155
6	552	550	547	169	1.146
7	527	521	520	170	1.131
8	514	505	498	170	1.119

These data are arranged in chronological order. The first and eighth runs represent normal operating conditions, i. e., a nominal loop temperature of 500°F. The temperature at "T" normally runs a few degrees higher due to its proximity to the hot trap.

The data fall into two groups. Runs 5, 6, and 7 are at essentially isothermal conditions while 2, 3, 4, and 7 are approximately constant temperature loop conditions and variable probe temperature.

The data from Runs 5, 6, and 7 give an apparent temperature coefficient of approximately $0.5 \text{ mv}/^{\circ}\text{F}$, which is the same as previously reported by Minushkin.⁽¹⁰⁾ However, when the data from Runs 2, 3, 4, and 7 are examined, it can be seen that the temperature of the probe has practically no effect on the emf.

According to Equation 13, and using free energy values from Wicks and Block,⁽⁴⁾ the temperature coefficients of the cell at a cold trap temperature of 260°F should be $-0.14 \text{ mv}/^{\circ}\text{F}$, and decreases to $-0.225 \text{ mv}/^{\circ}\text{F}$ at saturation (unit activity) of sodium oxide in sodium. [This is $(\partial E_o / \partial T)_A$ in Equation 13.] In contrast, McPheeters and Williams⁽¹⁾ derived an expression for the temperature coefficient of the cell which gives a positive temperature coefficient. However, they omitted the last term in Equation 15, $(\partial \log C_o = / \partial T)_A$, saying, in effect, that this term is zero. Theoretically, the temperature coefficient of the cell at unit activity of sodium oxide in sodium must be $(\partial E_o / \partial T)_A$.

IV. DISCUSSION

As was seen in Figures 2 and 5, the meter is indeed responsive to changes in oxygen content. However, the expected oxygen level was not obtained in any of the oxygen addition experiments, so quantitative data were not obtainable in these experiments. The probable explanation for the results is as follows. Initially, the loop was operated for several months with both the cold and hot traps in service. The oxide scale was removed from the stainless steel surfaces by reaction with sodium and was deposited in the cold trap. An equilibrium was reached in the oxygen-stainless steel-sodium system which represented well below 5 ppm oxygen in the circulating sodium. When oxygen was added to the loop, the stainless steel immediately gettered part of the oxygen so that the added oxygen was partitioned between the sodium and the steel. Because the partitioning factor is not constant from test to test, and depends to a great extent on kinetic factors, quantitative data cannot be determined in this manner. An example of this is seen in the third experiment with the first electrode; the oxygen concentration behavior of the system is quite interesting. Four days after the initial oxygen injection, it appeared that oxygen was being removed from the sodium. Then, the second oxygen addition again caused the expected decrease in emf, but the emf began to rise again almost immediately. It is therefore concluded that, at the higher oxygen concentration, the rate of gettering by the stainless steel was markedly increased, and the oxygen was rapidly gettered.

Minushkin and Goldman,⁽¹¹⁾ in their work on calibration of oxygen meter electrodes, also noted shifts in emf which they attributed to gettering of oxygen from the potassium by stainless steel.

Another illustration of the gettering phenomenon can be obtained by examining the meter readings during cold-trapping of the added oxygen. At the end of the first and third experiments shown in Figure 2, and of the experiment in Figure 5, there was an initial sharp rise in emf followed by a slower increase. The initial sharp rise represents removal by the cold trap of the available oxygen in the sodium, followed by a period during which the previously gettered oxygen on the stainless steel is slowly released to the sodium, and then is

deposited in the cold-trap. Finally, as can be seen in Figure 5, the previous equilibrium level of oxygen in the sodium (and the equilibrium amount of oxygen on the stainless steel surfaces) is reached.

In the temperature coefficient experiments, it was observed that the temperature of the electrode alone had little or no effect on the emf, while the loop temperature changes yielded an apparent temperature coefficient of +0.5 mv/°F. From these results, it appears that the actual temperature coefficient of this particular oxygen meter electrode is approximately zero. Therefore, the observed changes are a result of temperature-dependent changes in the stainless steel-oxygen-sodium equilibrium. Using the data of Wicks and Block,⁽⁴⁾ the standard free energy for the reaction,

changes from -7.6 kcal at 500°F to -5.8 kcal at 575°F. This changes the equilibrium constant for Reaction 17 by a factor of approximately 8. McPheeters and Williams⁽¹⁾ were also working in a stainless steel system, so their temperature coefficient results may be suspect.

There appears to have been a downward drift in the emf output of the first electrode. After the "steady state" condition was reached during cold-trapping, the downward drift was quite evident following the first and third experiments. Some of the decrease in emf during the second experiment may actually have been caused by the drift. A plausible, but unproven, explanation for this behavior is that there appeared to have been some reaction between the electrode and the sodium. This reaction could have caused the observed downward drift in emf because, if the reaction increased the conductivity of the oxide electrode material, sufficient current could flow to polarize the electrode and decrease its emf output.

V. CONCLUSIONS

The electrochemical oxygen meter appears to be a useful device for monitoring sodium systems. However, there are several perturbations which complicate the use of the instrument for precise determinations. Three of these are: (1) in a stainless steel system, the interaction of the steel with oxygen is an all-important factor; (2) it appears that the long-term stabilities of different electrodes are different; and (3) the activity coefficients of oxygen in sodium are not known, so that quantitative interpretations cannot readily be made.

The temperature coefficient of the emf of the presently installed electrode itself is approximately zero. There is, however, an apparent temperature coefficient which is believed to be due to the temperature dependence of the equilibrium in the stainless steel-oxygen-sodium system.

Obviously, additional studies are needed, and are in progress. Therefore, it can be concluded that the utility of the electrochemical oxygen meter will increase in the future.

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