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ARGENTIC OXYSALT ELECTRODES

by Harold F. Leibecki

Lewis Research Center
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SUMMARY

Silver ions with plus three oxidation states in complex silver compounds have been investigated as cathode electrodes in 45 weight percent potassium hydroxide (KOH) electrolytes with zinc as the anodes. The compounds studied were $2Ag(AgO_2)_2 \cdot AgNO_3$, $2Ag(AgO_2)_2 \cdot Ag_2SO_4$, and $2Ag(AgO_2)_2 \cdot AgF \cdot 1\frac{1}{2}\ H_2O$. The instability of $2Ag(AgO_2)_2 \cdot AgNO_3$ in 45 percent KOH was studied.

Temperature coefficient of open circuit potentials, current density as a function of cell voltage and coulombic efficiencies of the cathode at various discharge rates were obtained for $2Ag(AgO_2)_2 \cdot AgNO_3$ and the product obtained from the stability tests of $2Ag(AgO_2)_2 \cdot AgNO_3$ in 45 percent KOH. Changes of enthalpy, free energy, and entropy for the cell reaction for these two compounds were calculated from experimental data and compared with reported values for silver oxides AgO and Ag_2O.

It was found that a cell with a higher energy density is obtainable from the argentic oxysalts than the commercially available AgO cells and that reserve-type cells could be developed using these compounds.

The reaction product of $2Ag(AgO_2)_2 \cdot AgNO_3$ and 45 percent KOH was found to be a complex compound containing divalent silver atoms. The electrochemical reduction of this compound proceeds without the large loss of terminal voltage that is associated with argentic oxynitrate and AgO while they are being discharged. It also discharges without the characteristic voltage step associated with AgO.

INTRODUCTION

The preparation and chemical properties of the argentic oxysalts $2Ag(AgO_2)_2 \cdot AgNO_3$, $2Ag(AgO_2)_2 \cdot AgSO_4$, $2Ag(AgO_2)_2 \cdot AgF \cdot HF \cdot 1\frac{1}{2}\ H_2O$, and $2Ag_3O_4 \cdot AgClO_4$ have been of interest for many years. The interest in these compounds have received a rejuvenation within the last few years (refs. 1 to 7). The larger volume of work presented in the journals deals with the physical properties of the compounds, and little is known regarding the use of argentic oxysalts as electrode materials (refs. 8 to 13). If these compounds can be used as cathodes, their higher valence state, which should produce a higher half cell potential, and increased coulombic content would produce a cathode with a higher energy density than the presently used silver oxide (AgO).

These materials were studied in 45 percent potassium hydroxide (KOH). It will be shown that the compounds can be incorporated as cathodes in silver-zinc reserve-type cells with an improvement in the energy density of the presently used AgO cells.

If argentic oxynitrate was allowed to soak in 45 percent KOH, it would slowly decompose with the evolution of oxygen. A study of this material will also be incorporated in this report.

EXPERIMENTAL

Preparation

Argentic oxysalts were prepared by electrolyzing a 10 percent neutral solution of silver nitrate, silver fluoride, silver perchlorate, or silver sulfate at a current density of 10 milliamperes per square centimeter. Argentic oxysulfate was also prepared chemically as reported by de Boer and van Ormondt (ref. 7).

Figure 1 shows the electrolysis cell in which the argentic oxysalts were formed. The anode and cathode compartments were separated by a strong base ion exchange membrane (Gelmar Instrument Co.). The acid formed in the reaction was neutralized by intermittently adding a potassium hydroxide solution to the cathode compartment. The membrane allowed transport of hydroxyl ions to the anode compartment, while preventing contamination of the anode compartment by precipitated silver oxide (Ag_2O). The inert electrode for the anodic reaction was platinum, and that for the cathode was 304 stainless steel. The anode with the argentic oxysalts was removed from the electrolysis cell, washed with distilled water and then acetone, and dried in an oven at 60° C for 2 hours. It was found that there is no thermal decomposition below 100° C. These salts were then stored in a stoppered bottle.

A chemically reduced form of $2Ag(AgO_2)_2 \cdot AgNO_3$ was formed by allowing the argentic oxynitrate to soak in 45 percent KOH solution for 24 hours. Silver oxide (AgO) was prepared as stated by J. H. de Boer and J. Van Ormondt (ref. 7).

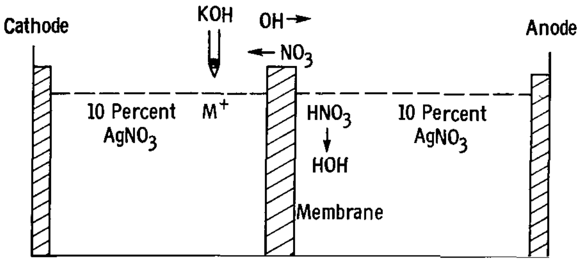


Figure 1. - Electrolysis cell for preparation of argentic oxysalts.

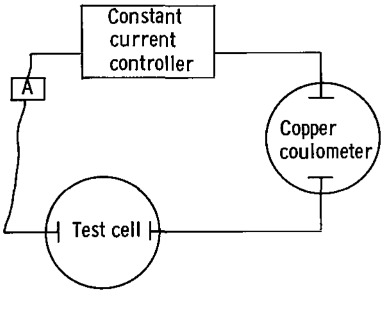


Figure 2. - Constant current discharge assembly.

Beaker Cells

Electrode preparation. - The dried argentic oxysalts were compressed at 3600 pounds per square inch into disks of either 0.635 or 1.128 inch in diameter. These disks were then weighed and cemented to a platinum support plate. A conductive silver cement was used to hold the disk to the platinum. After the cement had dried, the disk and support were painted with a vinyl paint, leaving only the front surface of the argentic oxysalt free to react.

Electrode discharge. - The argentic oxysalt cathodes were discharged in 45 percent KOH using a zinc anode of 4 square centimeters. The current was controlled with a constant current discharge unit. (A constant current discharge unit was built to NASA specification by Smith Electronics Inc. The current control was ±0.1 percent from 0 to 1 ampere.) A copper coulometer in series with the cathode was also used to check the coulombic content. Figure 2 shows a block diagram of the discharge assembly. Table I lists the electrons per molecule obtained. The copper coulometer and the constant current-time relation agree within experimental error (2 percent or ± 0.3 electron/molecule). The discharge of the cell was begun 1 minute after being placed in the electrolyte. It was found that this time was satisfactory for a stable open circuit voltage to occur, and this value is listed in table I. The cell voltages were measured with a digital voltmeter having an input impedance of 100 megohms and an accuracy of 0.1 percent. Figures 3 and 4 show typical constant current discharges of a $2Ag(AgO_2)_2 \cdot AgNO_3$ cathode in 45 percent KOH with a zinc anode. The chemically reduced form of $2Ag(AgO_2)_2 \cdot AgNO_3$ and AgO were treated in the same manner.

TABLE I. - MOLECULAR WEIGHT - COULOMBIC CONTENT

EQUIVALENT WEIGHT OF SILVER-OXYGEN COMPOUNDS

Compound	Molecular weight	Coulombic content, electrons molecule	Open circuit voltage against Zn anode in 45 percent KOH	Equivalent weight, g/V electron	Coulombic content experimentally determined, electrons molecule
$2Ag(AgO_2)_2 \cdot AgNO_3$	945.3	17	2.01	55.6	17
$2Ag(AgO_2)_2 \cdot Ag_2SO_4$	1087	18	2.01	60.2	18
$2Ag(AgO_2)_2 \cdot AgSO_4$	979.4	18	----	54.4	--
$2Ag(AgO_2)_2 \cdot AgF \cdot HF \cdot 1\frac{1}{2} H_2O$	949.3	17	2.01	55.8	17
AgO	123.9	2	1.85	62.0	2
$2Ag_3O_4 \cdot AgClO_4$	982.8	17	----	57.8	--
Ag_2O	231.8	2	1.60	115.9	--
$6AgO \cdot AgNO_3$	913.3	13	1.60	70.3	13

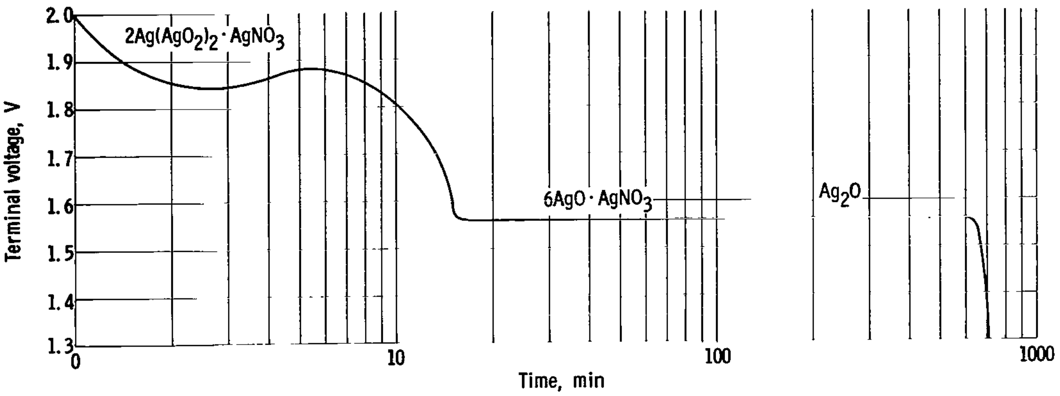


Figure 3. - Terminal voltage plotted against time. $2Ag(AgO_2)_2 \cdot AgNO_3$-45 percent KOH-Zn cell discharged at 9 milliamperes per square centimeter of $2Ag(AgO_2)_2 \cdot AgNO_3$.

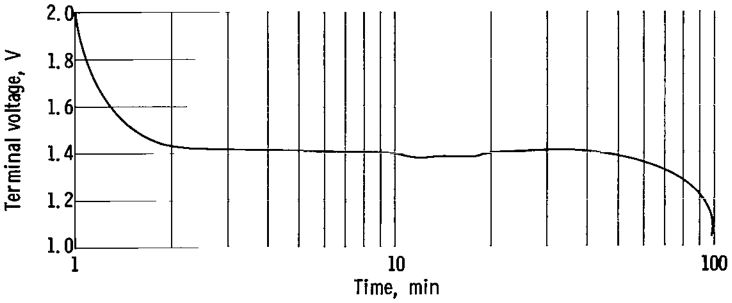
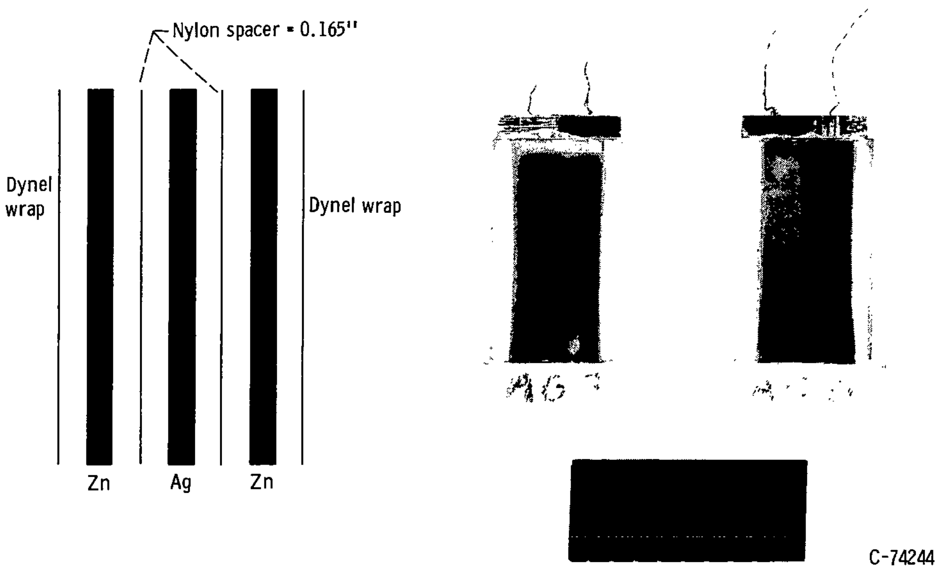


Figure 4. - Terminal voltage plotted against time. $2Ag(AgO_2)_2 \cdot AgNO_3$-45 percent KOH-Zn cell discharged at 99 milliamperes per square centimeter of $2Ag(AgO_2)_2 \cdot AgNO_3$.



(a) Internal construction of lab cell.

(b) External view of lab cell.

Figure 5. - Lab cell.

Lab Cell

Electrode preparation. - The dried argentic oxysalts were compressed at 3600 pounds per square inch into plates 1 by 3 centimeters. A platinum reinforcing screen with a platinum lead wire was included in the electrode.

Cell construction. - Figure 5(a) shows the construction used for keeping the electrodes permanently spaced. A polyamide (nylon) netting was placed between the anode and cathode to prevent the electrodes from physically contacting each other. The electrodes and polyamide netting were then wrapped with an acrylonitride mat to prevent the electrodes from separating. The combined electrodes and separators were then placed in a methacrylate case as shown in figure 5(b). Lab cells using AgO were constructed in the same manner.

Cell discharge. - Figure 6 shows typical current-terminal voltage curves for cells constructed with $2Ag(AgO_2)_2 \cdot AgNO_3$ and AgO cathodes. The anode for both cells was zinc. A 45 percent KOH solution was added to the cells, and after a stable terminal voltage was established they were discharged using a variable load. The terminal voltage was measured with a digital voltmeter having a 100-megohm input impedance and 0.1 percent accuracy. The current was measured with a similar voltmeter across a known resistance.

Thermal Coefficients of Terminal Voltage of Argentic Oxynitrate-Zinc-45 Percent KOH System

Beaker cells of $2Ag(AgO_2)_2 \cdot AgNO_3$ and the reduction product of $2Ag(AgO_2)_2 \cdot AgNO_3$ with 45 percent KOH were constructed with zinc as the anode and 45 percent KOH as the electrolyte.

The cells were placed in a constant temperature bath that controlled the temperature to ±0.5° C. The terminal voltages were read with a differential voltmeter having a 100-megohm input impedance and an accuracy of 0.05 percent.

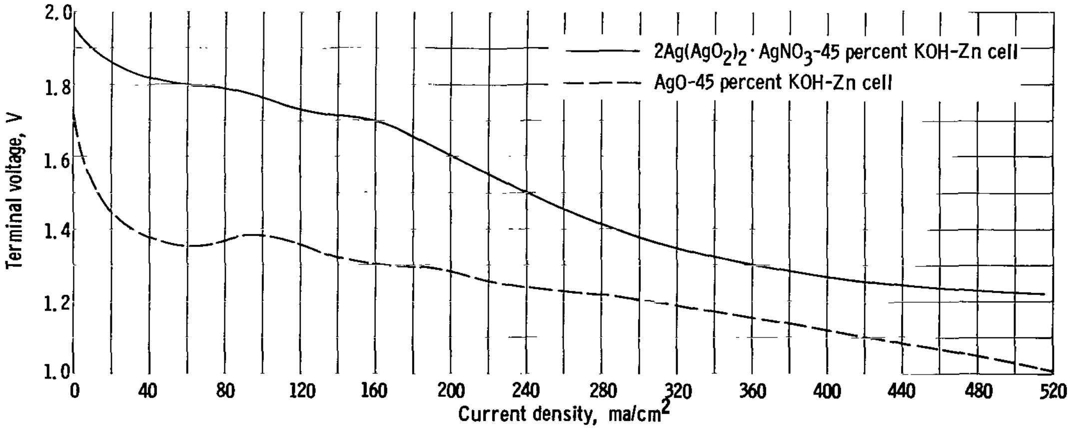


Figure 6. - Terminal voltage plotted against current of $2Ag(AgO_2)_2 \cdot AgNO_3$-45 percent KOH-Zn cell and AgO-45 percent KOH-Zn cell.

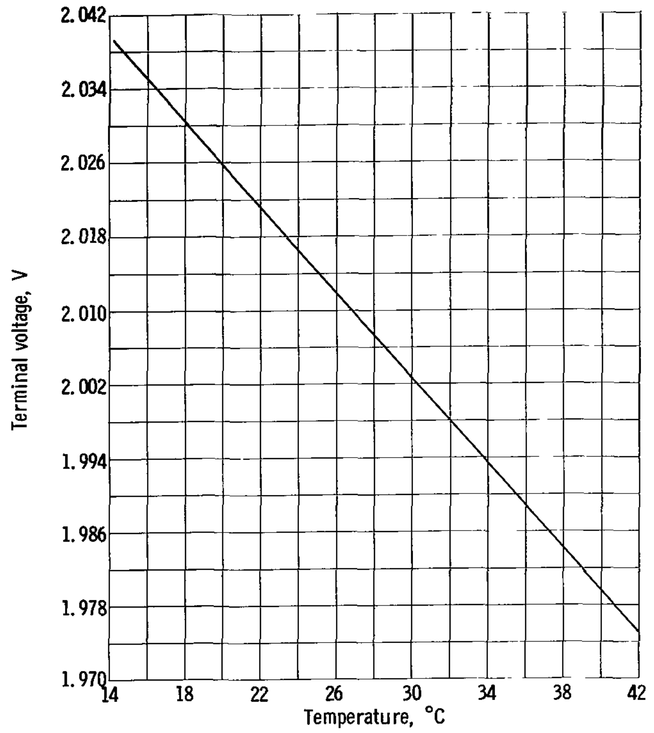


Figure 7. - Terminal voltage plotted against temperature of $2Ag(AgO_2)_2 \cdot AgNO_3$-45 percent KOH-Zn cell.

The cells were assumed to have reached the equilibrium potentials when the terminal voltage remained constant within the accuracy of the voltmeter for 5 minutes at each temperature.

Because of the reactivity of the $2Ag(AgO_2)_2 \cdot AgNO_3$ electrode, it was necessary to use a new electrode for each temperature-voltage measurement. The electrolyte and anode were brought to the temperature of the water bath before the cathode was introduced into the system. Figure 7 shows a plot of terminal voltage as a function of temperature for a cell constructed from $2Ag(AgO_2)_2 \cdot AgNO_3$. Figure 8 shows a plot of terminal voltage as a function of temperature for a cell constructed from the reduction product of $2Ag(AgO_2)_2 \cdot AgNO_3$ with 45 percent KOH.

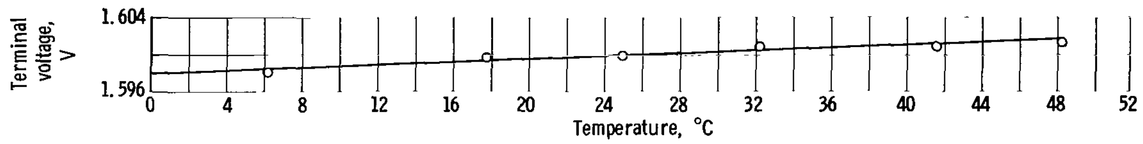


Figure 8. - Terminal voltage plotted against temperature of $6AgO \cdot AgNO_3$-45 percent KOH-Zn cell.

RESULTS AND DISCUSSION

Preparation and Identification

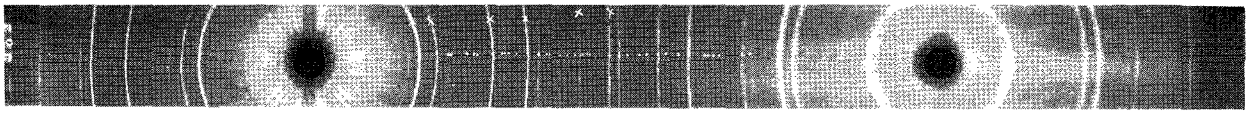
The products formed at the anodes during electrolysis of neutral solutions of silver nitrate, silver fluoride, or silver sulfate were analyzed both by chemical and X-ray methods (table II and fig. 9, respectively). These compounds were found to conform to a basic formula represented by $2Ag(AgO_2)_2 \cdot AgX$ crystals. Figure 10 is a photograph of $2Ag(AgO_2)_2 \cdot AgNO_3$ magnified 96 times. The product from the silver perchlorate electrolysis is too unstable (ref. 14) to allow an analysis to be performed. The formula for the product is given by M. S. Skanavi-Grigor'eva and I. L. Shimonovich (ref. 2) as $2Ag_3O_4 \cdot AgClO_4$.

The reaction for the deposition of argentic oxynitrate and argentic oxysulfate is (ref. 14)

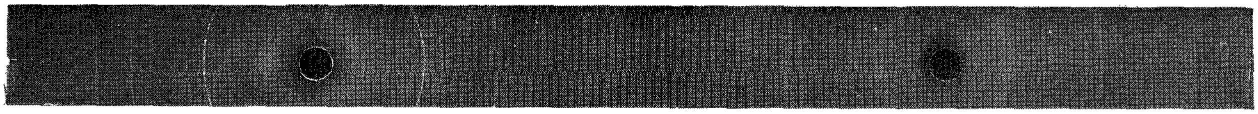
TABLE II. - CHEMICAL ANALYSIS OF

ARGENTIC OXYSALTS

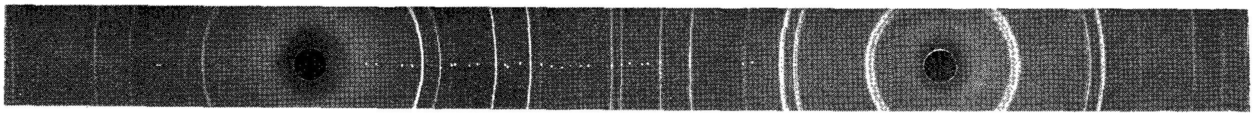
Argentic Oxysalts	Ag	O	NO_3	F	S
Electrolytically prepared compounds					
Argentic oxynitrate	79.6	13.3	6.6	---	---
Argentic oxysulfate	78.9	17.9	---	---	3.0
Argentic oxyfluoride	80.2	13.5	---	4.0	---
Chemically prepared compound					
Argentic oxysulfate	79.4	17.7	---	---	3.0



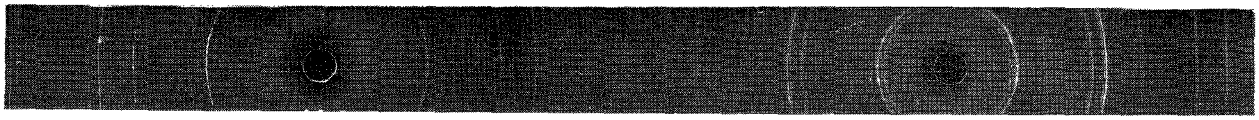
(a) Argentic oxynitrate electrolytically prepared.



(b) Argentic oxyfluoride electrolytically prepared.



(c) Argentic oxysulfate electrolytically prepared.



(d) Argentic oxy sulfate chemically prepared.

Figure 9. - X-ray diffraction pattern of argentic oxysalts.

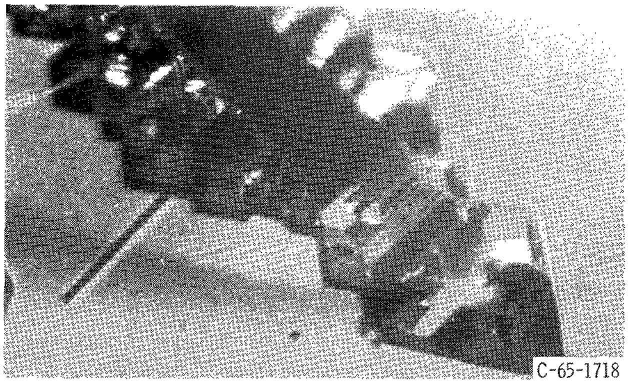


Figure 10. - Silver oxynitrate. X96. (Reduced 50 percent in printing.)

$$\text{Anode:}\quad 7Agx+8H_2O \rightarrow 2Ag(AgO_2)_2\cdot Agx+6Hx+10H^{+}+10e$$

$$\text{Cathode:}\quad 10Agx+10H^{+}+10e \rightarrow 10Ag+10Hx$$

$$\text{Cell:}\quad 17Agx+8H_2O \rightarrow 2Ag(AgO_2)_2\cdot Agx+16Hx+10Ag$$

Argentic oxysulfate was prepared electrolytically and chemically as reported in the work of de Boer and van Ormondt (ref. 7). The products obtained were examined by X-ray and chemical analysis. X-ray diffraction patterns (fig. 9) of both products showed them to be the same and the chemical analysis (table II) showed that they conformed to the formula $2Ag(AgO_2)_2\cdot Ag_2SO_4$. The product reported by de Boer was $2Ag(AgO_2)_2\cdot AgSO_4$. The divalent silver attached to the sulfate ion would produce a molecule with 18 available electrons and the lowest equivalent weight of the known argentic oxysalts. Table I (p. 3) lists the compounds and their respective equivalent weight.

If argentic oxynitrate was allowed to soak in 45 percent KOH it slowly decomposed with the evolution of oxygen. The product of this degradation was not AgO as was first thought. X-ray diffraction analysis, coulometric reduction and entropy values indicate this as a different compound. The coulometric reduction yielded 13 available electrons, where 17 were originally obtained, which indicates 6 divalent silver atoms with a monovalent silver atom attached. The open circuit voltage of this material in 45 percent KOH with a zinc anode was 1.60 volts at 25° C. This lower voltage would indicate a complexing of the silver atoms. This compound was then arbitrarily given the designation of $6AgO\cdot AgNO_3$.

Cell Discharge

The utilization of silver oxynitrate based on a 17-electron-per-molecule reaction was found to be 100 percent at current drains of 9 milliamperes per square centimeter for 0.2150 gram of material and 94 percent at 99 milliamperes per square centimeter for 0.3298 gram of material. This represents a discharge rate of C/11.5 and C/1.6 respectively where C/X equals the capacity of the electrode discharged at a rate so that it would be completely discharged in X hours.

Table III lists the standard basic solution electrode potentials (ref. 14) of zinc and silver oxide reaction referenced to a standard hydrogen electrode.

The electrochemical reduction of AgO is a two-step process represented by $Ag^{2+} \xrightarrow{1e} Ag^{+1} \xrightarrow{1e} Ag^{o}$. The electrochemical reduction of argentic oxynitrate would require a three-step process represented by $Ag^{3+} \xrightarrow{1e} Ag^{2+} \xrightarrow{1e} Ag^{+1} \xrightarrow{1e} Ag^{o}$. Each step would be characterized by a voltage associated with its reaction. A cell composed of a AgO cathode and a zinc anode in a KOH solution where the activity of the hydroxyl ion would be unity would have a terminal voltage of 1.854 volts. When the silver was converted to the monovalent silver oxide (Ag_2O), the potential would be 1.590. In practice these voltages have been found to be 1.856 and 1.602, respectively, for the electrodes in a 45 percent KOH electrolyte.

The same type of discharge would also be expected from argentic oxynitrate. The reduction mechanism would be $2Ag(AgO_2)_2 \cdot AgNO_3 + 2Zn \rightarrow 6AgO \cdot AgNO_3 + 2ZnO$ with a terminal voltage of 2.01 volts followed by $2(6AgO \cdot AgNO_3) + 2KOH + 6Zn \rightarrow 7Ag_2O + 2KNO_3 + H_2O + 6ZnO$ with a terminal voltage of 1.600. The third step would be the same as that reported for Ag_2O+Zn (1.602 V).

Figures 3 (p. 4) and 11 show the effect of low current discharge for a cell constructed from argentic oxynitrate, 45 percent KOH, and zinc. In figure 3 a plateau voltage above 1.85 volts followed by a plateau voltage of 1.56 volts is what would be expected from the aforementioned reaction mechanism for argentic oxynitrate. The difference between 1.600 and 1.602 volts representing $6AgO \cdot AgNO_3$ and Ag_2O reactions, respectively, is too small to be seen.

The deviation in the curve shown in figure 3 from 1.5 to 5 minutes is attributed to the penetration of electrolyte into the electrode producing a higher surface area resulting in a rise in the operating voltage.

Energy Density

The calculated energy density based on 1.85-volt open circuit voltage of AgO against zinc is 239 watt hours per pound. The best obtainable capacity to date is 100 watt hours per pound (ref. 15). The calculated energy density of active material of argentic oxynitrate against zinc based on 2.01 open circuit voltage is 275 watt hours per pound. The energy density of a "beaker" cell at 41-milliampere discharge with an operating voltage of 1.50 volts was 187 watt hours per pound.

TABLE III. - STANDARD AQUEOUS ELECTRODE POTENTIALS AT 25° C FOR BASIC SOLUTIONS

	E^0, V
$Ag_2O_3 + H_2O + 2e \rightarrow 2AgO + 2OH^-$	0.739
$2AgO + H_2O + 2e \rightarrow Ag_2O + 2OH^-$	0.609
$Ag_2O + H_2O + 2e \rightarrow 2Ag + 2OH^-$	0.345
$Zn + 2OH^- \rightarrow ZnO + H_2O + 2e$	-1.245
Cell reactions	
$Ag_2O_3 + Zn \rightarrow 2A_gO + ZnO$	1.984
$2AgO + Zn \rightarrow Ag_2O + ZnO$	1.854
$Ag_2O + Zn \rightarrow 2Ag + ZnO$	1.590

Polarization

Polarization loss is the loss of the reversible potential resulting from kinetics, mass transport, and resistance factors during the discharge of a cell. The polarization loss of AgO and $6AgO \cdot AgNO_3$ at various drain rates is shown in figure 12.

Thermal Coefficients of Terminal Voltages of Argentic Oxynitrate-Zinc-45 Percent KOH Systems

The thermal coefficients for the open circuit voltage were found by the method of least squares to be -2.26×10^{-3} and 1.01×10^{-4} volt per °K, respectively, for $2Ag(AgO_2)_2 \cdot AgNO_3$ and $6AgO \cdot AgNO_3$ cathodes

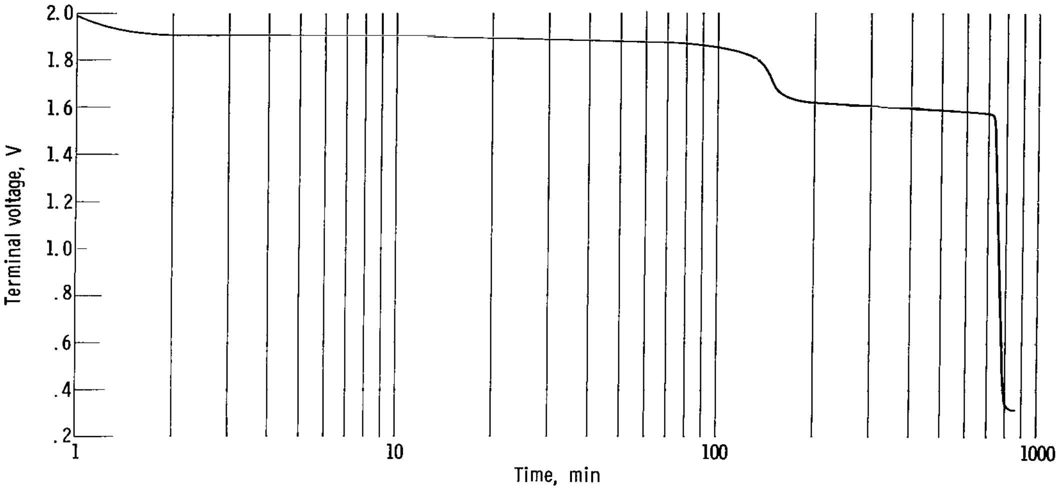


Figure 11. - Terminal voltage plotted against time at constant load of 1000 ohms of $2Ag(AgO_2)_2 \cdot AgNO_3$-45 percent KOH-Zn cell.

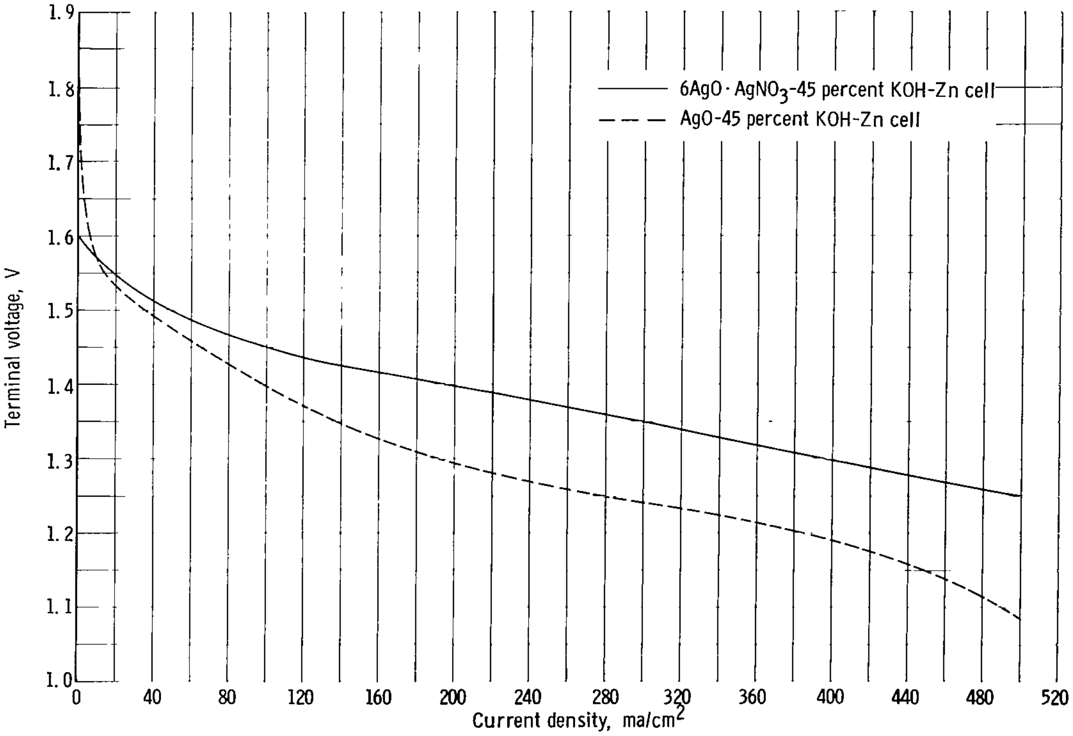


Figure 12. - Terminal voltage plotted against current of $6AgO \cdot AgNO_3$-45 percent KOH-Zn cell and AgO-45 percent KOH-Zn cell.

TABLE IV. - THERMODYNAMIC VALUES FOR SILVER OXIDES AT 25° C

Reactants	dE/dT, V/°K	Enthalpy, ΔH, kcal	Free energy, ΔF, kcal	Entropy, ΔS, eV
$2[2Ag(AgO_2)_2 \cdot AgNO_3]+4Zn$	-2.26×10^{-3}	-496.0	-371.2	-83.3
$2(6AgO \cdot AgNO_3)+2KOH+6Zn$	1.01×10^{-4}	-434.4	-442.8	28
$2AgO+Zn$	5.70×10^{-5}	-84.8	-85.6	2.68
$7Ag_2O+7Zn$	-1.69×10^{-4}	-533.4	-518.0	-51.7

in 45 percent KOH with zinc anodes. The values reported for the divalent and monovalent silver oxides (AgO and Ag_2O) are 5.70×10^{-5} and -1.69×10^{-4} volt per °K, respectively (ref. 16). The difference between the thermal coefficients of the cell voltage for the two types of divalent silver oxides ($6AgO \cdot AgNO_3$ and AgO), 1.01×10^{-4} and 5.70×10^{-5} volt per °K add substantiating evidence that the compounds are definitely different.

From the potential reductions given previously, the equation for the reduction of argentic oxynitrate in 45 percent KOH with zinc anodes is assumed to be

$$2[2Ag(AgO_2)_2 \cdot AgNO_3]+4Zn \rightarrow 2(6AgO \cdot AgNO_3)+4ZnO$$

$$2(6AgO \cdot AgNO_3)+2KOH+6Zn \rightarrow 7Ag_2O+2KNO_3+H_2O+6ZnO$$

$$7Ag_2O+7Zn \rightarrow 14Ag^0+7ZnO$$

Values for the change of enthalpy of the cell can be calculated by means of the Gibbs-Helmholtz equation ($\Delta H = nF[T(dE/dT) - E]/4.184$) for each reaction. The total cell enthalpy can then be obtained by taking the algebraic sum of each step. Similarly, the free energy ($\Delta F = -nFE/4.184$) and entropy ($\Delta S = (\Delta H - \Delta F)/T$) can be obtained for the cell. Table IV lists the thermodynamic values calculated for $2Ag(AgO_2)_2 \cdot AgNO_3$ and $6AgO \cdot AgNO_3$ and the reported values for AgO and Ag_2O.

The significance of the positive entropy is that the cell will absorb heat from the environment when discharged reversibly.

CONCLUSIONS

The use of argentic oxynitrate as cathodes in electrochemical cells has been shown to be feasible. Cells with an open circuit potential of 2.0 volts have been constructed and discharged at rates of C/10 to C/1 where C/X equals the capacity of the electrode discharged at a rate so that it would be completely discharged in X hours. These cells were found to have cathode utilizations between 94 and 100 percent. These compounds are characterized by high polarization losses. The thermal coefficient of argentic oxynitrate-zinc cell has an unusually high value of -2×10^{-3} volt per $^{\circ}K$.

The stability of argentic oxynitrate in 45 percent KOH was investigated, and it was found that there is a slow decomposition with the evolution of oxygen. The decomposition ends with the formation of a new stable compound which contains 13 available electrons per molecule. This compound has been arbitrarily assigned a formula of $6AgO \cdot AgNO_3$. The material has an open circuit voltage of 1.6 volts in 45 percent KOH with a zinc anode. The discharge is not affected by the large polarization loss present with electrodes of argentic oxysalts. The thermal coefficient of this material in a cell with a zinc anode is positive, which will allow the cell to be discharged without the liberation of heat if carried out near the reversible rate.

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