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DEVELOPMENT OF AN IMPROVED OXYGEN ELECTRODE FOR USE IN ALKALINE H_2-O_2 FUEL CELLS

Q-6
Report for Period
October 1-December 31, 1967

Contract NASA W-12,300

National Aeronautics and Space Administration
NASA Headquarters, Washington, D.C. 20546

DEVELOPMENT OF AN IMPROVED OXYGEN ELECTRODE FOR USE IN ALKALINE H_2-O_2 FUEL CELLS

The objective of this project is the synthesis of interstitial compounds for increasing the efficiency of the oxygen electrode in alkaline H_2-O_2 fuel cells. The work is being carried out for the National Aeronautics and Space Administration with Mr. E. M. Cohn as technical monitor. Principal investigators are Sayeed Akhtar, C.T. Grein, R.C. Diehl, W.H. Ryback, and D. Bienstock.

Bureau of Mines
Pittsburgh Coal Research Center

Quarterly Report for the period ending December 31, 1967

Development of An Improved Oxygen Electrode for Use in Alkaline H_2-O_2 Fuel Cells

Summary

A total of 26 preparations were synthesized and shipped to Tyco Laboratories in this quarter. Apparatus for testing activity of cathode catalysts in the reduction of oxygen is in operation now. Selected preparations from amongst the materials synthesized so far are being inducted by different methods and their activities tested. Results of different induction experiments on reduced 3Ni-1Au are presented. None of the induction methods tried so far has led to an improved catalyst.

Introduction

The object of this project is to develop catalysts for the cathodic reduction of oxygen in alkaline H_2-O_2 fuel cells. The work is being conducted in cooperation with Tyco Laboratories Inc., and the materials under investigation are the interstitial compounds of the transition metals, singly and in combinations, with carbon, nitrogen, and boron. Samples of the preparations are also sent to other firms designated by the Technical Monitor of N.A.S.A. This report deals primarily with the preparation of these compounds; information on the efficacy of the preparations in laboratory tests may be obtained from the reports of Tyco Laboratories, Inc., Waltham, Massachusetts.

Experimental

A. Preparation of Reduced Mixed Metals, Their Carbides, and Nitrocarbides

As described earlier (1), the following sequence of steps are involved:

$$\begin{tikzcd}
\text{Mixtures of salts of transition metals in solution} \arrow[d, "\text{NaOH, } 80^\circ\text{-}90^\circ\text{C}"] \\
\text{Coprecipitated hydroxides} \arrow[d, "H_2\text{, } 250^\circ \text{ or } 275^\circ\text{C, } \sim 85 \text{ hrs.}"] \\
\text{Reduced mixed metals} \arrow[d, "\text{CO, } 250^\circ\text{-}260^\circ\text{C, 100 hrs.}"] \\
\text{Carbides} \arrow[d, "NH_3\text{, } 260^\circ\text{C, 48 hrs.}"] \\
\text{Nitrocarbide}
\end{tikzcd}$$

Since platinum does not precipitate as hydroxide from a solution of its salt by the addition of NaOH, it was precipitated as Pt-black by reduction with alkaline sodium formate. The procedural details for the foregoing sequence of steps has been described earlier (2).

The mixed metals hydroxides that were reduced, carbided, and nitrocarbided during this quarter are listed in tables 1, 2, and 3, respectively.

TABLE 1.- Reduction of mixed metals hydroxides

Reducing gas: H_2
Space velocity: 1,000 hr^{-1}
Time: ~ 85 hrs.

Run No.	Temperature of reduction, °C	Composition of reduced material
80R[1]	250	3Ni-1Pt
81R	"	1Ni-1Pt
82R	"	1Ni-3Pt
84R	275	3Ni-1Ag
85R	250	1Ag-1Pd
86R	"	3Ag-1Pd
87R	275	4Cr-1Mo
88R	250	1Au-3Pd
89R	"	1Au-1Pd
90R	"	3Au-1Pd

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[1] The letter R stands for reduced material.

TABLE 2.- Preparation of carbides

Carbiding gas: CO
Space velocity: 1,000 hr^{-1}

Run No.	Charge[1/]	Duration of carbiding, hrs.	Temp., °C	Chemical analysis, weight-percent Total C	 Free C
80C[2/]	3Ni-1Pt	4	160-260		
		105	260	5.96	3.40
81C	1Ni-1Pt	5	160-260		
		100	260	3.66	2.57
82C	1Ni-3Pt	4	160-260		
		105	260	1.21	0.97
84C	3Ni-1Ag	4	160-260		
		103	260	5.75	0.35
85C	1Ag-1Pd	5	160-260		
		104	260	0.29	0.10
86C	3Ag-1Pd	4	160-260		
		105	260	0.5	0.23
87C	4Cr-1Mo	5	160-260		
		103	260		
88C	1Au-3Pd	4	160-260		
		104	260		

[1/] Reduced coprecipitated hydroxides.
[2/] The letter C stands for carbide.

TABLE 3.- Preparation of nitrocarbides

Nitriding gas: NH_3
Space velocity: 1,000 hr^{-1}
Time, hours: 48
Temperature: 260 °C

Run No.	Charge	Chemical analysis, weight percent Total C	 Free C	 N
58NC[1]	80C (carbided 3Ni-1Pt)	5.36	3.57	0.47
59NC	81C[2] (carbided 1Ni-1Pt)	3.57	2.86	0.26
60NC	82C (carbided 1Ni-3Pt)	1.11	0.88	0.24
61NC	84C (carbided 3Ni-1Ag)	5.42	0.34	0.57
62NC	85C (carbided 1Ag-1Pd)			
63NC	86C (carbided 3Ag-1Pd)			
64NC	88C[2] (carbided 1Au-3Pd)			

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[1] The letters NC stand for nitrocarbide.
[2] Space velocity: 1,500 hr^{-1}.

X-ray analysis of the preparations 80C-84C gives evidence for the formation of nickel carbide only. It may further be noted from Table 2 that, under identical conditions of preparation, a far larger proportion of free carbon was found in the carbided 3Ni-1Pt than in the carbided 3Ni-1Ag. Since the total carbon was about the same in the two preparations, the carbidic carbon must be less in the carbided 3Ni-1Pt than in the carbided 3Ni-1Ag. Pt, it seems, tends to catalyze the decomposition of nickel carbide.

In confirmation of x-ray evidence, chemical analysis appears to indicate only negligible, if any, carbide formation in the cases of 1Ag-1Pd and 3Ag-1Pd.

B. Electrochemical Test Cell

a) Techniques

During the quarter, an apparatus was assembled to permit the electrochemical testing of the catalytic materials (figure 1). This apparatus was patterned after the floating electrode cell (3) described by Tyco Laboratories, Inc. We wish to acknowledge our sincere appreciation to the staff of Tyco Laboratories for their help in this area.

The cell is a double oxygen half-cell,

$$4\ OH^- = 2\ H_2O + O_2 + 4\ e^-,$$

with the electrode under test consuming oxygen by the reverse reaction and a counter electrode employing the forward reaction. With the test electrode grounded, current required to maintain the counter electrode at a given potential may be determined, and, by repeated measurement, the current-voltage performance curve of the test material traced.

Since the applied potential difference associated with a given current will be decreased due to the internal resistance, R_c, of the cell, the current-potential curve, as measured, is not comparable to measurements made in a different cell. Therefore, the correction factor,

$$\Delta E = I\ R_c$$

is customarily added to the applied potential to allow comparison of the results of different workers. The resistance of the cell was

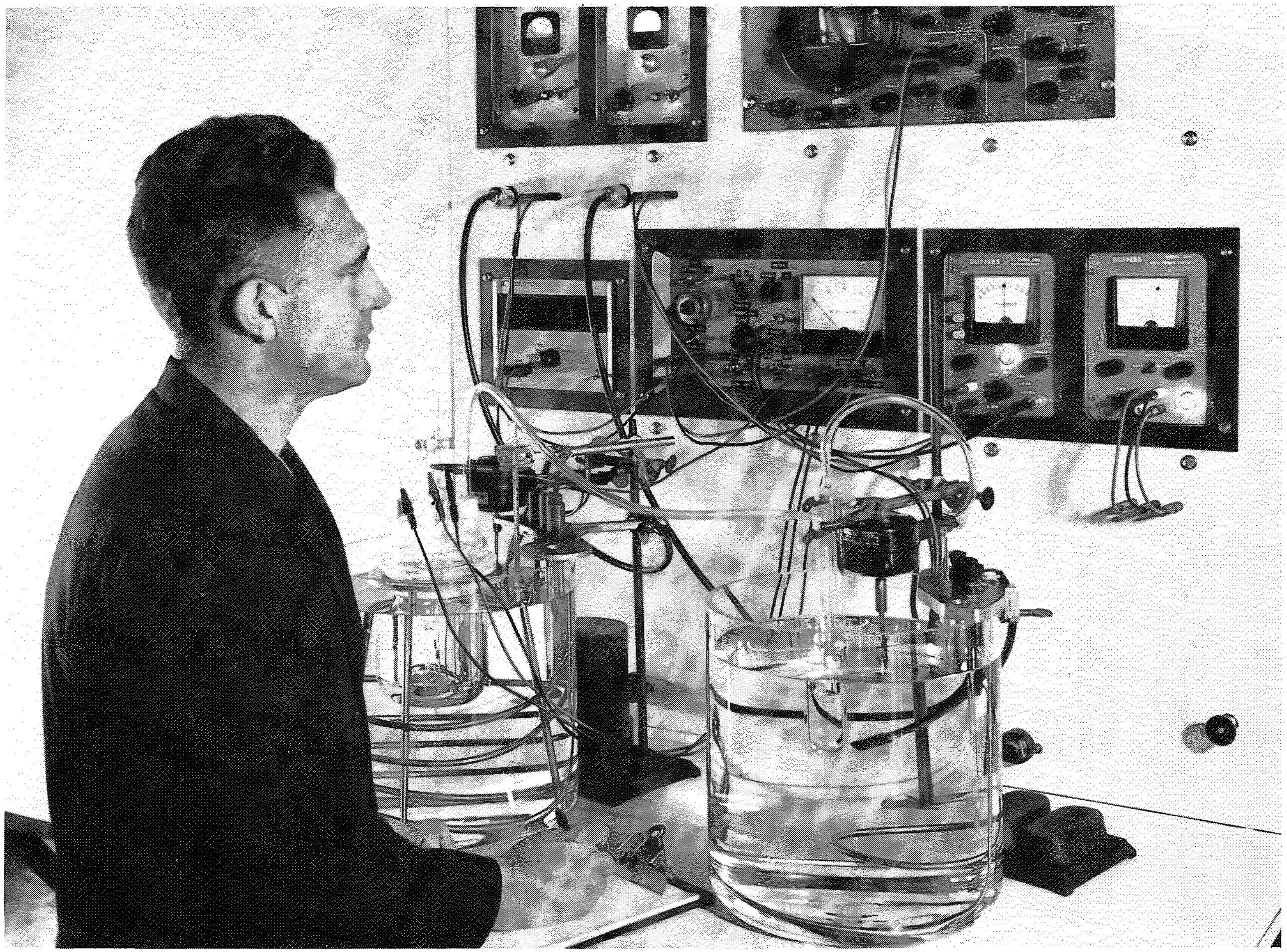

Figure 1- Apparatus for electrochemical testing of fuel-cell electrodes.

determined here by a relaxation technique. A known current was applied to the cell, then suddenly interrupted. The transient potential change was measured on an oscilloscope, with the instantaneous response attributed to resistive loss and the rest to capacitative decay. Then

$$\Delta E_{\mathrm{Inst.}}/I_{\mathrm{App.}} = R_c.$$

The reference electrode used was a hydrogen electrode of a type proposed by Giner (4). A pair of inert electrodes are immersed in the electrolyte and connected to a potential source to maintain a potential sufficient for the electrolysis of water. Once a steady state has been reached the negative electrode remains at a potential within about 30 mv of the hydrogen zero potential. The small correction required may be measured at the start or conclusion of an experiment by comparison with a truly reversible hydrogen electrode. A reference electrode of this type, operating at the potential of hydrogen evolution, is called a dynamic hydrogen electrode, or D.H.E. The D.H.E. is useful over an extreme range of pH and temperature, and lacks the elaborate equipment needed to operate a reversible hydrogen electrode for an extended period.

b) Physical Description

The physical construction of the test cell is shown in figure 2. The vessel is a 500 ml. commercial resin kettle, containing the electrolyte, 400 ml. of 35% KOH solution. The vessel is immersed in a bath of white oil thermostated at 75°±0.1°C. A four-hole cover provides entry for the electrical leads and for a supply of oxygen, which has been saturated with water vapor at 58° ± 0.1°C. Due to the corrosive nature of the electrolyte, all glass-to-glass contact is avoided by the use of teflon gaskets and plugs.

The support for the working electrode also serves as an isolated chamber for the D.H.E. The D.H.E. electrodes, a commercial formulation of platinum black on nickel screen bonded to platinum wire, are arranged so that oxygen generated at the positive electrode will not readily diffuse downward to the hydrogen electrode. Contact with the bulk electrolyte is through a Luggin capillary.

The counter electrode is simply a smooth platinum ring, as shown. This electrode supports the current required to maintain the working electrode at a fixed potential.

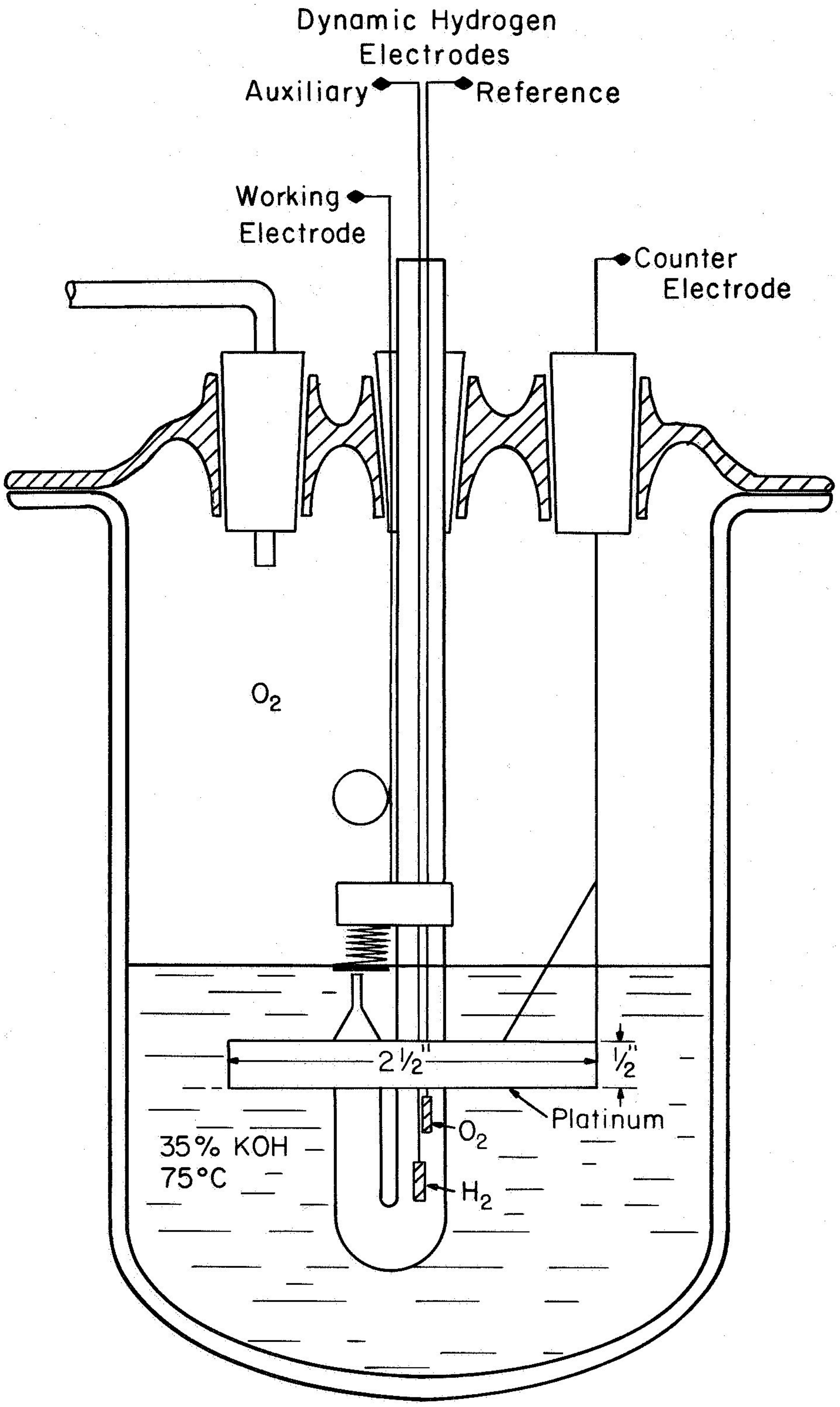

Figure 2.- Floating electrode assembly-electrochemical test cell for finely divided catalytic materials.

Electrodes to be tested are made by bonding finely-divided catalyst onto nickel screen. A standard amount, 250 mg in our experiments, of the finely-divided catalyst is kneaded with a few drops of water and a known volume of a 30% suspension of teflon. The dough-like kneaded mass is spread uniformly on an area at approximately 3 to 4 sq cm of a tightly stretched 100-mesh screen of 0.002-inch diameter nickel wire. The impregnated screen is dried for 15 minutes at 100 °C and then sintered in a stream of N_2 for 5 minutes at 275 °C. It is allowed to cool to room temperature in N_2 and then cut into circular discs of 1 sq cm with a sharp cork borer. The catalyst loading for each disc is calculated from the weight of the finished disc, the weight of a square centimeter of the blank screen, and the weight of sintered mass obtained from a unit volume of the teflon suspension in a separate experiment.

The disc thus obtained rests on a platinum ring on the working electrode support. A spring of platinum wire presses on the disc, to hold it in place and serve as an electrical contact. The disc is positioned just at the surface of the electrolyte but not immersed, so that electrolyte, oxygen, and catalyst are in intimate contact within the pores of the disc.

c) Circuit Description

The electrical circuitry is diagramed in figure 3. Current for the D.H.E. is furnished by a 45 volt battery, through a suitable resistance, to give a current density of 1 ma/cm^2 at the electrode. Provision is also made for operating the D.H.E. at 10 ma/cm^2. A high initial current density decreases the time required for the D.H.E. to stabilize.

For measurement of current-potential curves, the D.H.E. reference (negative) electrode and the working and counter electrodes are connected to the corresponding outputs of a potentiostat through a pair of double-pole double-throw switches. (Potentiostat requirements are rather high; the unit must be capable of handling 1.5 amps current at 1.5 volts. A commercial unit, designed primarily for electrodeposition experiments, was employed.) A ramp-step type digital voltmeter, with an input impedance of 10^6 ohms, is connected between working and reference electrodes, and an internal galvanometer on the potentiostat indicates the output current.

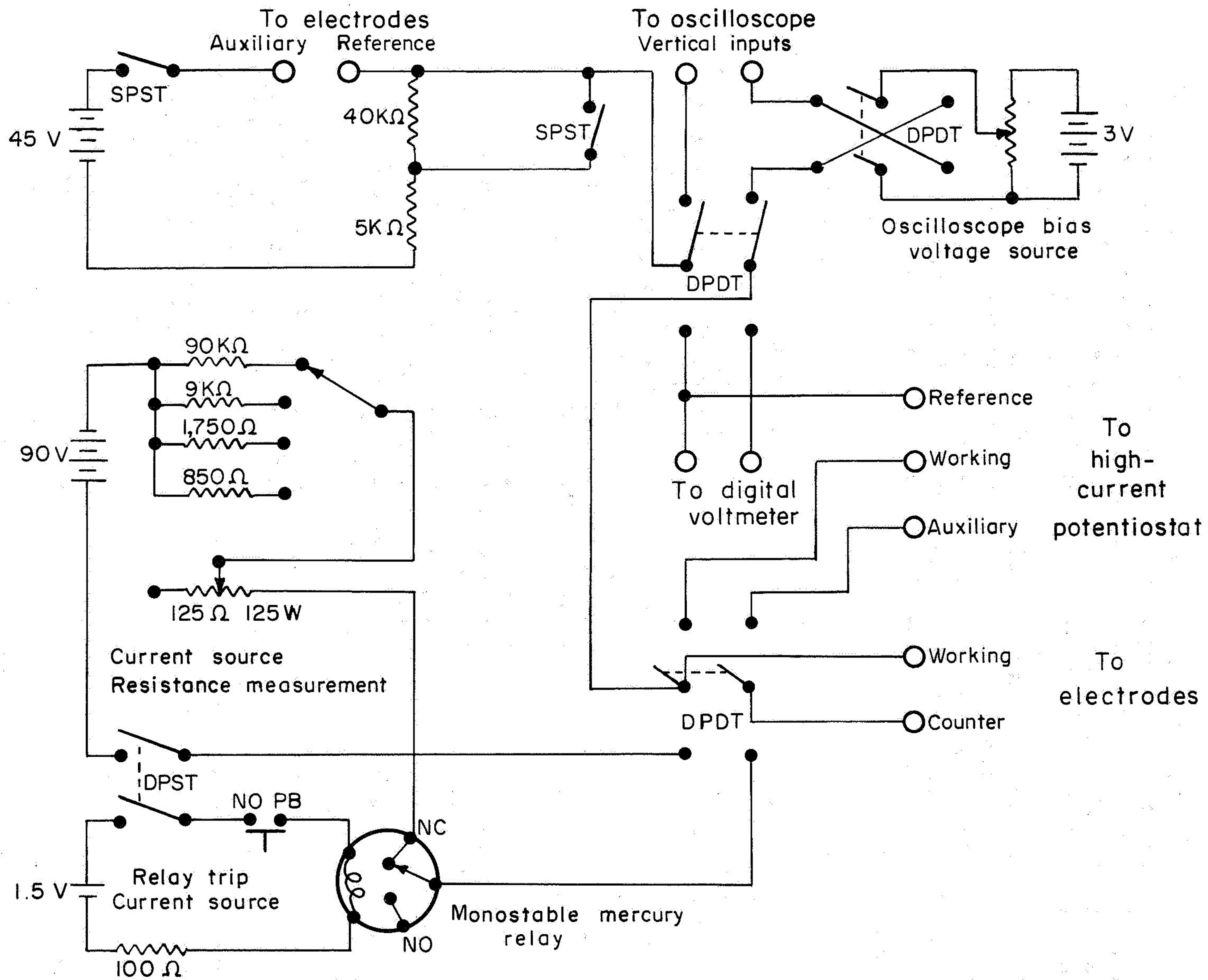

Figure 3.— Electrical circuit-electrochemical test cell for finely divided catalytic materials.

The opposite poles of the two main DPDT switches connect the cell to the resistance-measuring circuit. A 90 volt battery, connected through a series of resistances, furnishes a variable current between the working and counter electrodes, which is measured on an external ammeter. The voltage thus induced, relative to the D.H.E., is applied to the vertical input of a D.C. oscilloscope. When the mercury relay is tripped, current is interrupted abruptly. The instantaneous potential drop, discussed above, is measured from the oscilloscope trace. To increase sensitivity, the oscilloscope vertical amplifier is set for maximum gain, and a small battery and potentiometer are used to oppose the voltage applied to the cell and keep the oscilloscope trace on scale.

d) Operation

In practice, the following procedure is used:

Electrolyte solution, 35% KOH by weight, is prepared fresh before use from reagent grade KOH and distilled, deionized water treated to remove biological reducing agents. The newly prepared electrode disc is placed on the support and brought into surface contact with the electrolyte at 75°C. A slight stream of oxygen is passed over the electrolyte. The cell requires several hours to come to its resting potential; during this time, the D.H.E. is standardized.

The D.H.E. is initially operated at 10 ma/cm^2 current density for two minutes, to clear the electrode surfaces, then at 1 ma/cm^2. The potential of two identical reversible hydrogen electrodes is compared, to assure consistency, and one of the reversible electrodes is then compared to the D.H.E. The potential of the D.H.E. was found to stabilize within 10 minutes at the lower current density and remain constant, within ±1 mv, over a 6-hour period.

Once a stable resting potential is reached, a potential sweep is begun, starting at the rest potential and proceeding to zero. Current readings are taken at 25 and 50 mv intervals. The time required for the current to stabilize at the first point is noted, and all successive current readings are taken after equal stabilization periods. Upon completion of the downward sweep, a sweep upward to the rest potential is made.

Upon completion of the potential sweeps, the cell internal resistance is measured. A current is applied to the cell, and its value is adjusted to a convenient value (usually 25 ma). The potential of the bias circuit is adjusted so that the trace appears on scale at highest gain. The current is interrupted, and the transient recorded with an oscillograph camera for more leisurely evaluation. Average resistance for the teflon-bound discs is 0.7-0.8 ohm.

Graphically presented results include both IR-corrected and uncorrected curves.

e) Calibration

To evaluate the effectiveness of the apparatus, two samples of American Cyanamid Type AB-4 fuel cell electrode material were tested by the above procedure. AB-4 is a platinum black on nickel screen, but teflon is not employed as a binder. The test results are shown in table 4. The two samples gave identical values in the range indicated. The values obtained are in good agreement with other studies of commercial platinum electrodes in this system (5).

TABLE 4.- Performance of American Cyanamid type AB-4 electrode

I, ma/cm^2	E, (IR-free) mv (vs. hydrogen zero)
8	975
22	950
55	925
90	900

f) Activity Tests on 74R (3Ni-1Au) and 74C (3Ni-1Au, carbided)

A series of tests were performed on 74R and 74C, as such and modified by various methods of pretreatment. The results are presented in Figures 4-9. For secondary reasons, the internal resistance of the cell could not be determined in certain cases; hence, some of the curves are not corrected for IR drop.

Figures 4 and 5 were obtained with 3Ni-1Au when the loadings were different, whereas figures 6 and 7 were obtained with the same material after samples of it were treated with petroleum ether for different lengths of time. In one case, the material was left exposed to air for 24 hours after removal of petroleum ether. Each of the materials was then fabricated into two electrodes, one of which was tested immediately after preparation and the other 24 hours later. It will be seen that, in all cases, the activity of 3Ni-1Au was adversely affected by these treatments.

As is known, with a very few exceptions, finely divided reduced transition metals and their carbides are very sensitive to atmospheric oxidation, which leads to serious loss of their catalytic activity. The purpose of these pretreatments was an attempt to reduce their extraordinary reactivity with oxygen by covering the surface with a thin layer of oxide film by slow access of oxygen.

However, as has been pointed out, the activity of the material so treated was found to be far lower than that of the untreated 3Ni-1Au. Exposure to H_2S vapors poisoned both reduced 3Ni-1Au (fig. 8) and carbided 3Ni-1Au (fig. 9).

Work Plan

Further treatment of the preparations to prevent their oxidation and thus conserve their catalytic activity will be continued. A selected number of the materials which, on the basis of the earlier screening by Tyco Laboratories, appeared to offer better promise will be more intensively investigated. The few remaining preparations agreed upon with the technical monitor of N.A.S.A. will be completed.

References

(1) Quarterly Report to N.A.S.A. for the quarter ended September 30, 1967, Q-5, Development of an Improved Oxygen Electrode for Use in Alkaline H_2-O_2 Fuel Cells, Bureau of Mines, Pittsburgh Coal Research Center, Pittsburgh, Pa.

(2) Quarterly Report to N.A.S.A. for the quarter ended June 30, 1967, Q-4, Development of an Improved Oxygen Electrode for Use in Alkaline H_2-O_2 Fuel Cells, Bureau of Mines, Pittsburgh Coal Research Center,Pittsburgh, Pa.

(3) Report to N.A.S.A. for the Period ending January 31, 1966, Development of Cathodic Electrocatalysts for Use in Low Temperature H_2/O_2 Fuel Cells with an Alkaline Electrolyte, Tyco Laboratories, Inc., Bear Hill, Waltham, Mass., pp. 18-21.

(4) Giner, J., A Practical Reference Electrode. J. Electrochem. Soc., v. 111, No. 3, 1964, pp. 376-377.

(5) Quarterly Report to N.A.S.A. for the period ending June 30, 1966, Q-4, Development of Cathodic Electrocatalysts for Use in Low Temperature H_2/O_2 Fuel Cells with an Alkaline Electrolyte, Tyco Laboratories, Inc., Bear Hill, Waltham, Mass., p. 67.

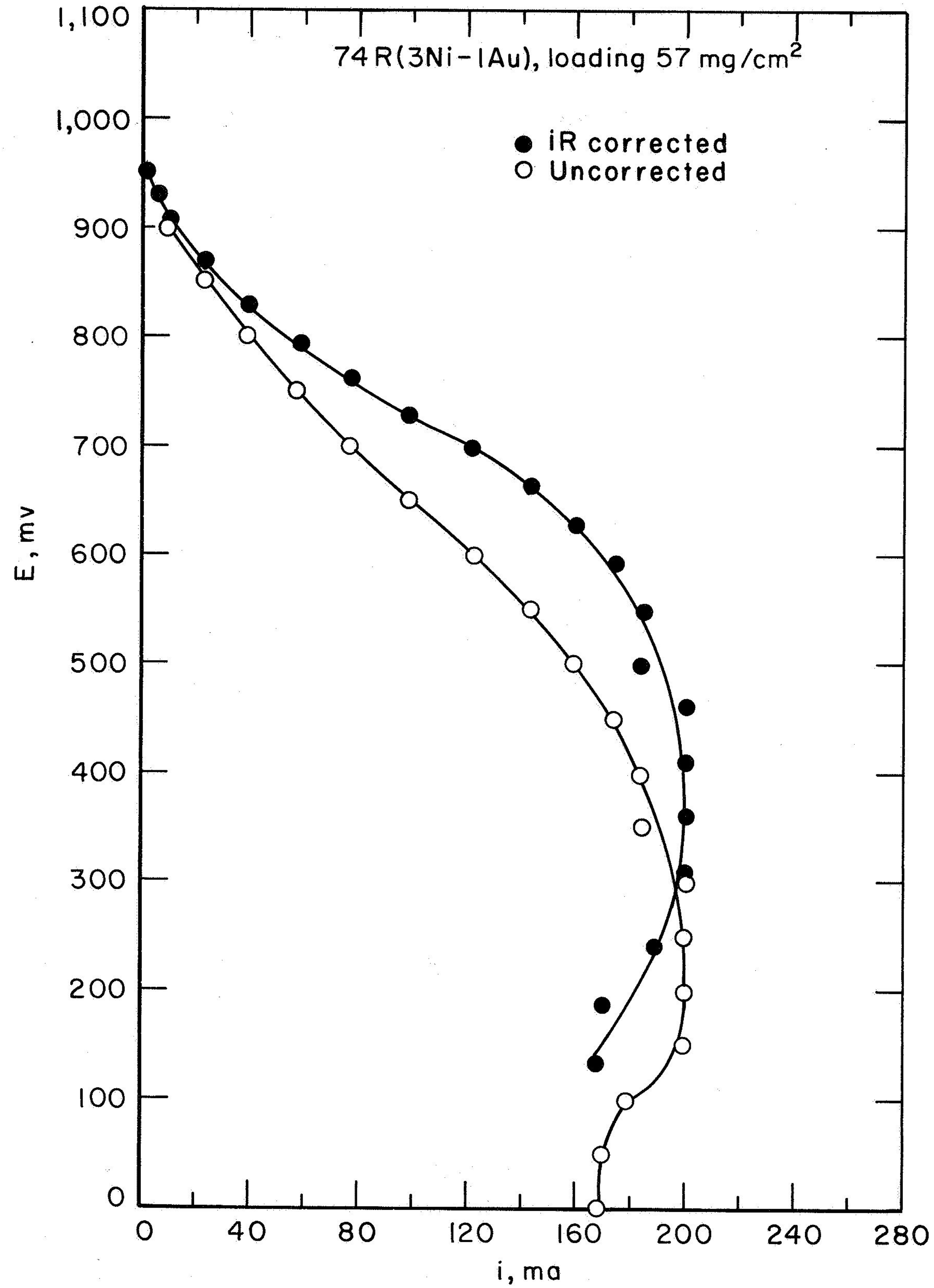

Figure 4.—i-E curve for 3Ni-1Au in the reduction of O_2.

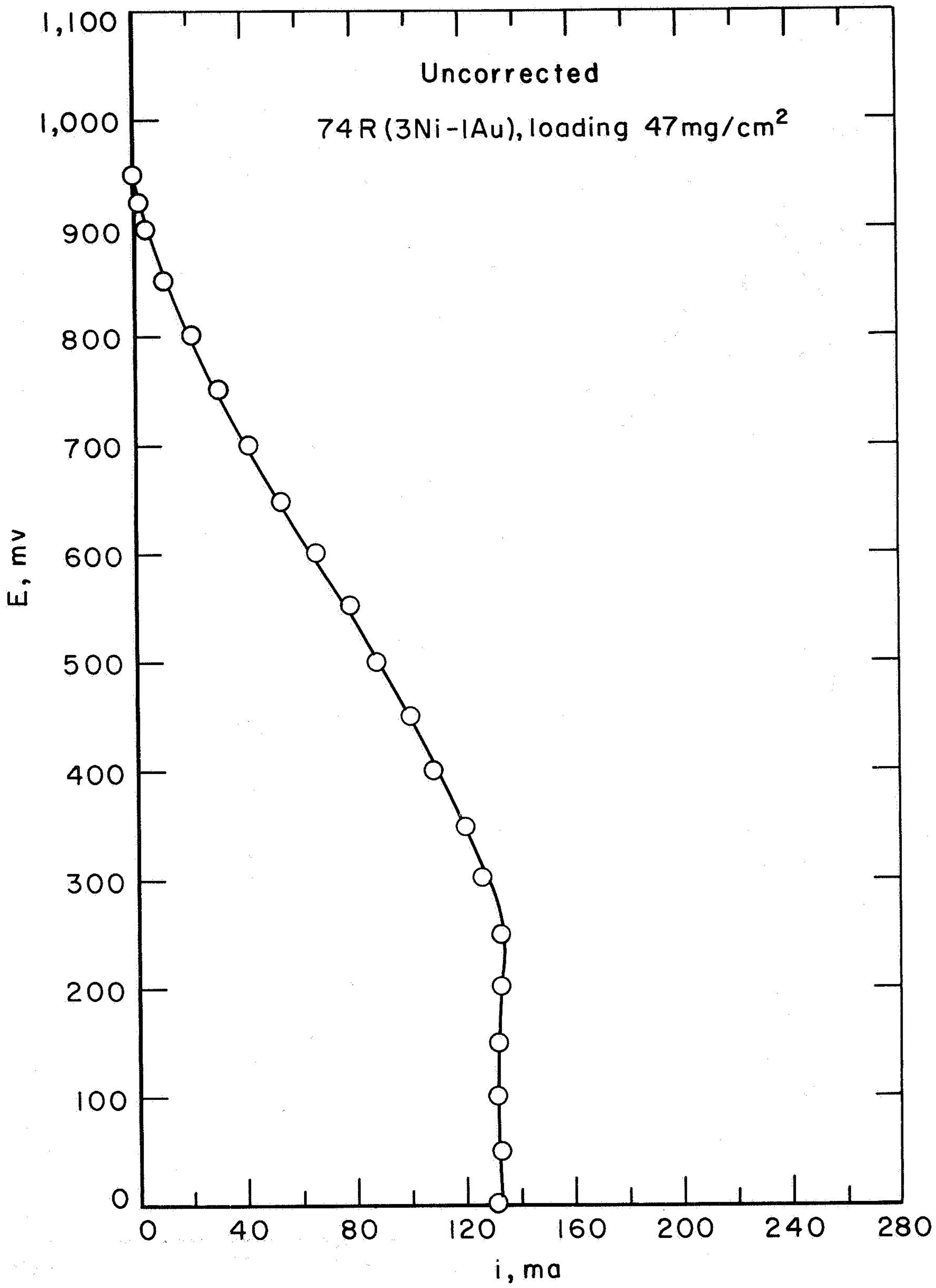

Figure 5.— i–E curve for 3Ni–1Au in the reduction of O_2.

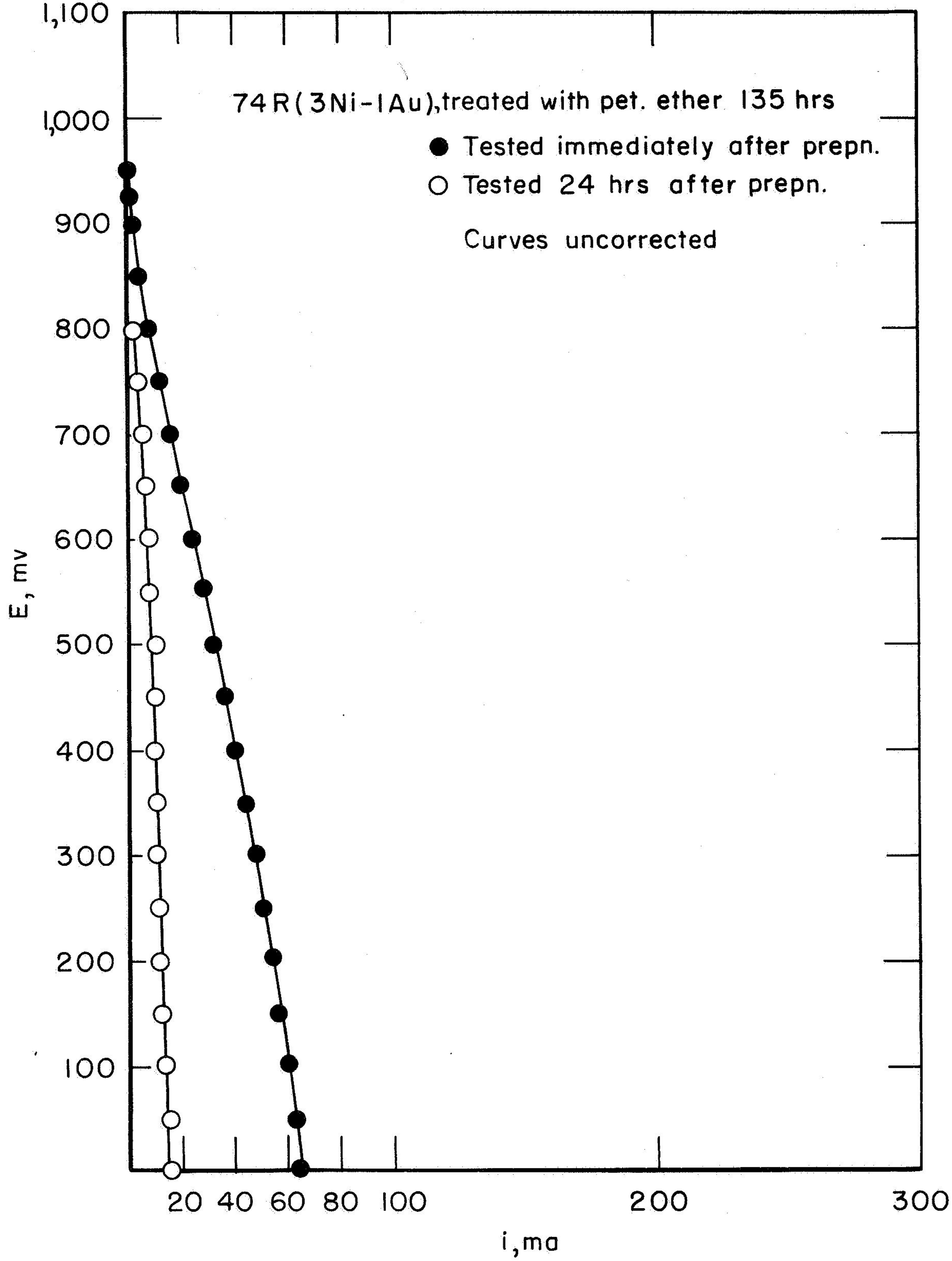

Figure 6.—i—E curve for 3Ni —IAu, treated with pet. ether for 135 hours.

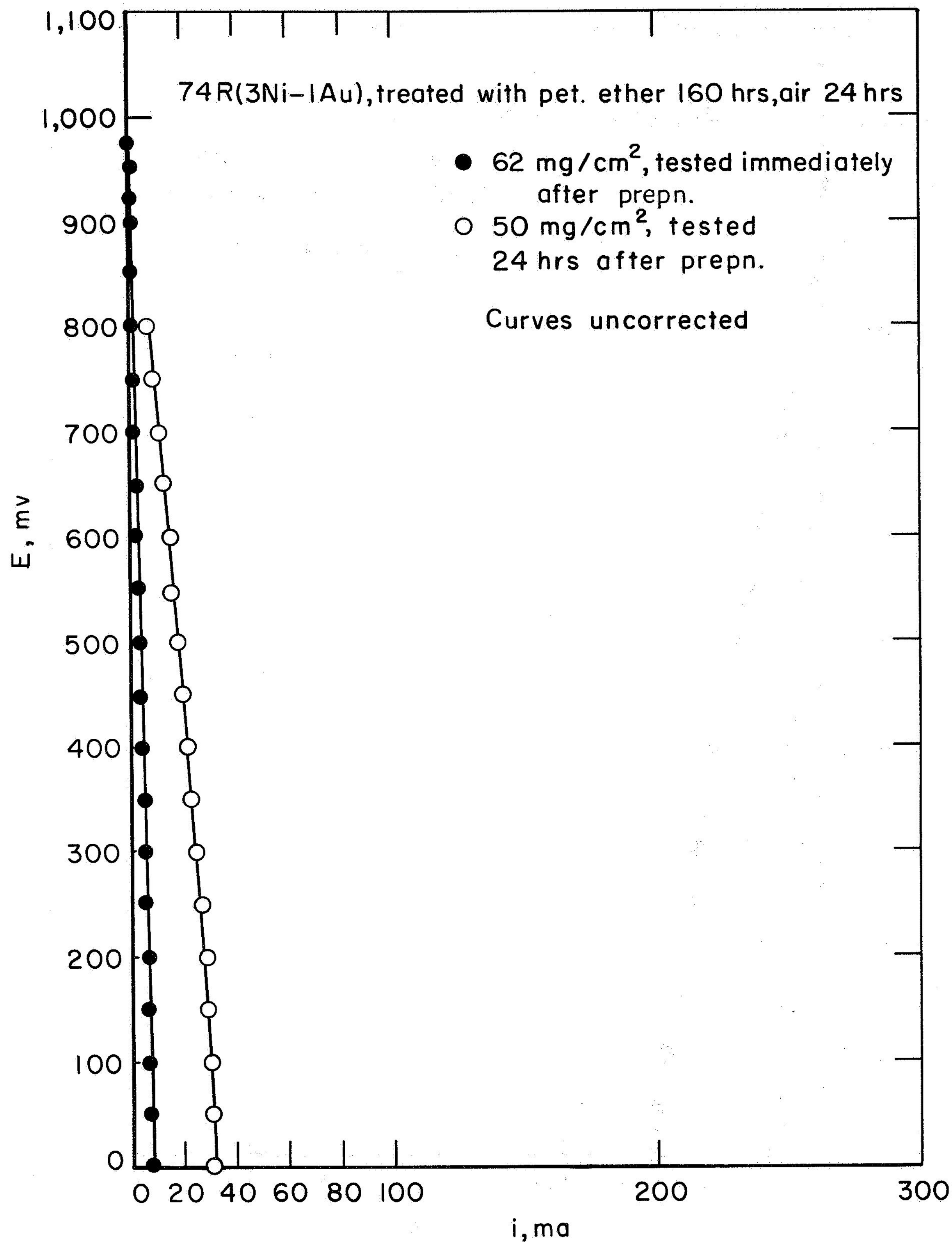

Figure 7.—i-E curve for 3Ni-1Au, treating with pet ether.

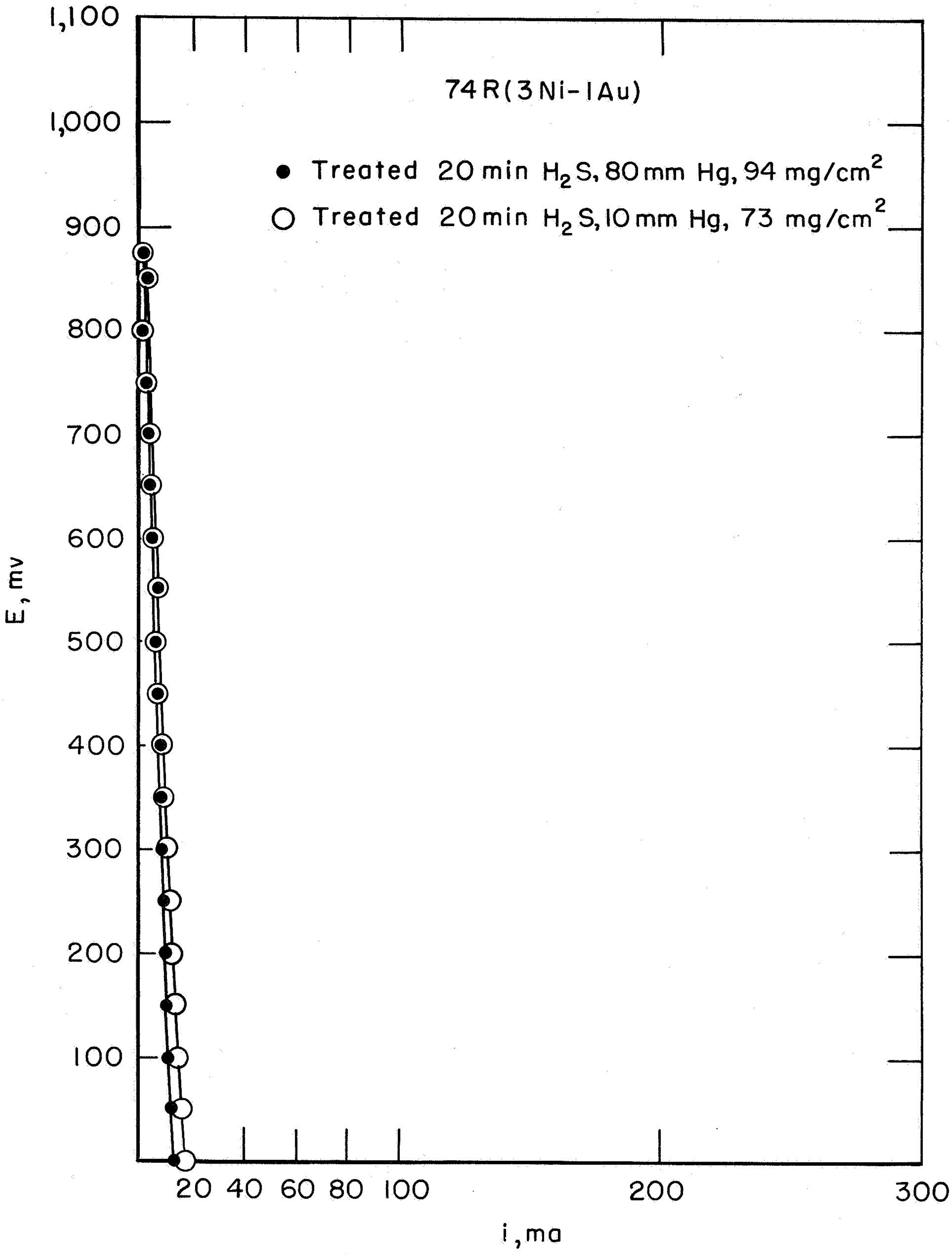

Figure 8.—i-E curve for 3 Ni-1Au treated with H_2S.

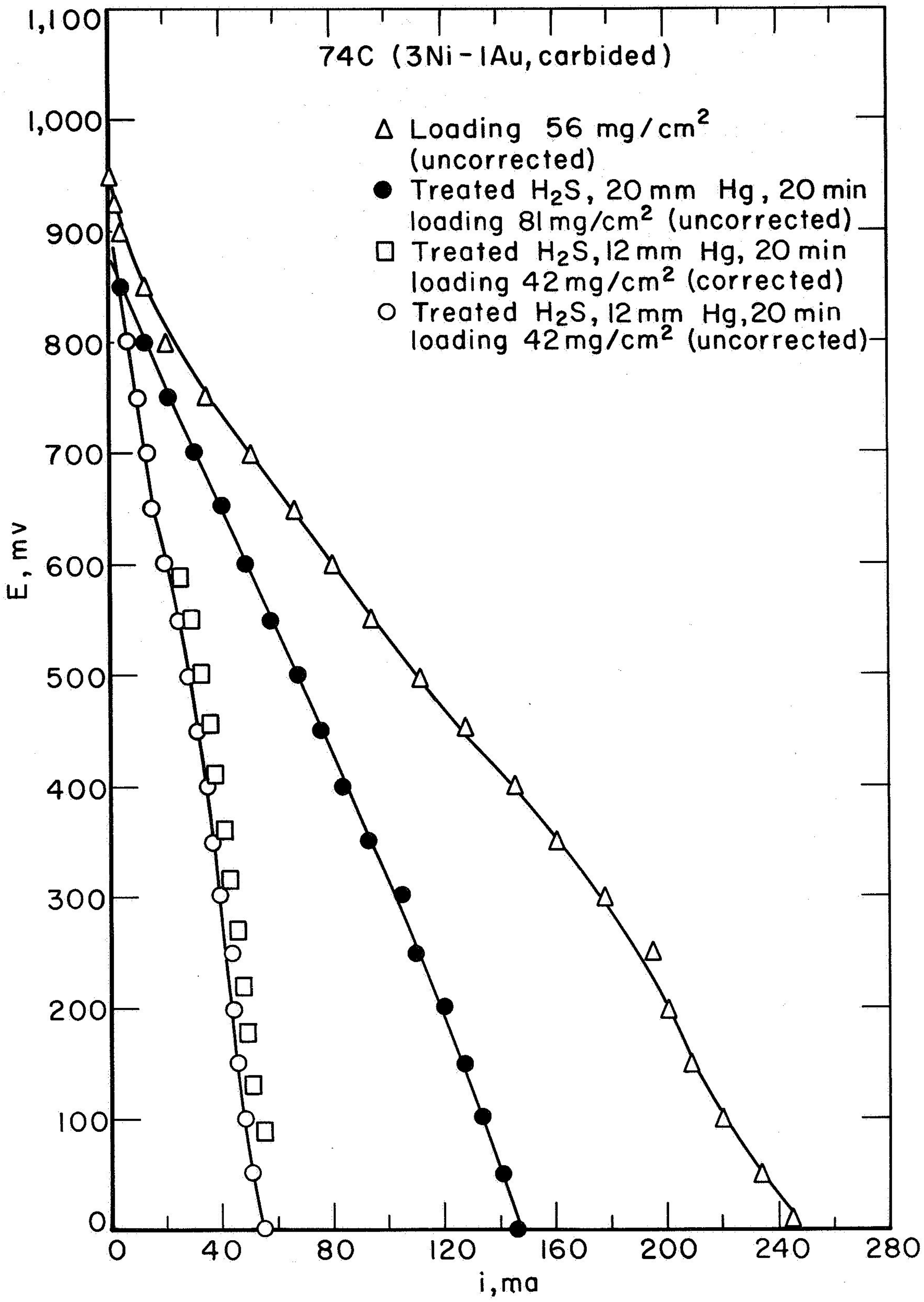

Figure 9.— i-E curve for 3Ni - 1Au, carbided.

APPENDIX I

Samples shipped to Tyco Laboratories this quarter ------------ 26

Samples shipped to Tyco Laboratories to date ---------------- 205

Samples shipped to non-cost users this quarter -------------- none

Samples shipped to non-cost users to date ------------------- 337

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Globe-Union, Inc.
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Attn: J.D. Onderdonk,
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Galton Industries, Inc.
212 Durham Ave.
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Ionics, Incorporated
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Lockheed Missiles & Space Co.
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LTV Research Center
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McDonnell Aircraft Corporation
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Midwest Research Institute
425 Volker Boulevard
Kansas City, Missouri 64110
Attn: Physical Science Library

Monsanto Research Corporation
Boston Laboratories
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Monsanto Research Corporation
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North American Aviation Co.
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Rocketdyne
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Attn: Library, Dept. 086-306-Zone 2

Speer Carbon Company
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