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QUARTERLY STATUS REPORT NO. 9

1 January - 31 March 1965

ELECTROCHEMICAL STUDIES IN THE SYNTHESIS
OF N-F COMPOUNDS

Contract No. Nonr-4054(00)

Research Project No. RR001-06-02

ARPA Order No. 399, Program Code No. 2910

June 30, 1965

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TRACOR, INC.

1701 Guadalupe St. Austin, Texas 78701

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Prepared by:

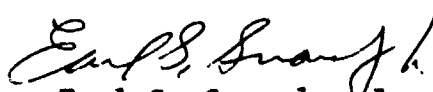
Larry G. Spears

Larry D. Fiel

Wallace E. Harrell, Jr.

Margaret H. Northrup

Approved by:



Earl S. Snavely, Jr.
Director of Chemical
Research

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Abstract

Work during the past quarter was devoted to the development of analytical methods for quantitative determination of N-F compounds and F_2 , and to the investigation of possible new electrode materials. Weight loss measurements, at open circuit and at constant anodic potential were performed on Incoloy 800, Inconel 600, and Ni-Span-C 902. Weight loss rates generally increased with increasing iron content and decreasing nickel content on these alloys. Weight loss measurements and polarization studies were performed on nickel in HF at various water concentrations. The addition of at least $10^{-2}\%$ H_2O reduced corrosion rates of nickel at 6.0 volts. The polarization curves showed a peak dependent on the water concentration on the initial run and a water-dependent plateau in the successive curves.

ELECTROCHEMICAL STUDIES IN THE SYNTHESIS OF N-F COMPOUNDS

I. INTRODUCTION

Anodic polarization curves of Monel in AHF solutions of NH_4F have not revealed any concentration-dependent plateaus which indicate a stepwise fluorination of the ammonium ion. This shows that the fluorination of ammonium ions proceeds by chemical reaction rather than by an electrochemical mechanism. However, the investigation is proceeding to determine if ions such as NH_3F^+ , NH_2F_2^+ , NHF_3^+ or NF_4^+ exist as intermediates in the fluorination of NH_3 in AHF. The possibility that NF_3 is oxidized at the anode to form the transient species $\cdot\text{NF}_3^+$ which may react with $\cdot\text{F}$ to form NF_4^+ is also being investigated.

At present, the electrochemical studies are being supplemented with IR spectrophotometric and gas chromatographic (GC) analysis to determine the relative contribution of current toward the formation of each species evolved at the anode (e.g., $\cdot\text{F}_2$, NF_3 , etc.). A quantitative analysis of each product formed is necessary to establish the mechanism for the fluorination of NH_3 in AHF.

Several new electrode materials were investigated during the past quarter in an attempt to find a better electrode material. Those investigated were: nickel, Inconel 600, Incoloy 800, and Ni-Span-C 902. Monel and nickel electrodes are currently being used in electrochemical fluorination studies as they have the best properties of the metals evaluated in past experiments.

II. EXPERIMENTAL APPARATUS

A gas and liquid sampling system has been installed to allow accurate sampling of the anodic and cathodic gases and the NH_4F -AHF solution during electrolysis (see Figure 1). A porous Teflon (TFE) cathode compartment has been installed to separate the anodic and cathodic gas products (see Figure 2). The electrode configuration in the electrolytic cell is shown in Figure 3.

The procedure for gas sampling is as follows:

- (1) All the 1/8" Monel tubing leading from the 1/8" Hoke needle valve, which connects to the anodic gas line, to and including the large Monel bomb, is evacuated.
- (2) The anode condenser valve is closed and the cathode condenser valve opened. (If the cathode gas was being analyzed the reverse would be done.)
- (3) The electrolysis is started. After periods of approximately five minutes each, the needle valve is opened slightly to allow the cell pressure to decrease back to atmospheric pressure and thus collect the anodic gas products in the large bomb. The Monel pressure gauge indicates the amount of sample collected. Once the desired amount of gas is collected the bomb is sealed off and the electrolysis stopped.
- (4) One or both of the smaller Monel bombs and/or the IR gas cell is then evacuated. Then a sample of the collected gas is introduced into

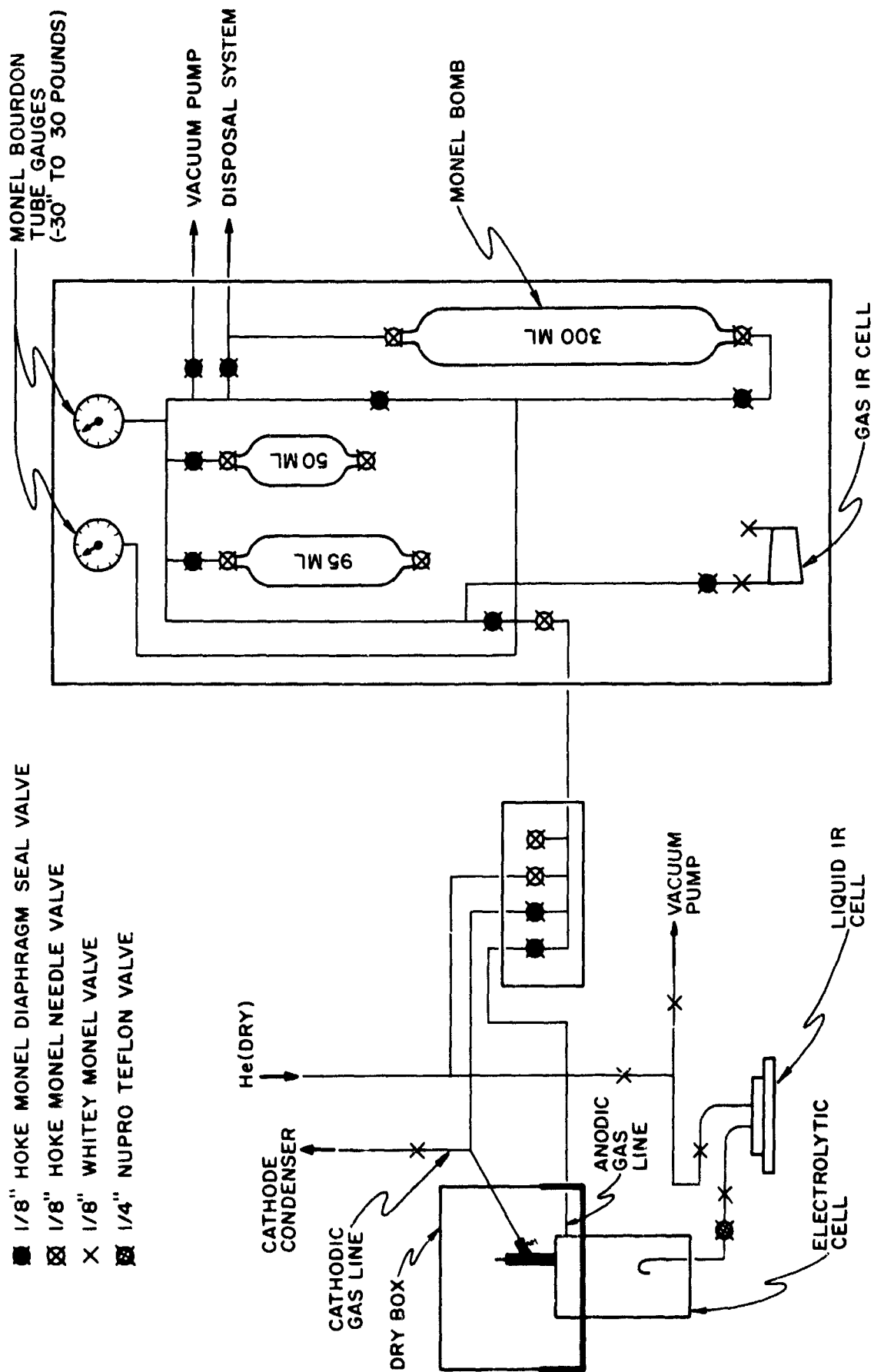


Fig I - GAS AND LIQUID SAMPLING SYSTEM

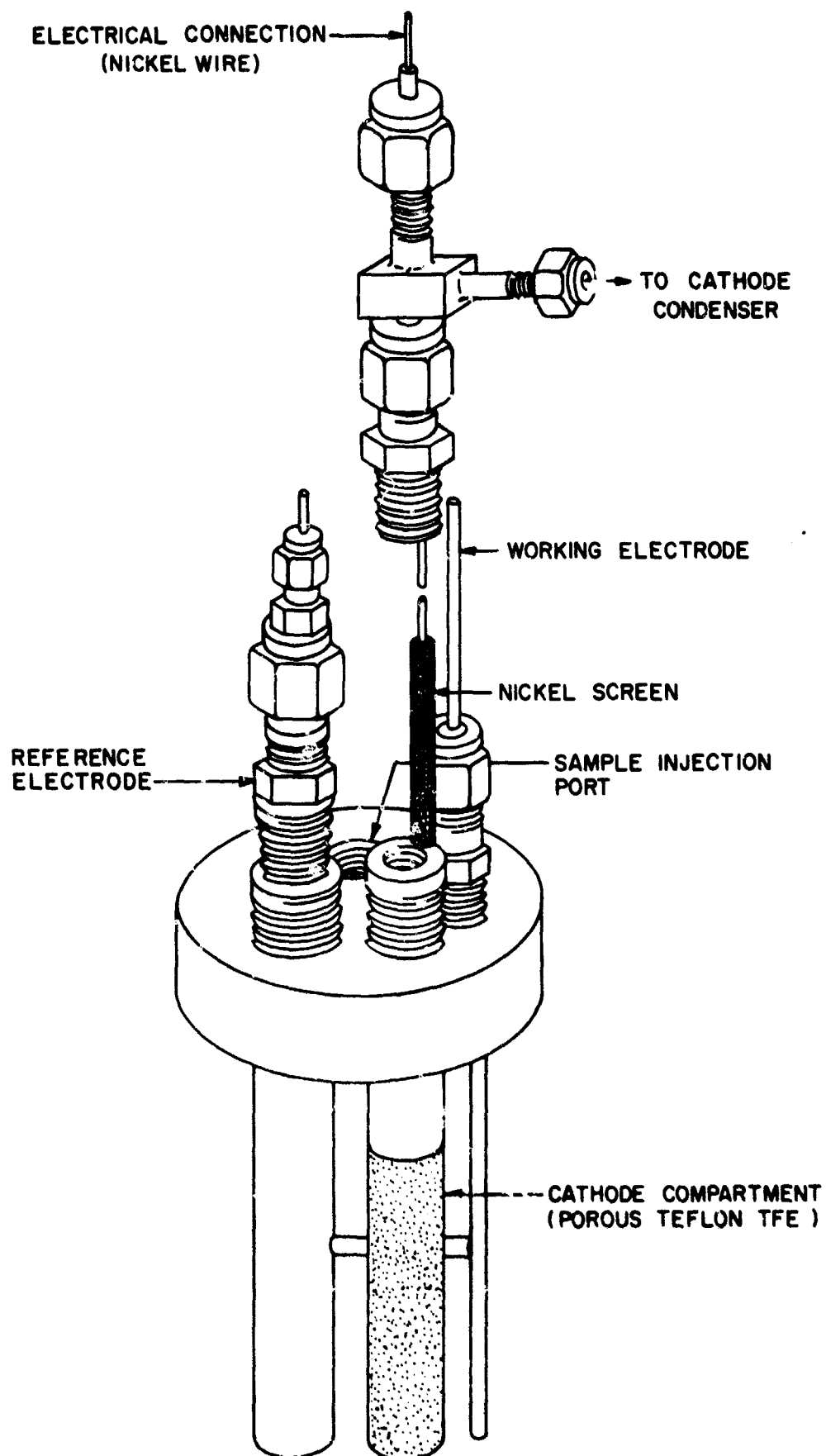


Fig. 2 - CATHODE COMPARTMENT MOUNTED IN
CELL LID

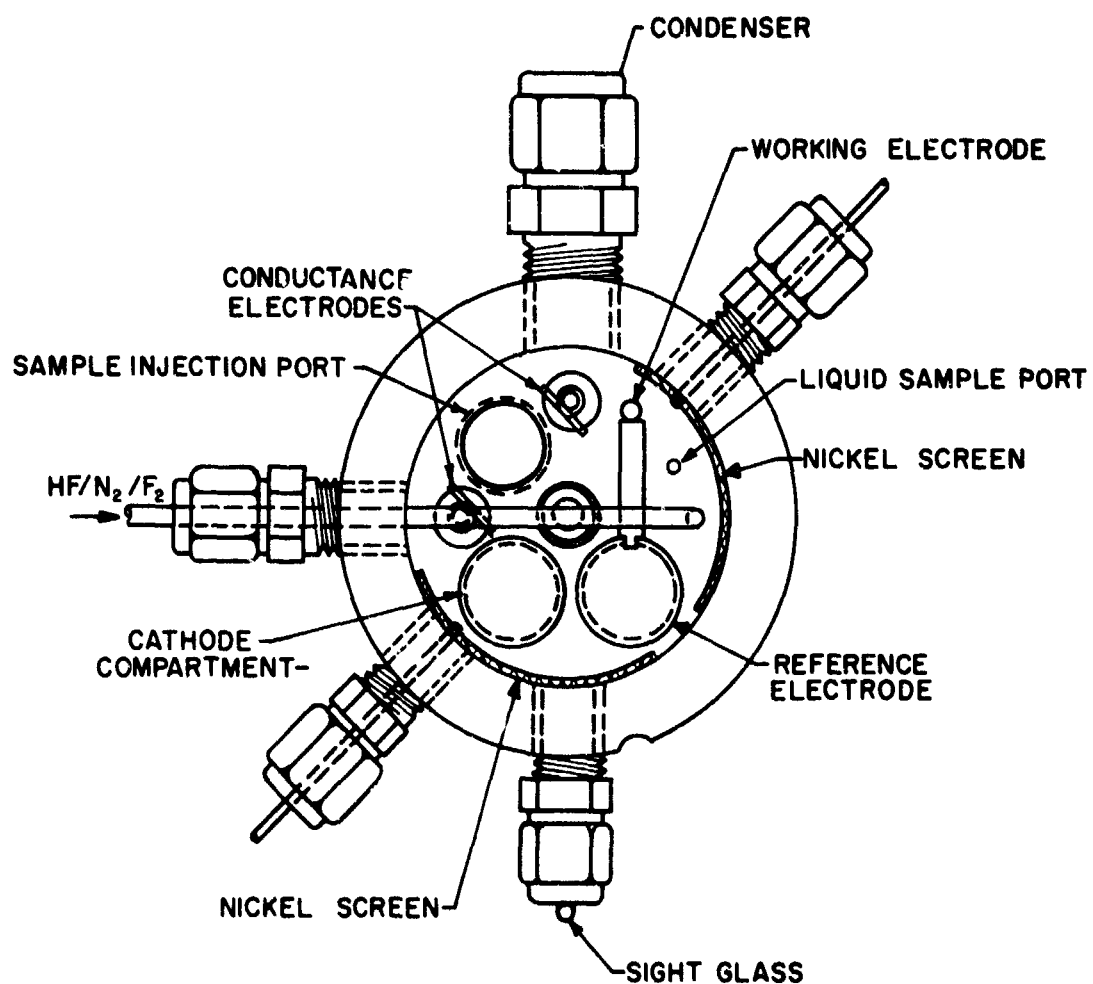


Fig. 3 - ELECTROLYTIC CELL ELECTRODE CONFIGURATION
TOP VIEW

the desired container for analysis. Samples from the smaller bombs are used for GC, NMR, and mass spectral analysis. Dry helium is used to pressurize any part of the sampling system or to flush out any part of the system.

The procedure for liquid sampling is as follows:

- (1) Dry helium is used to flush out the Kel-F liquid sample loop in the base of the cell.
- (2) A vacuum is pulled up to the 1/4" Nupro Teflon valve and then the 1/8" Whitey valve nearest the vacuum pump is closed.
- (3) Then the Teflon valve is opened to allow the liquid cell to fill. After several seconds the Teflon valve is closed and the other Whitey valve on the IR cell is closed.

A liquid IR cell was developed for use with liquid AHF. The cell is vacuum and pressure tight and should be sufficient for any low temperature studies.

III. ANALYSIS OF ELECTROLYSIS PRODUCTS

Much work during this past quarter has been devoted to developing IR and GC methods for analysis of the electrolysis products.

An attempt was made to analyze any electrolysis products remaining in solution after electrolyzing a 0.10M solution of NH_4F in AHF for four hours. This was not successful due to the reaction of the sample with the Kel-F grease which was needed to seal the commercial liquid cell used at that time. The Kel-F grease reacted to form a black residue. A new liquid IR cell has been developed in which the sample contacts only Irtran-2, Monel, and Teflon. This cell will be used at subambient temperatures in order to approximate the conditions in the electrolytic cell. A more accurate description of this cell will be given in the next quarterly report. An attempt was also made to analyze the anodic volatile products from this electrolysis. However, the Irtran-2 windows in the IR gas cell reacted to give a complicated spectrum between 800 to 2500 cm^{-1} . Due to the lack of a better gas cell window material, it will be necessary to analyze the volatile electrolysis products at subambient temperatures. However, Irtran-2 does not react in liquid HF at -19°C . A highly polished piece of Irtran-2 was placed in AHF for 48 hours and showed no weight change or dullness. Results from the GC analysis of these products were also erroneous since the Kel-F oil on the shredded Teflon packing apparently reacted with the sample to give a very poor chromatogram. Peak separation for this column is also very poor.

Investigation of gas chromatographic methods for determining F_2 and N-F compounds has been concentrated in two areas. The first is a conventional approach employing either a coated support, an uncoated active solid, or a liquid modified solid that would be

selective enough to separate components of interest and still remain unreactive to them. The second approach is to use sub-ambient distillation and thermal conductivity to detect the components. Due to the reactivity of F_2 towards Monel and stainless steel, it is necessary to passivate all the metal parts of the gas chromatograph that come in contact with the samples. Also, the normally used stainless steel detector block with tungsten filaments was replaced by a nickel detector block with Teflon-coated tungsten filaments. This was necessary since the uncoated filaments deteriorated rapidly when used with F_2 thus yielding irreproducible results. The Teflon coated filaments have shown no change after repeated exposure to fluorine and fluorine-containing compounds.

The selection of a good column packing proved to be a difficult task. Silica gel will separate air and NF_3 best, but experiments showed that F_2 reacts with silica gel, thus eliminating any quantitative measurements for F_2 if it is used as a column packing. Molecular sieves were also rejected as a column packing due to the reactivity between F_2 and metal oxides such as aluminum and magnesium oxide. Shredded Teflon (30-50 mesh) and Kel-F molding powder (30-50 mesh) were tried as supports while #10 and #40 Kel-F oils and #13-21 Halocarbon oil were investigated as liquid phases. Even when immersed in dry ice-acetone baths, these columns gave poor retention times and were not reproducible. These columns have also been rejected.

Reaction chromatography was also considered. An example of this is the quantitative analysis for F_2 . A fluorine-containing sample is injected into a precolumn filled with NaCl to form $NaF + Cl_2$. Then the chlorine is directed into a GC column for analysis. This was rejected because compounds such as N_2F_2 (a possible anodic product) would also react with NaCl.

Based on preliminary experiments, the best approach is subambient distillation or cryogenic programming to separate the volatile products and identifying them by gas chromatography and infrared spectroscopy. This method will also allow the determination of the boiling point for any unknown compounds. An F & M Chromatograph, Model 720, with a nickel block and Teflon coated filaments and a 0-1 mv strip-chart recorder with a one-second full-scale response is used for this method. A coiled, 16', 1/4" o.d. Monel tube (previously passivated with F_2) placed inside a large Dewar is used as a column and liquid nitrogen as a coolant. Helium, 99.95% pure, is used as the carrier gas. Before it enters the GC column it passes through a 10' x 1/4" o.d. copper tube filled with molecular sieves and then through a 10' x 1/4" o.d. copper tube immersed in a liquid nitrogen bath. Before a sample from one of the sample bombs is allowed to enter the sample loop, the loop is evacuated to at least 1 cm of Hg, and then the loop is pressured up to atmospheric pressure with the sample. Six minutes after the sample is injected into the column the liquid N_2 is drained through a valve in the bottom of the Dewar, and a heating rod located in the center of the coil (1000 watts at 35 volts) is turned on. Nitrogen gas is then allowed to flow through the valve in the bottom of the Dewar at a rate of 150 cc/min for uniform heating. Two Chromel-Alumel thermocouples, one in an ice bath and the other next to the GC column, and a student potentiometer are used to determine the change in temperature of the column. With the parameters given above, a linear heating rate of $4^\circ C/min$ from -196° to $20^\circ C$ is obtained. Data from this method and the chromatographic columns are given in Table I.

TABLE I
PERFORMANCE OF G. C. COLUMNS FOR N-F COMPOUNDS

COLUMN AND CONDITIONS	(THEORETICAL PLATES PER COLUMN) ON NF ₃	COLUMN EFFICIENCY HEIT (cm)	AIR	RETENTION TIMES, MIN.				COMMENTS
				F ₂	NF ₃	N ₂ F ₄	HF	
Column 1 16-ft. - 1/4" O.D., Monel tube, liq. N ₂ starting temperature, 50 cc/min. He flow. Program rate = ~ 3.2°C/min.	903	0.54	7.5	7.7	24.2	31	72.4	This column and para- meters still under development. Peaks are leading and carrier gas needs purifying.
Column 2 20-ft. - 1/4" O.D., Monel column packed with 50% Halocarbon Oil 13-21 on 30-50 mesh Kel-F molding powder. 65 cc/min. He flow. 40°C.	100	6.1	0.8	0.8	1.0	-	2.4	Not enough resolution between F ₂ and NF ₃ .
Column 3 10-ft. - 1/4" O.D., aluminum column packed with 30-60 mesh Silica gel, 60°C, 60 cc/min. He flow.	345	0.89	3.7	-	6.5	-	-	Peaks are well defined and sharp.
Column 4 50-ft. - 1/8" O.D., Kel-F tubing, packed with 50% Halocarbon Oil 13-21 on 30-50 mesh Kel-F molding powder, 45 cc/min. He flow. -72°C.	1410	1.1	7.0	7.0	7.5	-	-	Not enough resolution between F ₂ and NF ₃ .
Same as Column 4, ambient temp.	503	3.3	5.1	-	5.6	-	-	Sharp, Gaussian peaks.

IV. WORKING ELECTRODES

Work on inert anode materials was concentrated on nickel alloys since these possess the desirable characteristics (low corrosion rates in AHF and formation of a passive conducting film) of a good working electrode. In view of this, Inconel 600(1), Incoloy 800(1), Ni-Span-C 902(1), and nickel(2) were investigated during the past quarter for possible use as an anode material in AHF.

A. Nickel Alloys

Corrosion measurements were made on Inconel 600, Incoloy 800, and Ni-Span-C 902 in AHF at open circuit and at anodic bias. Weight loss rates are shown in Table II and the periodic weight losses are shown in Figure 4.

In comparing the three alloys, it is noted that corrosion rates generally increase with increasing iron content and with decreasing nickel content.(1) These alloys did not show sufficient promise for use as anodes in AHF to warrant further study, and future investigations of anode materials will be concentrated on nickel.

B. Nickel

Corrosion studies were conducted on nickel electrodes in HF with NaF added as a supporting electrolyte at various water concentrations. Increased resistance to attack in the presence of water was shown previously by Donohue and Nevitt (3). Open circuit weight loss measurements were made on nickel electrodes in $\text{HF} + 0.01\text{M NaF} + \text{H}_2\text{O}$. Water concentrations of $\leq 10^{-3}$ wt.% to 10^{-1} wt.% were used. Weight losses were low ($\leq 0.2 \text{ mg/cm}^2$) for all water concentrations for periods of time up to nine hours. The weight

TABLE II

WEIGHT LOSS RATES OF NICKEL ALLOYS AT OPEN CIRCUIT
AND AT CONSTANT-CURRENT ANODIC BIAS IN AHF

<u>Metal</u>	<u>Current Density</u> <u>($\mu\text{a}/\text{cm}^2$)</u>	<u>Time</u> <u>(hrs)</u>	<u>Weight Loss Rate</u> <u>($\text{mg}/\text{cm}^2/\text{hr}$)</u>
Inconel 600	open circuit	16	0.002
Inconel 600	40^{a}	24	0.013
Inconel 600	600^{b}	2	0.535
Incoloy 800	open circuit	16	0.005
Incoloy 800	40^{a}	24	0.028
Incoloy 800	600^{b}	2	0.610
Ni-Span-C 902	open circuit	16	0.006
Ni-Span-C 902	40^{a}	24	0.046
Ni-Span-C 902	600^{b}	2	0.780

a -- Below Fluorine evolution potential

b -- Above Fluorine evolution potential

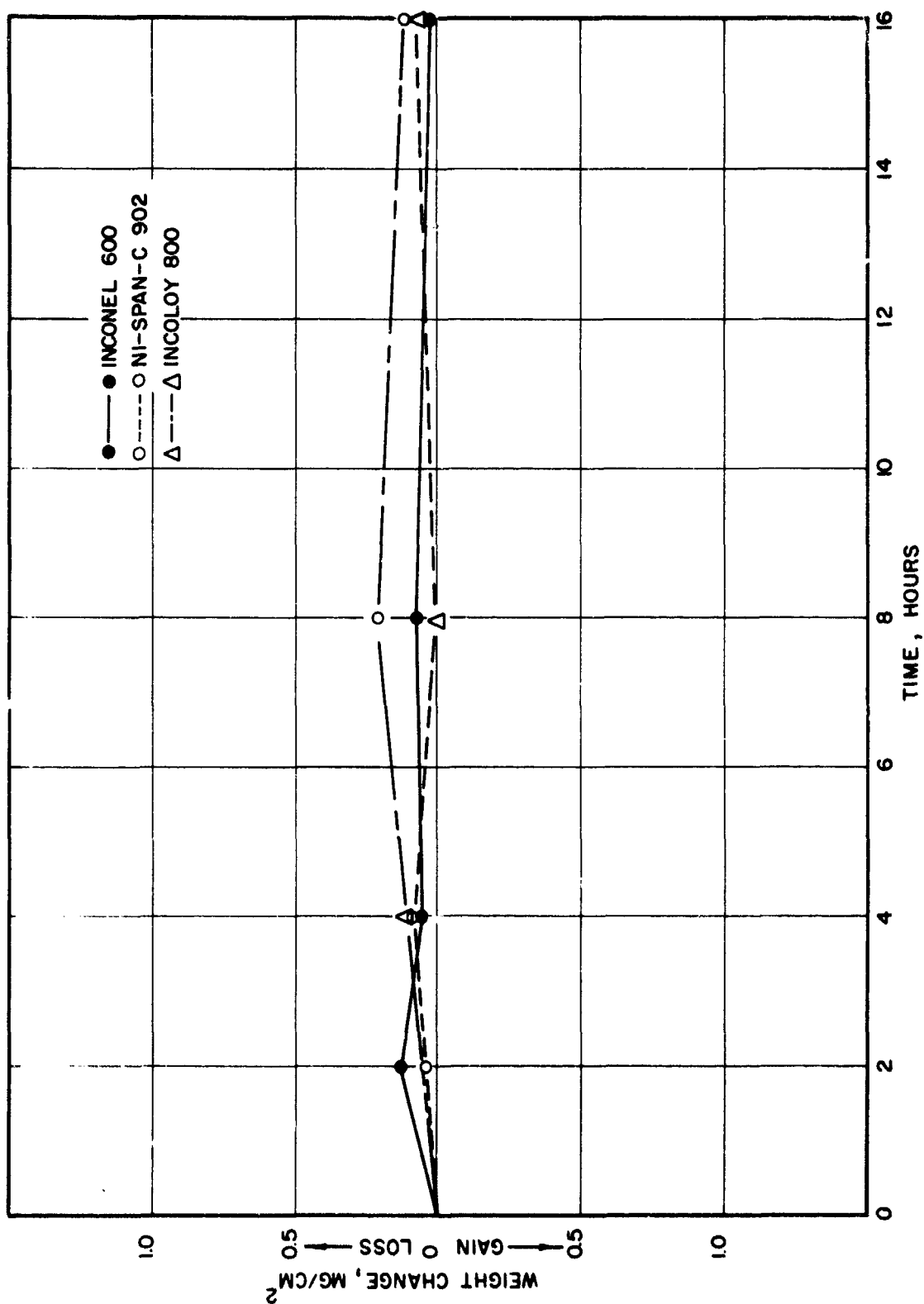


Fig. 4- WEIGHT CHANGES OF NICKEL ALLOYS IN AHF AT OPEN CIRCUIT.

losses approach a constant value with time, indicating formation of a passive film, which protects the electrode from further corrosion.

Anodic weight losses at 1.0 v, 3.0 v and 6.0 v are tabulated in Table III. It is noted that the addition of a small amount of water, on the order of 10^{-2} wt%, causes a large decrease in the corrosion rate at 6.0 v (see Figure 5). This effect has been observed on Hastelloy-F in our laboratory (4) and on nickel by Donohue and Nevitt (3). Weight losses at 1.0 v and 3.0 v are small and show no significant changes with water concentration.

A change in the character of the film was noted with increasing water concentration, particularly at 6.0 v. In HF with $\leq 10^{-3}$ % H_2O , the film was thick and crusty, and weakly adhering. This film was light green in color and was easily washed off in hot water. Large weight losses were observed with this type of film. At higher water concentrations, a dark (gray-black), thin, tightly adhering film was formed on the electrode. This film was difficult to remove without attack on the nickel itself, which probably accounts for the discrepancies in the measured and calculated weight losses. A cleaning process using cathodic protection of the nickel is being developed, which should facilitate more accurate weight measurements.

Anodic polarization curves were run on nickel electrodes in HF + 0.012M NaF with water concentrations from $\leq 10^{-3}$ % to 10%. The initial polarization curves, begun after the electrode was in solution long enough for the open circuit potential to reach equilibrium, are shown in Figure 6. (It is noted that the open circuit potential is shifted cathodically as the water concentration is increased as indicated by the arrows.) A sharp spike appeared at 0.0 v after the first water addition. The intensity of the peak increased as the water concentration was increased.

TABLE III

WEIGHT LOSSES OF NICKEL IN HF + 0.012M NaF + H₂O

H ₂ O Concentration (wt%)	Potential (volts) vs Hg/Hg ₂ F ₂	Time (hrs)	Weight Loss (mg/cm ²)		
			Calc. from defilmed wt.	Calc. from Gain*	Calc. from Charge*
0.001	1.0	17	-	1.050	0.055
0.02	1.0	24	0.102	0.132	0.036
0.05	1.0	24	0.810	1.2	0.108
0.1	1.0	21	0.04	0.21	0.081
1.0	1.0	23	0.108	-	0.180
10.0	1.0	24	0.080	0.015	0.039
0.001	3.0	16	-	0.132	0.480
0.02	3.0	16	0.048	0.090	1.10
0.05	3.0	16	0.050	0.700	0.720
0.1	3.0	16	-	0.40	0.708
1.0	3.0	16	0.270	-	0.864
10.0	3.0	16	0.090	-	0.680
0.001	6.0	0.06	1.73	3.01	2.34
0.02	6.0	0.06	0.14	0.42	2.64
0.05	6.0	0.06	0.13	0.69	3.0
0.1	6.0	0.06	0.21	0.43	2.9
1.0	6.0	0.10	0.16	0.22	4.5
10.0	6.0	0.16	0.12	0.027	1.8

* Assuming formation of NiF₂

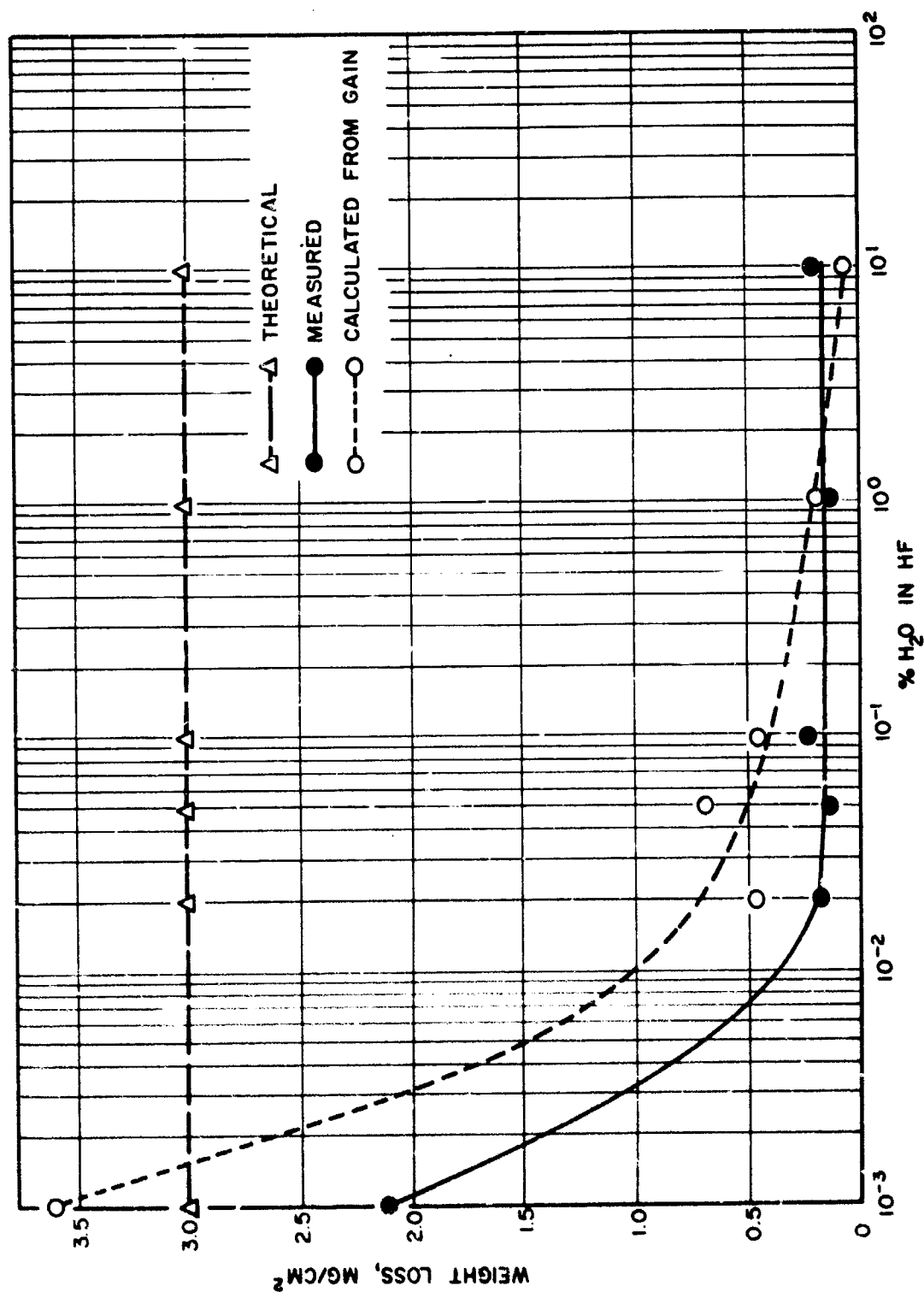


FIG. 5—WEIGHT LOSSES ON NICKEL IN HF + 0.012M NaF + H₂O AT 6.0 V

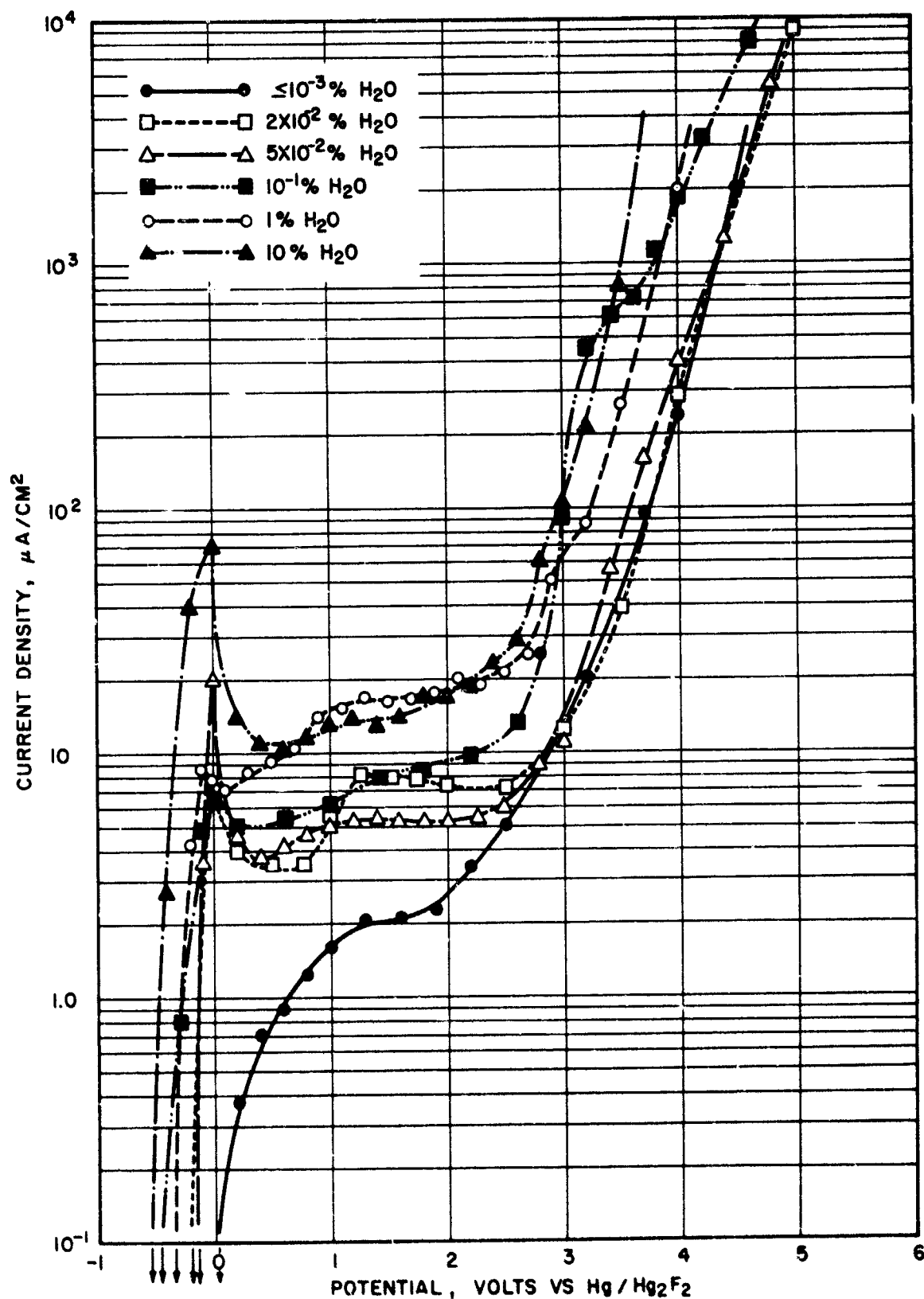


Fig.6-INITIAL POLARIZATION CURVES FOR NICKEL
IN HF + 0.01M NaF + H₂O

This peak has been observed on many metals in the course of our investigations and is probably related to the formation of a metal-metal fluoride film. Successive polarization curves on the same electrode (Figure 7) do not exhibit this peak, indicating that a passive film is formed on the initial polarization run. However, these curves do show an increase in the current density with water concentration in the potential range of 0.0 v to 3.0 v (see Figure 8). It has been ascertained by weight loss measurements that this increase is not due to metal dissolution.

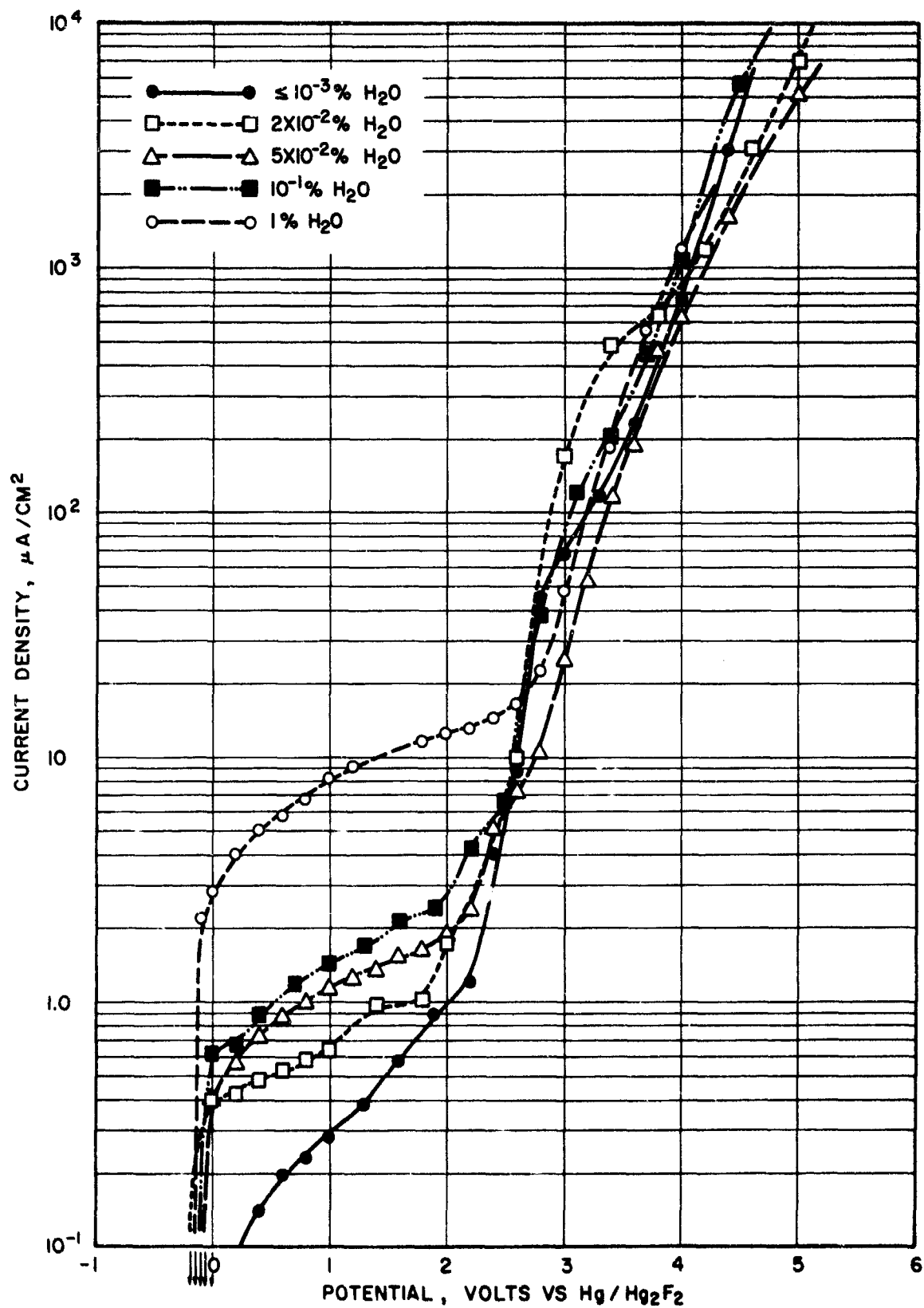


Fig. 7 - POLARIZATION CURVES FOR NICKEL
IN HF + 0.01M NaF + H₂O

V. DISCUSSION

Results to date indicate that it is necessary to perform the IR gas analysis at subambient temperatures and the IR liquid analysis at temperature below the boiling point of HF (19°C). Due to the lack of a suitable material to pack the GC columns, the volatile electrolysis products will be analyzed on the gas chromatograph using a 16-foot long, 1/4" diameter coiled Morel tube immersed in liquid nitrogen initially and cyrogenically programmed to the desired temperature.

The behavior of a nickel anode in HF is very much dependent on the water concentration of the solution. A passive behavior is observed when the water concentration reaches approximately $10^{-2}\%$.

VI. FUTURE WORK

The studies on the electrochemical fluorination of NH_4F at various anodic potentials and identifying the products formed by IR and GC analysis will be continued. Cryogenic programming will be used for the GC analysis. Cathodic stripping curves, galvanostatic polarization curves, and further weight loss studies will be performed on nickel in HF to determine the role of anode material during electrochemical fluorination.

REFERENCES

1. Huntington Alloy Products Division, The International Nickel Company, Inc., Huntington, West Virginia.
2. Nickel Analysis:

Cobalt	-	0.060%
Copper	-	0.006%
Iron	-	0.010%
Sulfur	-	0.015%
Carbon	-	0.008%
Nickel	-	99.901%
3. J. A. Donohue, and T. D. Nevitt, ARPA Propellant Contractor's Conference, Chicago, Illinois, April 13-15, 1964.
4. E. S. Snavely, Jr., et al, "Electrochemical Studies in the Synthesis of N-F Compounds," Quarterly Status Report No. 7, TRACOR, Inc. Document No. 65-103-U, dated January 8, 1965, p. 12.

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Fiel, Larry D.
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13. ABSTRACT

Work during the past quarter was devoted to the development of analytical methods for quantitative determination of N-F compounds and F_2 , and to the investigation of possible new electrode materials.

Weight loss measurements, at open circuit and at constant anodic potential were performed on Incoloy 800, Inconel 600, and Ni-Span-C 902. Weight loss rates generally increased with increasing iron content and decreasing nickel content on these alloys. Weight loss measurements and polarization studies were performed on nickel in HF at various water concentrations. The addition of at least $10^{-2}\%$ H_2O reduced corrosion rates of nickel at 6.0 volts. The polarization curves showed a peak dependent on the water concentration on the initial run and a water-dependent plateau in the successive curves.

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