

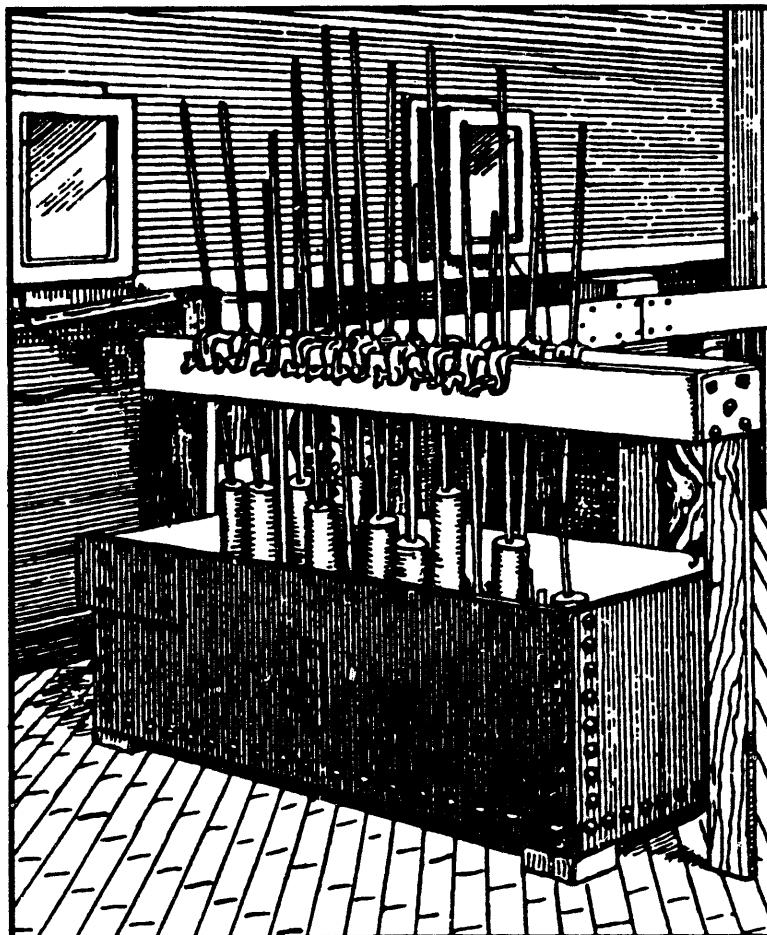
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Inert Electrodes Program

Test Plan for the Pilot Cell Test of Inert Anodes: Report on the June 1991 Meeting at the Reynolds Metals Company Facility



September 1991

Work Supported by the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
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On the cover:

Aluminum reduction pots at the Pittsburgh Reduction
Company's (Alcoa's) plant in 1889. Adapted from a
photograph, courtesy of Alcoa.

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Inert Electrodes Program

TEST PLAN FOR THE PILOT CELL TEST OF INERT ANODES:
REPORT ON THE JUNE 1991 MEETING AT THE REYNOLDS METALS
COMPANY FACILITY

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September 1991

Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

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MASTER

PREFACE

The Inert Electrodes Program at the Pacific Northwest Laboratory (PNL)^(a) is supported by the Office of Industrial Processes (OIP) of the U.S. Department of Energy (DOE) and is aimed at improving the energy efficiency of Hall-Heroult cells through the development of inert anodes. The inert anodes currently under study are composed of a cermet material of the general composition NiO-NiFe₂O₄-Cu. The program has three primary objectives: 1) evaluate the anode material in a pilot cell facility, 2) investigate the mechanisms of the electrochemical reactions at the anodes surface, and 3) develop sensors for monitoring various anode and/or electrolyte conditions. This report discusses a test plan that has been developed for the pilot cell test of the inert anodes.

(a) PNL is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

SUMMARY

A meeting was held at the Reynolds Metals Company (RMC) facility, Sheffield, Alabama, on June 24, 1991, to discuss the pilot cell test of the inert anodes. The outcome of that meeting included a detailed plan for performing the pilot cell test. In general, Pacific Northwest Laboratory (PNL) and RMC will work together in evaluating the performance of up to 13 inert anodes, tested six-at-a-time in a cluster arrangement, using the pilot cell at the RMC facility. During most of the 4-week test, the anodes will be operated at a target current density of 0.5 A/cm^2 and the cell will be run with alumina concentration at a target value close to saturation. Other operating conditions and activities are discussed in the body of the text. Following the test conducted jointly by PNL and RMC, Eltech Research Corporation (ERC), Fairport Harbor, Ohio, will proceed (also in conjunction with RMC) with their evaluation of the "Cerox" technology using the same pilot cell facility. No details regarding the operation of the pilot cell during the RMC test are given in this report.

ACKNOWLEDGMENTS

The authors acknowledge the many suggestions and comments by PNL staff including D. M. Strachan, L. R. Bunnell, L. G. Morgan, R. E. Westerman, E. N. Greenwell, N. D. Stice, N. C. Davis, and S. M. Faber; the RMC pilot cell staff; and the program consultants including W. E. Haupin. The authors are also grateful for the programmatic support by M. J. McMonigle and the Office of Industrial Processes of the U.S. Department of Energy.

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1.0 INTRODUCTION

The pilot cell test will be the first scaled-up evaluation of the cermet inert anodes being developed at Pacific Northwest Laboratory (PNL) using a self-heated electrolysis cell with the anodes in an arrangement similar to that being considered for their deployment in a retrofitted, industrial-scale aluminum reduction facility. The test is perceived as a necessary step toward commercialization. For the aluminum industry to accept the inert anode technology, the anodes must be shown to corrode or wear at acceptably low rates in a cell larger than those typically used for development work in the laboratory.

The pilot cell test will be conducted in two parts. In the first part, conducted jointly by PNL and the Reynolds Metals Company (RMC), the cermet inert anodes will be evaluated. In the second part of the test, Eltech Research Corporation (ERC) (also in conjunction with RMC) will proceed with their evaluation of the "Cerox" technology using the same pilot cell facility.

On June 24, 1991, a meeting was held between PNL staff (C. F. Windisch, Jr.) and RMC staff (T. Alcorn and A. T. Tabereaux) to discuss the details of the plan for conducting that part of the pilot cell test involving PNL. This report summarizes the results of that meeting. In particular, a test plan is given that will serve as a set of guidelines for performing the test. The report is intended to convey the basic objectives of the test and the primary activities that will take place. Most of the activities should occur as indicated, although it is cautioned that unforeseen events may require that these procedures be modified to some extent. No details regarding the operation of the pilot cell during the ERC test are discussed in this document.

2.0 PILOT CELL DESIGN AND OPERATING CONDITIONS

This section of the report covers the design of the pilot cell. In the first part of this section, the overall design of the pilot cell is discussed. In the second part, the cermet inert anodes are described.

2.1 PILOT CELL

The pilot cell at the RMC facility in Sheffield, Alabama, is a small, self-heated, aluminum reduction cell with the capacity for running two industrial-size carbon anodes. During the pilot cell evaluation of the inert anodes, an industrial-size carbon anode will be run in conjunction with a six-pack cluster of inert anodes.

The design of the pilot cell and the arrangement of the carbon anode and the inert anode cluster are shown in Figures 1 and 2. The carbon anode has a rectangular bottom with dimensions of 15.5 in. x 21.0 in. and is identical to those used in typical commercial reduction facilities. The inert anode cluster consists of six cermet inert anodes in a 2-by-3 arrangement situated about 4.5 in. from the carbon anode. (Details on the inert anodes are given in Section 2.2.)

The electrical bus allows individual control of current to the carbon anode and the inert anode cluster. Current through each of the inert anodes cannot be controlled separately, but will be monitored (through each anode) throughout the test using six 500 A:100 mV current transducers. Current through the carbon anode will be monitored using a 6000 A:100 mV transducer. It is anticipated that about 3000 A of current will be used for the carbon anode and about 100 A of current for each of the six inert anodes.^(a) These currents will be maintained

(a) The target current density for the inert anodes is 0.5 A/cm^2 . The surface area of each anode that is exposed to the electrolyte, given the planned 1.5-in. submersion depth (and accounting for the curvature shown in Figure 3), is about 200 cm^2 . Assuming that the current is uniformly distributed over the surface of an anode, 100 A per anode are required to give the target 0.5 A/cm^2 . Non-uniformity in current distribution will, of course, affect the current density (both the average value and the values at different locations on the surface). Voltage profiling will be attempted (see Section 4.2) early in the pilot cell test, using voltage probes, to determine the current distribution more accurately. The current through the inert anodes may be adjusted to compensate for irregularities, pending the outcome of these measurements.

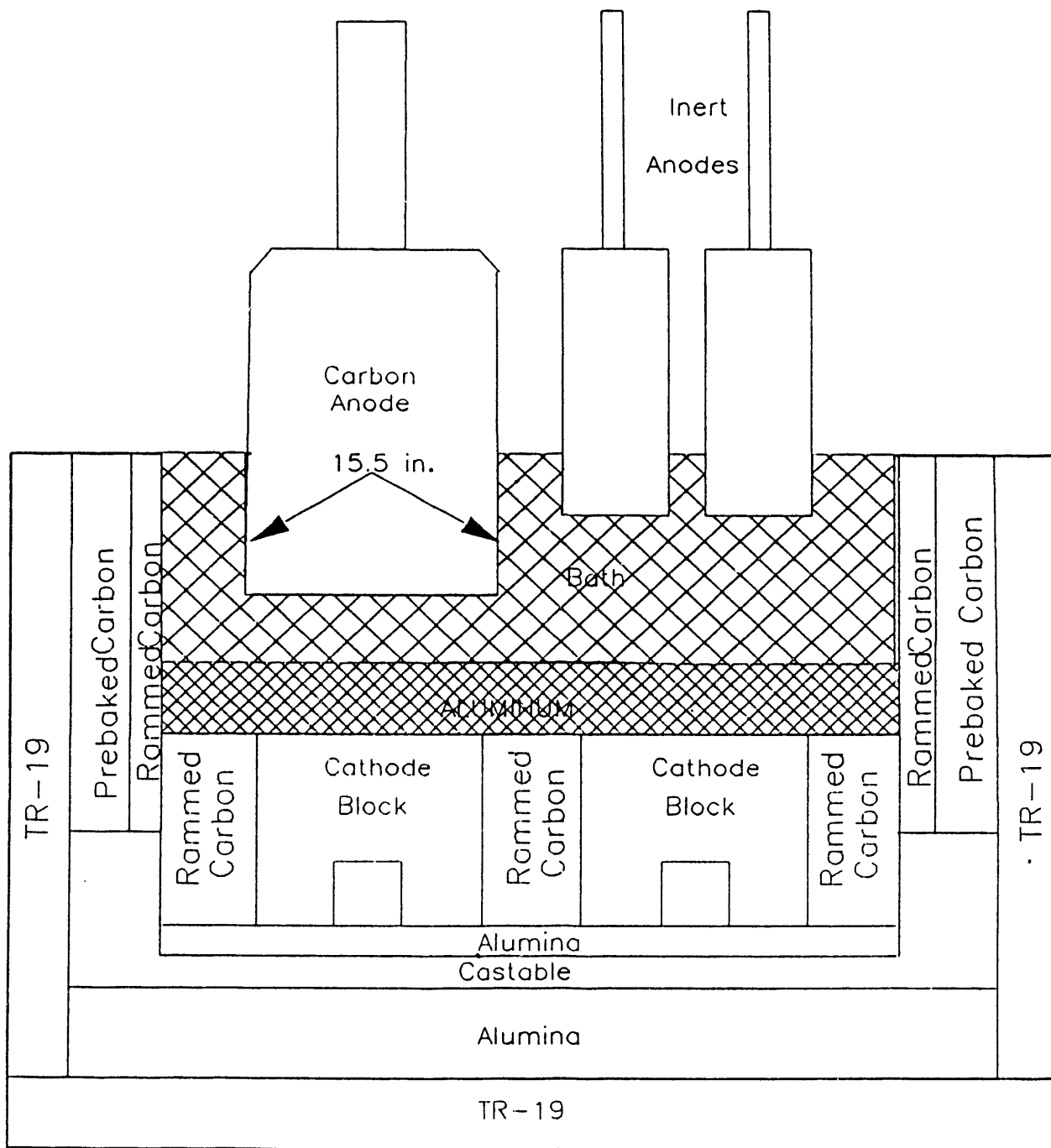


FIGURE 1. Side View of the Pilot Cell

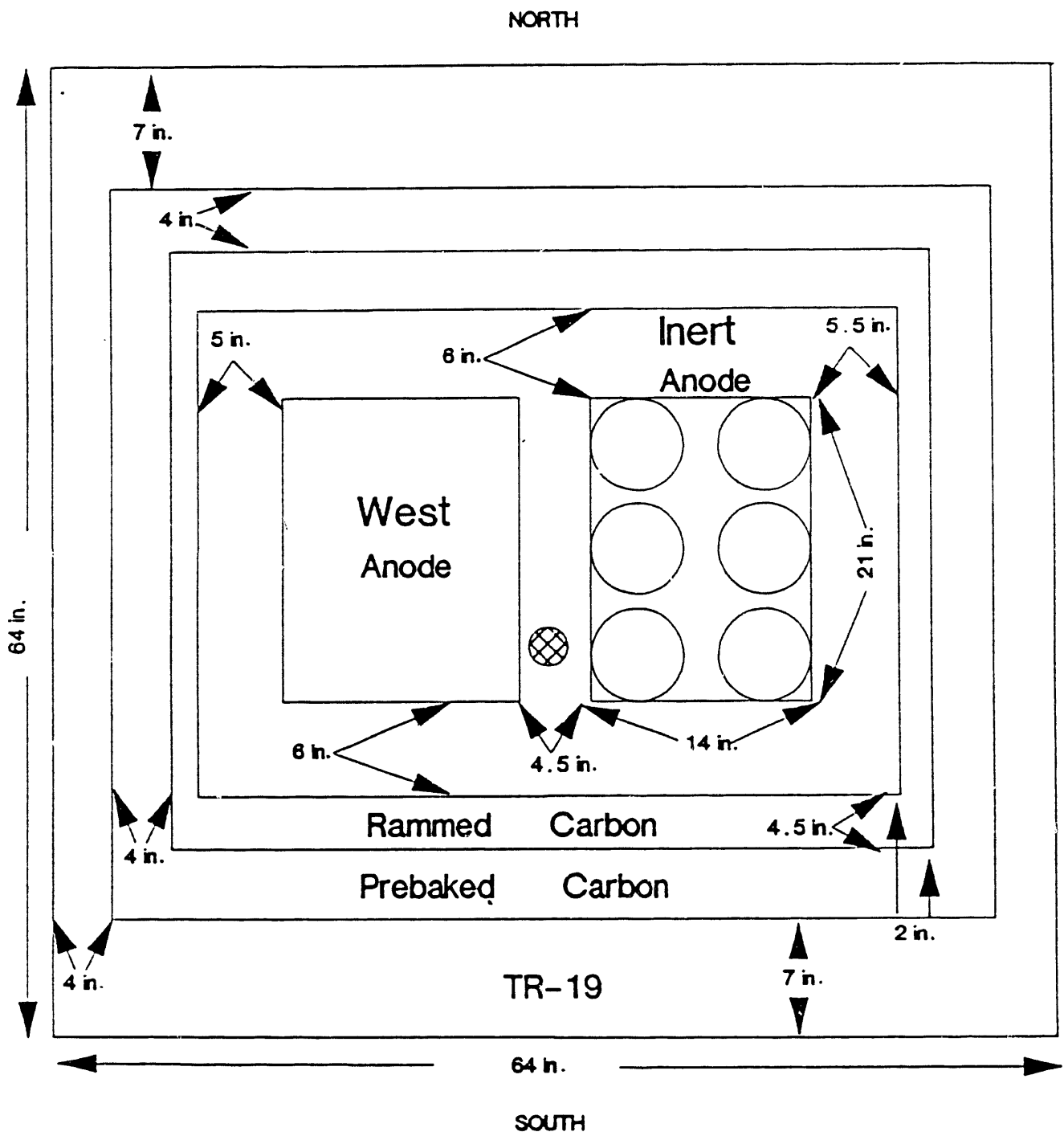


FIGURE 2. Top View of the Pilot Cell

throughout the pilot cell test (except when volt/amp curves are obtained or when anodes are exchanged).

The carbon anode will be immersed to a depth of about 6 in. into the bath. It is planned to insert the inert anodes to a depth of about 1.5 in. With a bath depth of about 8 in. and a metal depth of about 3 in., this will give anode-to-cathode distances (ACD) of 2 in. for the carbon anode and 6 in. for the inert anodes.

The electrolyte will have the following composition:

Bath Ratio:	1.35 +/- 0.05
CaF ₂ :	4 - 6 wt%
MgF ₂ :	< 0.5 wt%
LiF:	< 0.5 wt%
Al ₂ O ₃ :	6 - 7 wt%(a)

Chemical additions will be made to the bath to keep the concentrations as constant as possible during the test. Alumina, in particular, will be fed on a routine basis (estimated 0.25 lb. "dump" every 3 min.) using an automated alumina feeder (whose position is marked as the cross-hatched circle in the center of Figure 2).

The temperature of the bath will be maintained between 970° and 975°C throughout the test by manually adjusting the power input through the carbon anode (by varying current and ACD). During the last three days of the PNL portion of the test, alumina concentration will be varied deliberately in an attempt to collect sensors data (see Section 5.0) at different alumina concentrations, and to see if significant variation from saturation has any serious effect on the inert anodes over the three-day duration.

(a) This is the target concentration range for alumina. One of the requirements of this test is to perform the evaluation at alumina saturation, which is between 6 and 7 wt%, based on estimation by RMC for the pilot cell conditions.

2.2 INERT ANODES

The cermet inert anodes of the type NiO-NiFe₂O₄-Cu were made by Ceramic Magnetics Inc. (CMI), Fairfield, New Jersey, from spray-dried oxide powder (synthesized from 51.7 wt% NiO and 48.3 wt% Fe₂O₃) and Cu metal at 17 wt% of the cermet product, using methods similar to those reported previously (Hart et al. 1987).^(a)

The cermet inert anodes were designed as shown in Figure 3. The anodes are roughly cylindrical with a radius of about 3 in. and a height of about 3 in., with an additional 1-in. lip extending above the top face of the cylinder. The bottom edge of the anodes were rounded with a radius of curvature equal to 1.5 in. The upper edges of the anodes (on both sides of the lip) were also rounded with a radius of curvature of 0.5 in. An 18-in.-long, 1-in.-diameter Ni connector rod is to be screwed into the center of each cermet inert anode. The cermet was threaded directly (no separate core material was used) to accommodate the Ni rod.^(b) A 1.25-in.-diameter alumina tube (of a length to be determined by the proximity of the anode cluster superstructure) will be placed over the Ni rod to minimize exposure of the Ni to the bath vapors. Additionally, alumina (possibly mixed with cryolite) will be placed on top of the inert anodes to protect the anode-to-Ni rod connection.

Thirteen cermet inert anodes were to be made by CMI for testing by PNL and RMC. (An additional thirteen anodes were to be fabricated for use by ERC.) The anodes will be "pre-evaluated" at PNL before the pilot cell test by

-
- (a) Differences between the procedures in Hart et al. (1987) and those used by CMI will be discussed in the final report on the pilot cell test scheduled for publication in FY 1992.
- (b) Alternative approaches using a high-alloy core (50 wt% Cu-Ni alloy) in the cermet were also tested. The high-alloy core was advantageous in that it gave a good electrical connection and was easily threaded to accommodate the Ni rod. Cracks formed in these anodes during sintering, however, so the design was abandoned at this stage of the research. Because of its superior electrical and machining properties, it is recommended that the high-alloy core approach be reevaluated at a later date with additional scale-up development work to reduce cracking. Insufficient time and funds were available to fix the cracking problem at this time. A summary of the results of the high-alloy core anodes will be given in the final report on the pilot cell test that will be published in FY 1992.

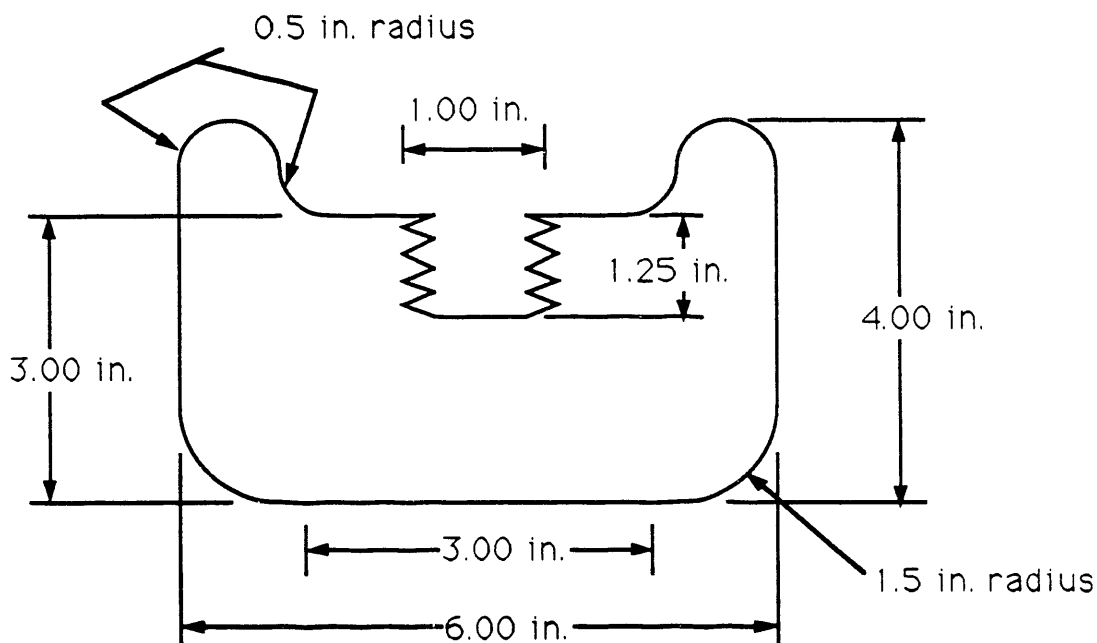


FIGURE 3. Schematic of a Cermet Inert Anode

using nondestructive methods to look for cracks and by making careful geometric measurements that will be compared with post-test geometries (for possible quantification of anode wear). These anodes will be tested in the pilot cell using the schedule described in Section 3.0.

One of the important characteristics of the cell operation will be the voltage drops through the various parts of the cell. To measure the voltage drop across the connection between the cermet anode and the Ni connector rod, a separated lead wire will be connected to the first six inert anodes that will be tested. This Pt lead wire will be connected to the anodes following a procedure similar to the one used in the prototype anode test (Strachan et al. 1990). The wire will be channeled up through the alumina protection tube and inside of a smaller-diameter alumina tube to insulate it from the Ni connector rod. For additional voltage profiling, a voltage tap will be installed at the top of the Ni rod, and, for one anode, at the bottom of the Ni rod also. (A more complete description of the approach to voltage profiling is given in Section 4.2.)

Thermocouples will be inserted between the Ni rod and the alumina sheath for each inert anode to sense the temperature right at the anode surface. These thermocouples will be K-type and sheathed with Hastelloy. Anode surface temperatures will be measured periodically with these thermocouples during both heatup and electrolysis testing as described in Section 4.1.

3.0 PILOT CELL SCHEDULE

This section of the report discusses the series of critical operations (other than sampling and measurement) that will take place during the pilot cell test and the schedule for performing them. The operations are the following: 1) startup of the pilot cell, 2) introduction of the first six inert anodes, 3) exchange of the inert anodes during the test, 4) a three-day period at the end of the test when alumina concentration is varied, and 5) transition to testing by ERC. The diagram shown in Figure 4 summarizes the various critical operations to be performed during the pilot cell test.

3.1 CELL STARTUP

Startup of the pilot cell goes through several phases prior to installation of the inert anodes. These phases are the following:

1. A 24-h gas-bake period in which the cathode is heated to approximately 700°C and aluminum metal is melted in the cell.
2. A 24-h period in which two full-size carbon anodes are shorted to the metal pad. This serves to further heat the cathode to approximately 850°C.
3. Following initial heatup, molten cryolitic bath is created in the cell by placing solid cryolitic bath around the carbon anodes and adjusting the anodes to arcing conditions. Within a 4-h period, 4 to 6 in. of molten bath will be created.
4. The cell will then be operated for 2 to 4 days with two full-size carbon anodes to achieve chemical and thermal stability.
5. One carbon anode will then be replaced with a six-pack cluster of small carbon anodes with dimensions similar to the inert anodes. The cell will then be operated for 2 to 4 days with the carbon anodes in order to achieve stability with the six-pack arrangement before the inert anodes are inserted.

3.2 INTRODUCTION OF FIRST SIX INERT ANODES

Once the cell has been started up and stabilized (after about 6 days), the six small carbon anodes in the cluster will be replaced, one-at-a-time, with six cermet inert anodes. The six inert anodes will have Ni connector

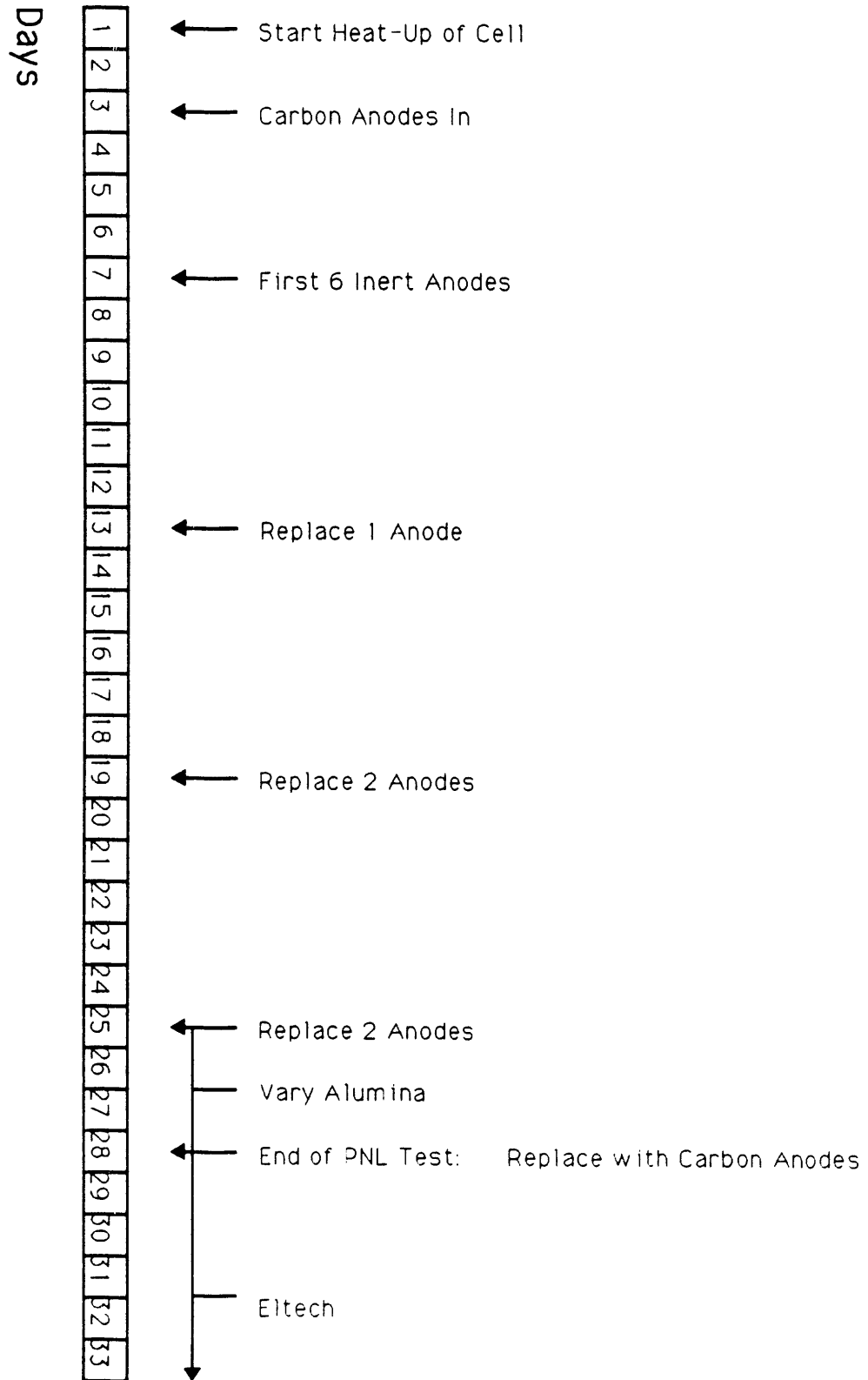


FIGURE 4. Schedule for the Pilot Cell Test

rods inserted into them (protected by an appropriate length of alumina tubing); and voltage taps will be secured to the top of the Ni rod, the bottom of the Ni rod (in one case), and directly to the anode body (see Section 4.2). The anodes will be preheated in an electric furnace at a temperature between 100°C and 300°C to remove moisture and help reduce thermal shock when the anodes are inserted into the cell. (They will be heated to this temperature at a rate of about 50°C/h.)

After preheating, the inert anodes will be moved, one-at-a-time in sets of two, using the following procedure. Current to one of the small carbon anodes will be shut off. The small carbon anode will then be removed and the space made available for an inert anode. In particular, the clamps and fittings will be made ready for the inert anode and the crust removed from the bath in the vicinity of the anode. One of the inert anodes will then be removed from the preheat furnace, encased in insulation, and moved by hand over to the pilot cell. The inert anode will then be attached to the anode cluster and positioned so the anode is above the bath. Temperature measurements at the anode surface will be made. The inert anode will then be lowered slowly into the bath (while trying to keep the crust from forming) using the thermocouple readings to gauge the rate of lowering. While this anode is being lowered, a second inert anode will be transferred using the same procedure. Once the two inert anodes are inserted to the desired depth (about 1.5 in.), the current to them will be turned on. Their operation will be observed. If no problems are encountered, the rest of the inert anodes will be inserted using the same procedure. It is estimated that the entire replacement procedure may take at least one full working day.

3.3 EXCHANGE OF INERT ANODES

As indicated in the schedule in Figure 4, it is planned to replace the inert anodes three times during the test, at six-day intervals. The test conditions for the thirteen anodes will be as given in Table 1. The anode positions are labeled A through F as shown in the diagram in Figure 5. The inert anodes, in turn, are labeled according to the position they are in, and indexed by the number exchanged in that position so far. (For example, anode A₂ is the second anode to occupy the position A in the anode cluster as shown in Figure 5.)

TABLE 1. Test Conditions for the Inert Anodes

<u>Anode</u>	<u>Day Inserted</u>	<u>Anode Replaced</u>	<u>Days in Cell</u>	<u>Conditions of Anode</u>
A ₁	7	Carbon	12	100 A; 6-7 wt% Al ₂ O ₃ (Always)
B ₁	7	Carbon	21	100 A; 6-7 wt% Al ₂ O ₃ (18 days), Varied Al ₂ O ₃ (last 3 days)
C ₁	7	Carbon	18	100 A; 6-7 wt% Al ₂ O ₃ (Always)
D ₁	7	Carbon	6	100 A; 6-7 wt% Al ₂ O ₃ (Always)
E ₁	7	Carbon	18	100 A; 6-7 wt% Al ₂ O ₃ (Always)
F ₁	7	Carbon	12	100 A; 6-7 wt% Al ₂ O ₃ (Always)
D ₂	13	D ₁	15	100 A; 6-7 wt% Al ₂ O ₃ (12 days), Varied Al ₂ O ₃ (last 3 days)
A ₂	19	A ₁	9	100 A; 6-7 wt% Al ₂ O ₃ (6 days), Varied Al ₂ O ₃ (last 3 days)
F ₂	19	F ₁	9	100 A; 6-7 wt% Al ₂ O ₃ (6 days), Varied Al ₂ O ₃ (last 3 days)
C ₂	25	C ₁	3	100 A; Varied Al ₂ O ₃ (3 days)
E ₂	25	E ₁	3	100 A; Varied Al ₂ O ₃ (3 days)
G, H				Extra Anodes

Some of the reasons used to develop the above anode exchange scheme include the following: 1) An anode in position B is the most difficult to exchange. Therefore, only one exchange in position B will be performed, i.e. at the end of the test. 2) An anode in position D is one of the easiest to exchange. Therefore, an anode in position D will be exchanged first. 3) Two anodes will be removed on days 19 and 25, since these are toward the end of the test at the constant conditions of 100 A/anode and 6 to 7 wt% alumina. Results obtained for day 25, in particular, will be critical in drawing conclusions on long-term anode performance.

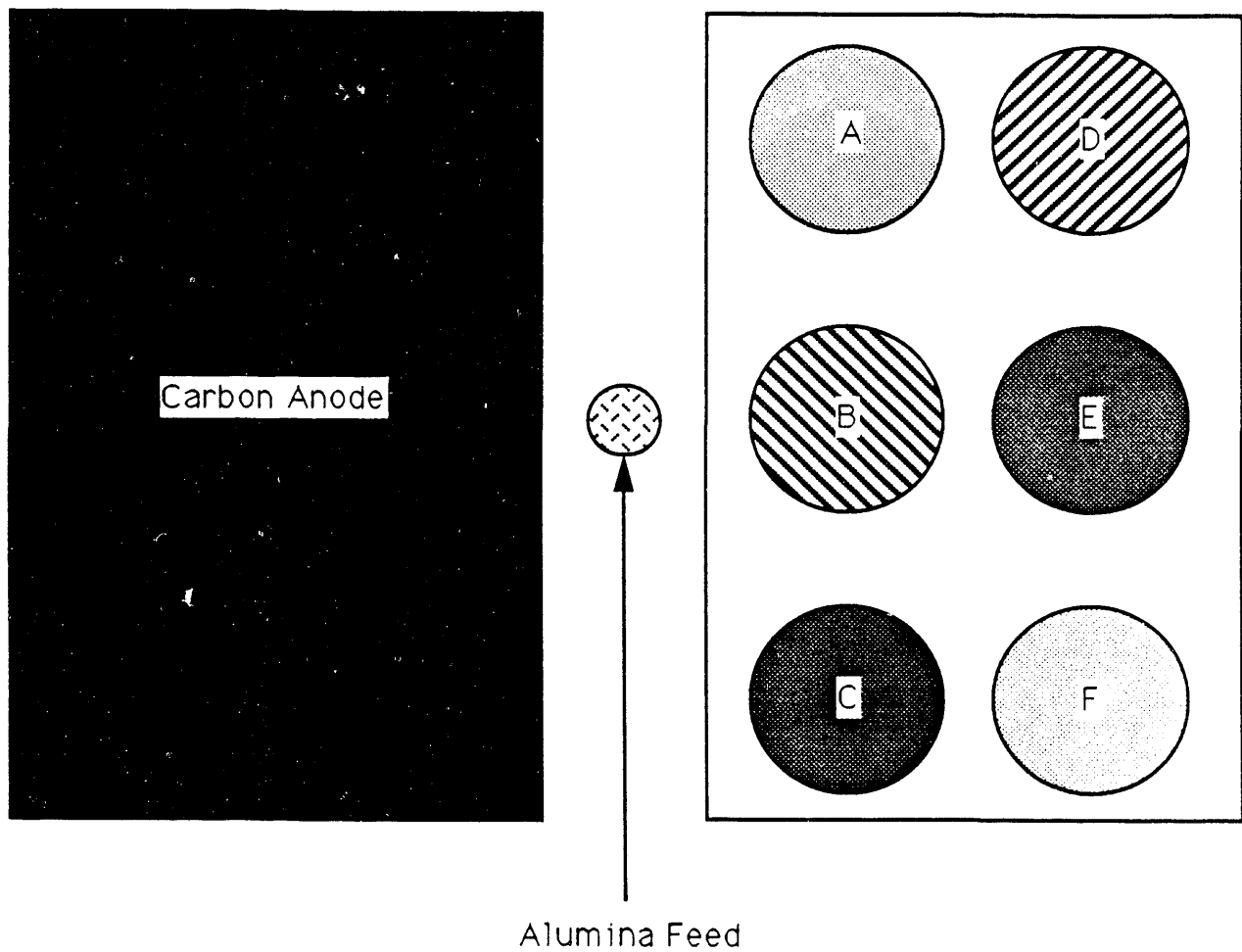


FIGURE 5. Pilot Cell Anode Exchange Scheme

In addition to the position of the anodes indicated in Figure 5, the orientation of the various anodes will be recorded. It is possible that uneven wear may result, giving information on the effects of non-uniform alumina concentrations and/or bath dynamics.

The procedure for exchanging the inert anodes will be similar to that used earlier for replacing the carbon anodes with the inert anodes. The only difference will be that special care will have to be used in removing the inert anodes. In particular, thermal shock should be minimized if possible; otherwise, it is expected that the anodes will crack severely during cool down. While cracking during cool down is not detrimental to the success of the inert anodes,

it will preclude demonstration of an intact tested anode. The plan is to remove the inert anodes slowly, wrap them in insulation, and then let them cool in air.

3.4 PERIOD OF VARIED ALUMINA

During the last three days of the test, attempts will be made to vary alumina concentration. The cell will be deliberately starved of alumina in an attempt to operate it at concentration significantly below saturation. Alumina will be measured directly (see Section 4.0) and sensor measurements will be made (see Section 5.0). At the end of this three-day period, the six inert anodes will be replaced with carbon anodes.

3.5 ERC TESTING

On day 28, the six inert anodes will be replaced with carbon anodes, and over the next few days the cell will be "cleaned up" in preparation for the testing by ERC. Details of the ERC testing program will be provided by ERC; however, it is planned that PNL staff will be present during some part of the ERC test to provide assistance and collect additional data.

4.0 PILOT CELL MEASUREMENTS

This section of the report summarizes the types of measurements (other than measurements in support of the Sensors Development Program discussed in Section 5.0) and the measurement schedule that will be used during the pilot cell test. It also contains a discussion of a set of special measurements to profile the voltage drops throughout the cell and map the voltage drops over the surface of the six inert anodes.

4.1 DESCRIPTION OF MEASUREMENTS AND SCHEDULE

A list of the measurements (other than Sensors Development Program measurements discussed in Section 5.0) that will be made during the pilot cell test, the methods used, and the frequency of these measurements are give in Table 2.

TABLE 2. Pilot Cell Measurements

<u>Condition</u>	<u>Method</u>	<u>Frequency</u>
Temperatures		
Bath	Thermocouple: Insert manually.	Hourly
Anodes	One thermocouple on each anode.	Every 30 s during heatup. Every 10 min when hot.
Voltages	See Section 4.2.	See Section 4.2.
Currents		
Total to Cluster	Transducer on bus.	Every 30 s.
Each Anode	Transducers on each anode leg.	Every 30 s.
Checks	Clamp-on meter.	Once each shift.
Volt/Amp Curves	100 A; 80 A; 60 A; 40 A	Every other day.
ACD	Manual probes.	Twice a shift on a "reference" anode.

TABLE 2. (contd)

<u>Condition</u>	<u>Method</u>	<u>Frequency</u>
Bath Ratio	Pyrotitration.	Sampled twice a shift.
Alumina	Wet lab analysis.	Sampled twice a shift.
	Alumina probe.	Every 2 h.
Bath Composition ^(a)	ICP and AA (Mg,Li), titration (CA).	Sampled twice a shift.
Aluminum Composition	ICP or Quantometer.	Sampled twice a shift.

(a) Includes analysis for Fe, Ni, Cu, and Si.

The periodic sampling of bath and metal for bath ratio, alumina, and composition (although performed twice a shift) will not yield results (i.e. analysis will not be complete) for up to 24 h later. Consequently, in the case of alumina in particular (one of the most important parameters for the inert anodes), another, more immediate, approach will be used. An alumina probe (Tabereaux and Richards 1983) will be employed to obtain alumina concentrations every 2 h. This will permit adjusting alumina concentration to keep it sufficiently close to the target value 6 to 7 wt%.

Most of the measured values will be monitored and recorded by a datalogger supplied by RMC. These include the temperature measurements on the six inert anodes, the cell current and voltage, and the currents through the six inert anodes and the carbon anode.

Volt/amp curves and ACD measurements will be performed periodically as indicated.

4.2 VOLTAGE PROFILING

The various voltage drops through the pilot cell will be determined. A schematic of the positions for the voltage measurements is given in Figure 6. By

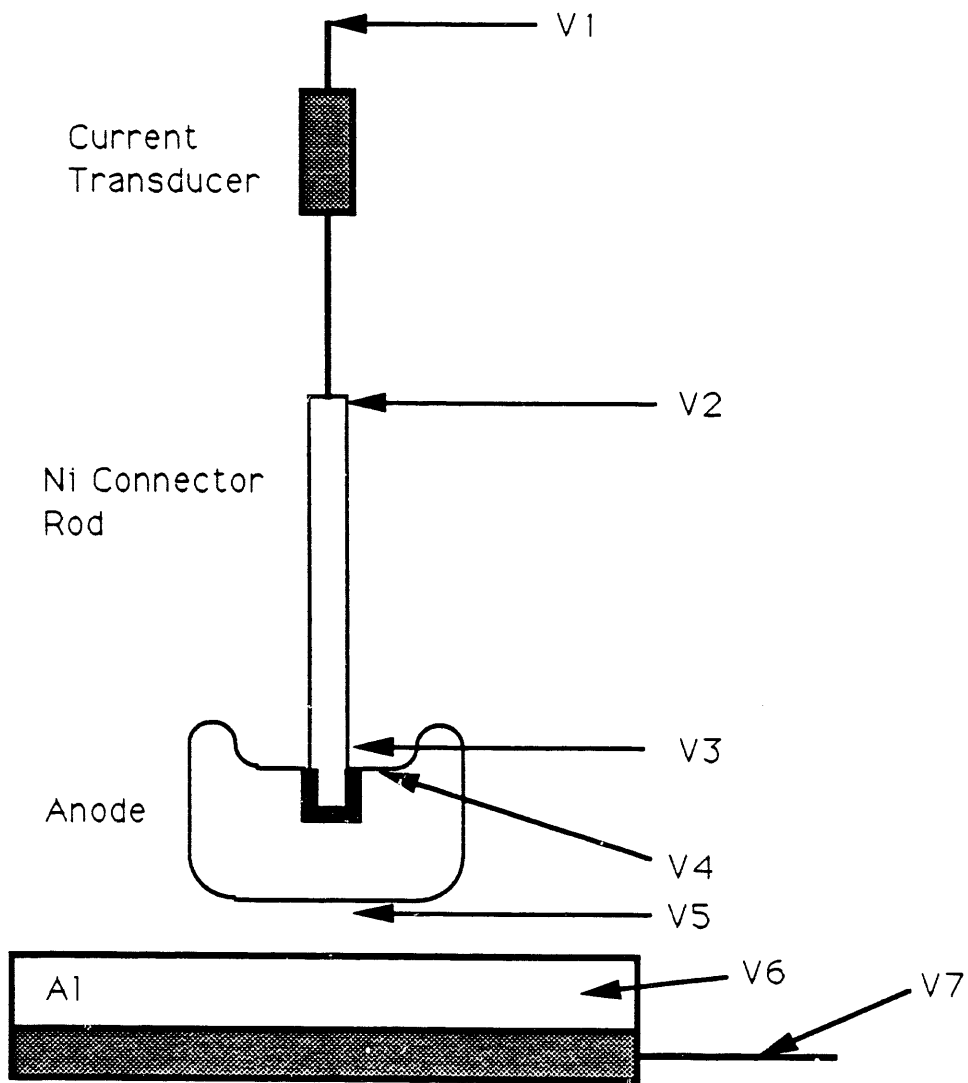
monitoring voltages between these points the voltage characteristics of the cell, particularly in the vicinity of the inert anodes, will be determined. The voltage drops and their significance are given in Table 3. The voltage drop through the bath will also be determined, by varying the ACD or by using the V-probes.

In addition to measuring the voltage drops through the cell, the voltages at various positions within the electrolyte will be determined using voltage probes. This information will help define the potential distribution, and hence current distribution, through the cell and over the anode surfaces. This information may be important in explaining uneven wear on the anodes and in determining the optimum current to use to obtain the targeted nominal current density of 0.5 A/cm². It is intended that this procedure will be performed once, at the beginning of the test.

The voltage probes that will be used are shown in Figure 7. Two of the probes are similar and based on a design by Haupin (1971). They consist of W wire threaded through heavy-walled quartz tube. BN powder will be used to pack the tube and minimize leakage of electrolyte in the event of a small crack. One of these probes is curved and will be used to measure voltage under the anode at various locations. The second probe is straight and will be used to measure voltages further away from the anodes, in corners, and along the side of the cell. The quartz-sheathed probes will be used by inserting them into the cell to

TABLE 3. Voltage Drops

<u>Voltage Drop</u>	<u>Significance</u>	<u>Frequency of Determination</u>
V1-V7	Cell Voltage	Every 30 s.
V2-V3	V-drop through Ni rod.	Once during test.
V3-V4	V-drop across Ni-to-anode connection.	Once each shift.
V4-V5	Electrode potential using V-probe.	Once during test.
V6-V7	V-drop across cathode.	Once each shift.



- V1: Anode Connection to Bus Bar
- V2: Connection at Top of Ni Rod
- V3: Connection at Bottom of Ni Rod
- V4: Tap to Anode Surface
- V5: Measurements in Bath (Voltage Probes)
- V6: Aluminum Pad
- V7: Cathode Connection

FIGURE 6. Schematic of Locations of Voltage Measurements

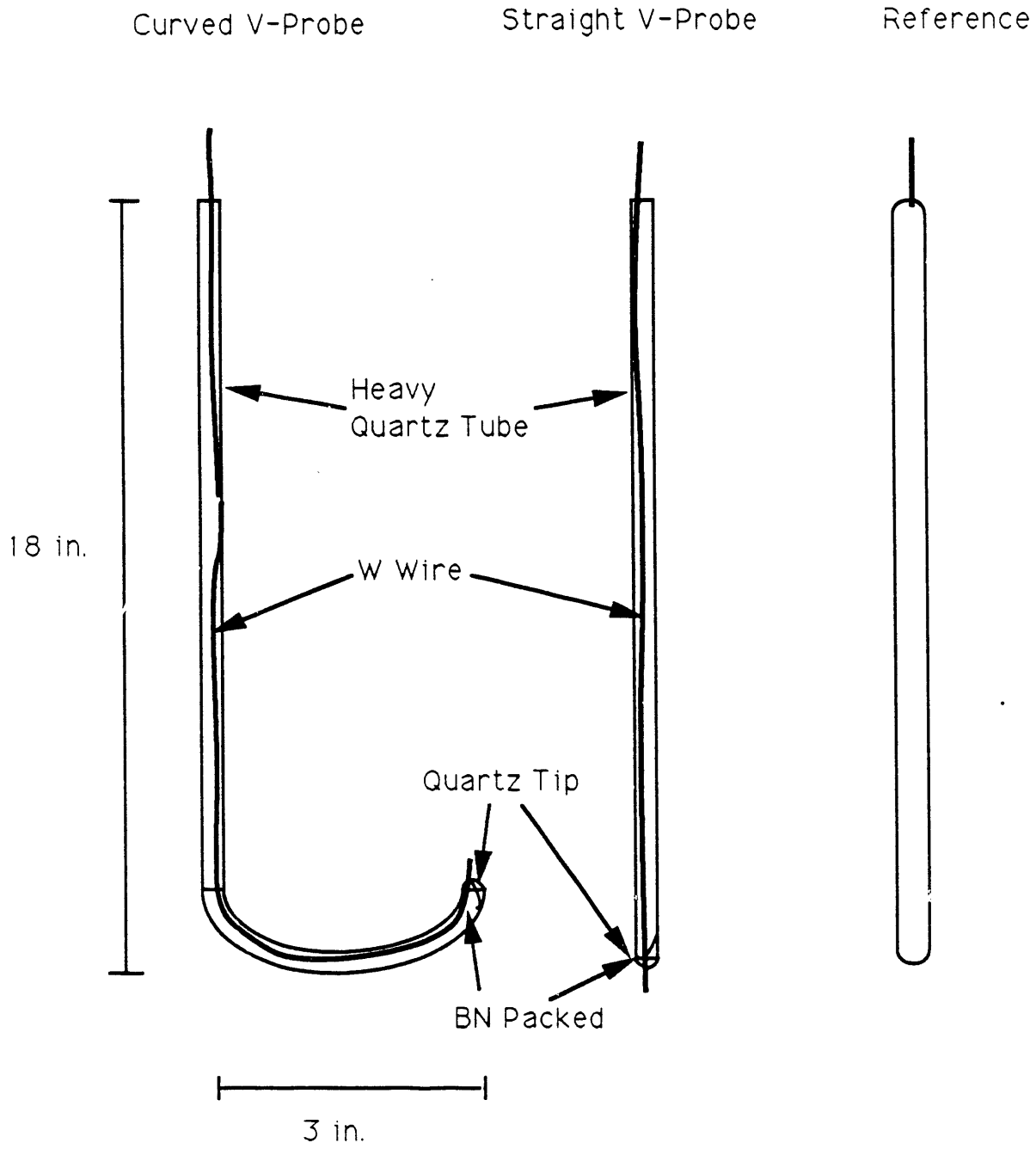


FIGURE 7. Voltage Probes to be Used in the Pilot Test

the desired position, momentarily polarizing them cathodically to plate out some Al metal on the W sensor wire, and then taking a measurement. It is hoped that at least five measurements can be made in the vicinity of each inert anode using the curved probes, and a dozen or so at a position more remote from the anodes with the straight probes. The third type of probe is a reference electrode based on the design by Burgman, Leistra and Sides (1986), and will be used in the event a more stable reference electrode is required.

4.3 POST-TEST ANALYSIS

PNL will be responsible for the post-test analysis of the anodes. The anodes will be sectioned and examined using optical microscopy. Additional samples of the anode will be analyzed by SEM, ion microprobe, and X-ray techniques as required to characterize the anode reactions. Attempts will be made to quantify anode wear by measuring dimensions of the wear regions and comparing with geometries before the test.

RMC will be responsible for the disassembly of the cell and performing any post-test analysis on the cathode or other cell components. A metal inventory will be performed by RMC.

5.0 SENSORS STUDIES MEASUREMENTS

The Sensor Development Program at PNL is being funded by DOE-OIP to develop an alumina sensor for aluminum reduction cells. Currently, digital signal analysis (DSA) methods are being used in an attempt to extract alumina concentration information from current and voltage signals generated by the electrolysis cells. One of the objectives for FY 1991 is to use DSA methods on data collected from the pilot cell. The following sections detail the type of data that will be collected from the pilot cell for the Sensors Development studies. In general, two types of sensor data will be collected: "DSA-type", and "Scope" data. The DSA-type data will be collected for all six inert anodes simultaneously (using up to 16 channels), with a 1-kHz sampling frequency, and a sampling period of 0.5 s. The scope data will be collected on only one inert anode at a time (using only 2 channels), with a 1-Mhz sampling frequency, and a sampling period of 4 ms.

5.1 INSTRUMENTATION

The instrumentation for collecting data for the Sensors Development studies will be provided by PNL.

The equipment for the DSA-type data studies includes an IBM AT and an Analog Devices RTI-860 high-speed simultaneous analog input board (for signal conditioning). HEM Data Corp. Snapshot Storage Scope software will be used to collect, display, and store the data. Inputs to the signal conditioning modules will be over the 16 channels listed in Table 4. In summary, currents through each of the six inert anodes (measured as a voltage drop across the current transducers) will be recorded; and voltage drops across the six inert anodes will be measured using the most reliable connection points, i.e. V2 and V7. Corrections to V2-V7 for voltage drops away from the anode can be made at a later time using the separate voltage drop measurements (e.g., for the cathode) that were described in Section 4.2. All wiring will be performed using shielded cable. Each of the signals will be filtered appropriately for the 1-kHz sampling frequency and the gain of the signal conditioning modules adjusted to maximize signal output.

TABLE 4. Signals for Sensors Studies

<u>Channel</u>	<u>Description of Voltage Source (Refer to Figures 5 and 6)</u>
1	V2-V7 for Anode in Position A
2	Current through Anode in Position A
3	V2-V7 for Anode in Position B
4	Current through Anode in Position B
5	V2-V7 for Anode in Position C
6	Current through Anode in Position C
7	V2-V7 for Anode in Position D
8	Current through Anode in Position D
9	V2-V7 for Anode in Position E
10	Current through Anode in Position E
11	V2-V7 for Anode in Position F
12	Current through Anode in Position F
13	Voltage across Carbon Anode
14	Current through Carbon Anode
15	Available for Anode-to-Reference Voltage
16	Available for Anode-to-Reference Voltage

As indicated in Table 4, channels 15 and 16 will be available for acquisition of potential data with respect to a reference electrode. This data may be taken periodically during the test.

It is important to note that the HEM software functions using a 4-channel "sample-and-hold," meaning that the sampling is not truly simultaneous. For a 1-kHz sampling frequency, however, the delay between acquisition steps is sufficiently small (about 20 μ s) to consider the acquisition over the 16 channels essentially simultaneous.

The scope data will be collected using a Nicolet Model 3091 digital storage scope. The scope will be used to sample current and voltage for one of the six

inert anodes periodically during the test. The scope data are being collected in response to concerns that some information might be missed by neglecting the very high-frequency portion of the signals. The possibility that this is the case is remote, however, in light of the results of previous studies on lab cell data (Windisch et al. 1990).

5.2 SENSOR DATA COLLECTION SCHEDULE

A schedule for the collection of the DSA-type and scope data is given in this section. All sensors data will be collected in triplicate to permit the determination of the precision of the data and the identification of any anomalous signals. The data will be stored on 1.4 MB floppy disks. Given the length of the data files, three files will be stored on a single disk. Consequently, each disk will contain the data in triplicate for a single time and/or set of cell operating conditions.

5.2.1 DSA-Type Data

It is anticipate that the following six kinds of DSA-type data will be collected.

5.2.1.1 Typical Data

These data will be collected routinely during the test. It is anticipated that the data will be collected every 4 to 6 h. Given a 4-week test, with data collected in triplicate, about 450 typical data sets should be obtained. The typical data will provide the opportunity to analyze the stability of the signals and the calculated DSA parameters at alumina concentrations close to saturation. This type of data will also be collected on the carbon anodes for comparison purposes.

5.2.1.2 Volt/Amp Curves

These data sets will be collected at least once a week, when the volt/amp curves are obtained. Assuming data can be collected for four different currents, it is estimated that about 50 of these data files will be obtained. Data collected in conjunction with these curves will show whether changing the current density has any effect on the signals and on the various DSA parameters.

5.2.1.3 ACD Variation

At least once during the test, the ACD will be varied deliberately to obtain additional information. During this variation, DSA-type data will be collected for at least two ACD values. It is estimated that about 50 data files may be acquired during these studies.

5.2.1.4 Varied-Alumina Studies

During the three-day period at the end of the test, DSA-type data will be collected as alumina concentration is varied. These data sets are very important in that they will provide information on the sensitivity of the DSA approach to measuring alumina concentration. It is estimated that about 100 files will be collected during the varied-alumina studies.

5.2.1.5 Chaos Theory Data

One of the DSA approaches that will be used to analyze the data involves chaos theory. Recent work at PNL has shown that very large data sets (50,000 to 100,000) points are beneficial for this type of analysis. Consequently, additional DSA-type data sets will be acquired with more measurements on a given anode. By restricting the data collection to one inert anode and chaining five or more acquisitions, data sets with this many points will be obtained. It is estimated that about 100 files of this type will be obtained.

5.2.1.6 ERC Data

It is anticipated that PNL will collect some DSA-type data during the ERC testing. The details of the conditions for this work will be made clear after ERC has prepared a test plan for their studies.

5.2.2 Scope Data

Data will be taken on a single anode on a periodic basis using the storage scope. As with the DSA-type data, the scope data will be collected in triplicate. It is estimated that the data will be acquired about three times a week and comprise about 36 files.

6.0 OTHER TEST ACTIVITIES AND REQUIREMENTS

6.1 REPORTING AND DATA RECORDING

Numbered PNL laboratory record books will be used to record most data. RMC will also record the data in their own notebooks. Some of the data will be recorded on preprepared data sheets and on magnetic media and strip charts. The data shall become the property of PNL, RMC, and the DOE. A report will be published describing the pilot cell test results approximately four months after completion of the test. This report will be produced jointly by PNL and RMC. Two additional reports will be published by PNL concerning the Sensors Development studies: one report on the results of applying DSA methods and another on the use of chaos theory.

6.2 STAFFING

The pilot cell will be staffed with RMC personnel throughout the test. PNL staff will also be available on-site throughout the test. It is planned that two PNL staff members will be available throughout the test with up to four staff members present during critical activities such as the exchanging of inert anodes. The RMC staff will be primarily responsible for running the cell and performing the various operations and day-to-day activities. The PNL staff will be available to assist the RMC staff in these activities and to provide guidance for operations involving the inert anodes.

6.3 SAFETY

Due to the nature of the test, certain precautions will be necessary to ensure the safety of all personnel and equipment. PNL personnel will abide by the safety policies of the RMC facility. PNL personnel will abide by any restrictions RMC may place on access to certain facilities and operations.

The following is a list of safety clothing required by RMC for the pilot cell operation:

1. Safety glasses
2. Safety shoes (steel toed)
3. Long sleeve shirts (must be worn while working on the cell)

4. Head covering (cap) while working on the cell
5. Cotton clothing
6. Dust masks

PNL staff members will bring all of the above equipment with them with the exception of the dust masks which will be provided by RMC.

During special activities (tapping, anode changes), other specialty equipment (face shields, aluminized coat, hot gloves, etc.) will be provided by RMC. There will be an experienced RMC employee on each shift who will be responsible for ensuring that proper safety precautions are followed.

Additional orientation to safety while working around the cell will be given to each of the PNL staff members upon arrival.

6.4 QUALITY ASSURANCE

The test is a QA Level III activity, and PNL personnel will abide by the requirements of the PNL Quality Assurance Program Good Practices Standards. RMC personnel will abide by their corresponding quality assurance program. PNL may request copies of calibration records for data collection instruments, analytical standards, etc., that are applicable to items provided by RMC.

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