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# Screen Test for Cadmium and Nickel Plates as Developed and Used Within The Aerospace Corporation

15 August 1994

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Prepared for

VICE PRESIDENT
Technology Operations

Engineering and Technology Group

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THE AEROSPACE CORPORATION
El Segundo, California

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# SCREEN TEST FOR CADMIUM AND NICKEL PLATES AS DEVELOPED AND USED WITHIN THE AEROSPACE CORPORATION

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## ABSTRACT

A new procedure described here was recently developed to quantify loading uniformity of nickel and cadmium plates and to screen finished electrodes prior to cell assembly. The technique utilizes the initial solubility rates of the active material in a standard chemical deloading solution at fixed conditions. The method can provide a reproducible indication of plate loading uniformity in situations where high surface loading limits the free flow of deloading solution into the internal porosity of the sinter plate. A preliminary study indicates that "good" cell performance is associated with higher deloading rates.

## ACKNOWLEDGMENTS

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## CONTENTS



## FIGURES

# I. INTRODUCTION

The performance of nickel cadmium (Ni-Cd) and nickel hydrogen ($Ni-H_2$) battery cells has been found to be critically dependent on the variability in active material loading of the nickel and cadmium electrodes used in them. Several recent ground test problems and orbital battery anomalies have been attributed to such inappropriate loading of active material, which is often caused by process instability, or a lack of manufacturing process control. At the present time, no convenient procedure is available to quantify the loading uniformity at the microscopic level, or to screen the finished electrodes prior to cell assembly. The new technique described here shows correlation between plate performance and deloading rates of cadmium and nickel plates. This report describes the procedure and communicates the preliminary results obtained from the new method.

# II. EXPERIMENTAL

*Experimental Procedure for the Deloading Rate Measurements for Nickel and Cadmium Electrodes*

*A. Preparation of Standard Chemical Extraction Solution*

Prepare 1 M of ammonium acetate in 5.4 M ammonium hydroxide solution.
The following items are needed:

- Three-neck-round-bottom-flask: 1000 mℓ, Pyrex (24/40 fitting)
- Condenser: This fits into the mouth of the flask
- Magnetic stirrer
- Heating mantle
- Thermometer
- Sample holder: To keep the sample electrode in a vertical position. Pure nickel holder is suggested as an ideal material because it is inert in basic ammonia solution.

*B. Procedure*

1. Two small samples (1.5 cm x 1.5 cm) are chosen for analysis from each finished plate to be examined. One sample is cut from the center of the electrode using a paper cutting board (away from the edges) and the other sample taken from near one edge.
2. The edges of the samples are coated with viscous epoxy (5-minute epoxy made by Devcon Corporation in Danvers, Mass.) to prevent the leaching of the active material from the cut edges of the samples. Care is taken to avoid epoxy smearing on the sample.
3. The epoxy-edged samples are allowed to cure at room temperature in a desiccator for approximately 24 hours.
4. After curing, the weights, including the epoxy and the dimensions of samples, are obtained. During the epoxy coating process, small portions of the samples are occasionally smeared with wet epoxy. The length and width of the smeared area are measured, and the area of the uncoated surface is obtained by subtracting the smeared area from the geometrical area of the sample.

5. An experimental setup as shown in Figure 1 is used and should be placed in a hood. After 400 mℓ of extraction solution are added to the flask, the assembly is placed on the heating mantle controlled by a variable transformer. The setup is then mounted securely on a magnetic stirring plate. Place the magnetic bar, thermometer, and condenser in the mouths of the flask and initiate cold water flow through the condenser. Stir the solution vigorously and bring solution temperature to 50°C. When the temperature of the solution is stabilized at 50 ± 2°C, the sample electrode is placed vertically in the flask via the sample holder.

6. Active material in the sample electrode is allowed to leach out for 5 minutes by complexing with the ammonia solution which is held at 50 ± 2°C.

7. After 5 minutes, the sample is withdrawn from the extraction flask and thoroughly rinsed until the rinse solution is near to a neutral pH condition. The sample is patted dry and transferred to a desiccator for 24 hours.

8. After it is dry, the sample is reweighed, and the weight loss recorded to determine the standard deloading rate at 50°C.

9. Deloading rate at 50°C = (weight loss)/[(uncoated area) x ($\Delta$t)]

10. Repeat steps 1-9 for the second sample from the same plate.

11. The deloading rate will be an average of the two rates obtained from two different samples from the same electrode.

12. A new extraction solution should be used for every pair of samples.

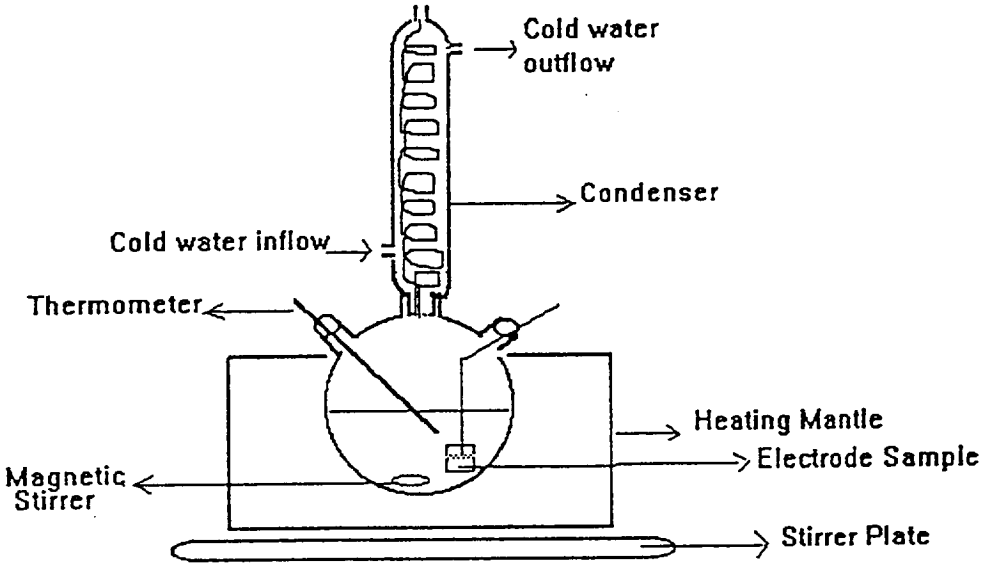


Figure 1. Schematic of the Deloading Experimental Setup

## III. RESULTS AND DISCUSSION

*A. Method Development*

Cadmium electrodes were obtained from "bad" and "good" cells, and subjected to this procedure to determine the appropriate extraction conditions which could provide useful data representative of surface loading characteristics. "Bad" electrodes, as defined here, exhibited anomalously rapid cadmium migration in cells, causing premature short circuits in some cases. It is expected that any surface loading which blocks movement of the deloading solution into the interior of the porous plate should reduce the deloading rate. Figure 2 indicates the kinetic data obtained from two different cell lots, L6A and L2A, which are known to be "good" and "bad", respectively. The preliminary data indicates that the initial solubility rate of active material in the standard extraction solution for fixed conditions provides reproducible measurements of surface loading uniformity of cadmium plates. An extraction time of 5 minutes was selected as the standard time interval to measure the initial deloading rate of cadmium in the ammonia solution. This resulted in approximately 10 to 20% of the active material being deloaded.

*B. Results*

Figure 3 shows preliminary data, which indicate that "good" cell performance is typically associated with higher deloading rates, ranging from 6 to 10 mg/(min.cm$^2$). This implies that the interior structure of the electrode is more open to chemical deloading and thus there is less blockage within the porous structure of the electrodes. Samples from lots 2A, 2B, 3, and 4 are identified with "bad" electrodes, which are usually characterized by lower deloading rates, i.e., 0.3 to 3 mg/(min.cm$^2$).

Figure 4 shows the variation in deloading rates for electrode samples from different spirals within one production post. These differences could be attributed to the variation of the spiral locations in the impregnation tank, resulting in higher or lower surface active material loading. These results clearly show that variability can be significant, not only from lot to lot, but also within a single post of production.

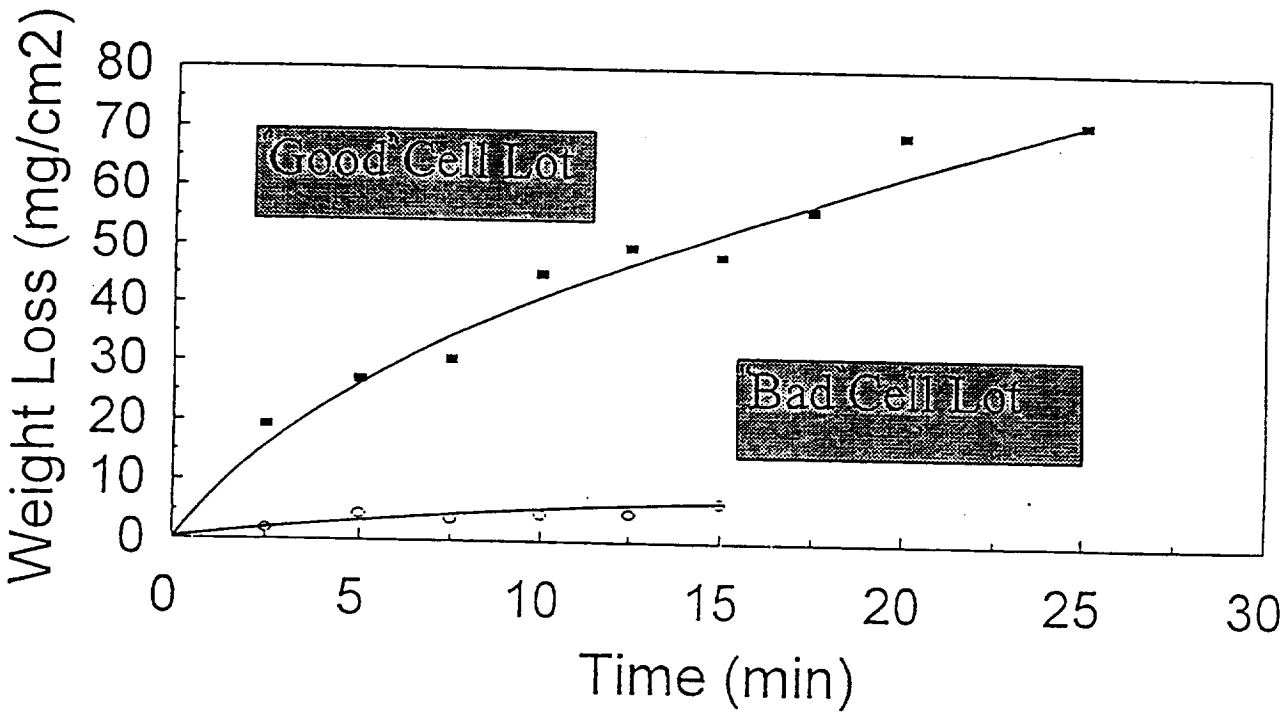


Figure 2. Kinetic Data

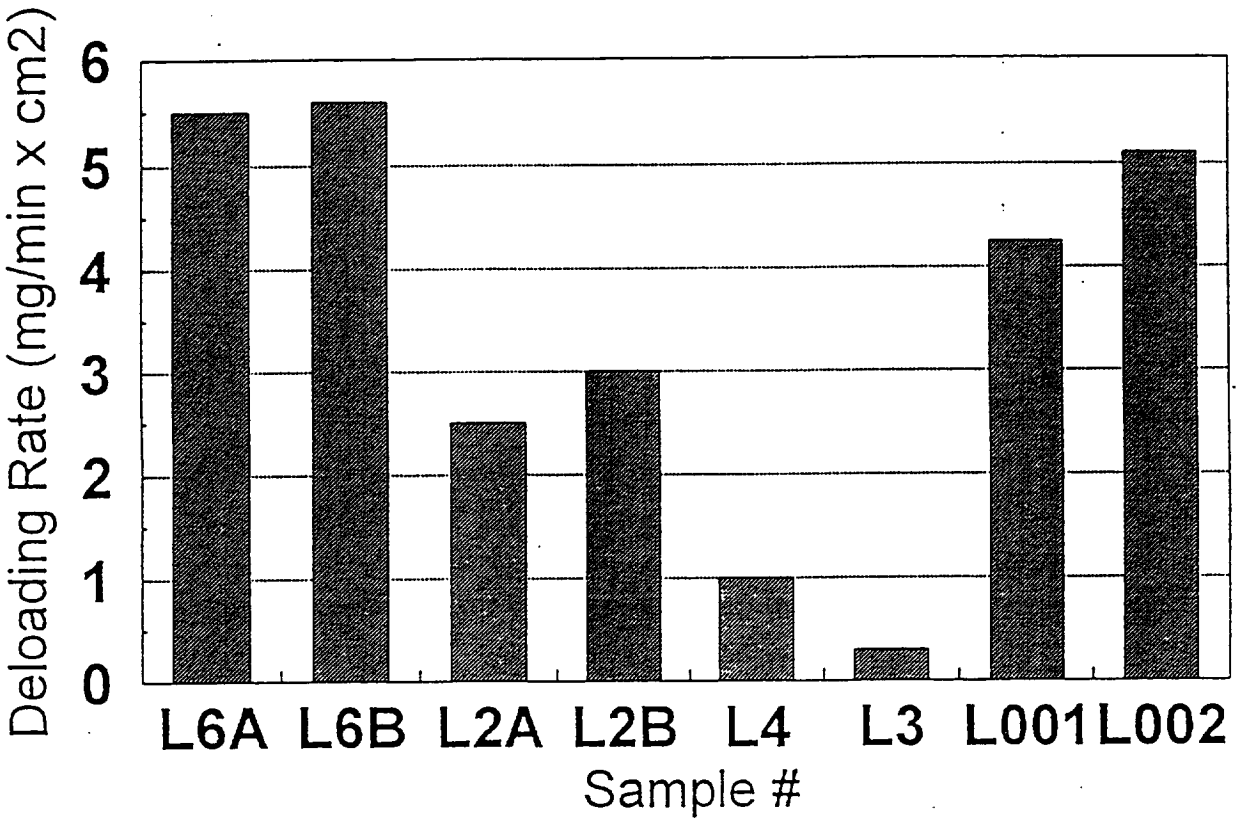


Figure 3. Deloading Experiment for Cadmium Plates

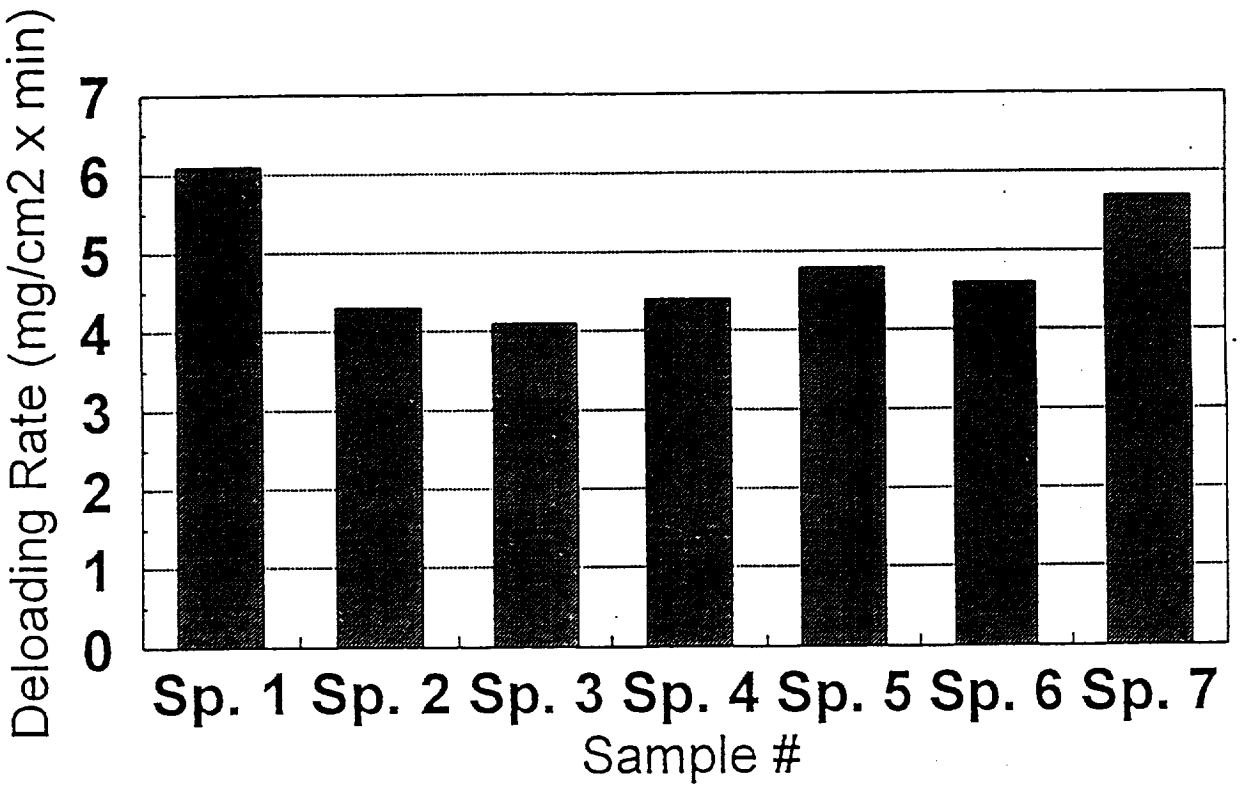


Figure 4. Deloading Experiment for Cadmium Plates, Lot 1B

This screen test is also applicable to the nickel electrode, for which excessive amounts of surface loading of active material have often resulted in poor electrode utilization and accelerated swelling over its cycle life. As shown in Figure 5, the deloading rates of nickel plate samples were found to be proportional to the fractional utilization which is defined as the ratio of the C/2 plate capacity to 0.0 V (vs. Hg/HgO) over the total chemical capacity stored in the electrode as determined by chemical analysis and assuming a one electron valence charge. It is believed that with an appropriate database, an expression of electrode utilization as a function of various parameters, Eq. (1), could be obtained and used to screen finished plates prior to cell assembly, thus preventing costly problems from "bad" plates in spacecraft batteries or in ground tests.

$$\text{Utilization} = k_1L + k_2P + k_3W + k_4Co + k_5D + k_6 \quad (1)$$

Where

L : Loading level (gm/cc void)

P: Sinter porosity (%)

W: Wt. of sinter (gm/cm$^2$)

Co: Percentage of cobalt in active material (wt %)

D: deloading rate, related to surface loading uniformity (gm/min x cm$^2$)

$k_1$, $k_2$, $k_3$, $k_4$, $k_5$, $k_6$ are constants

L, P, W, and Co can be found by chemical analysis of electrodes, and D can be determined from the deloading experiment.

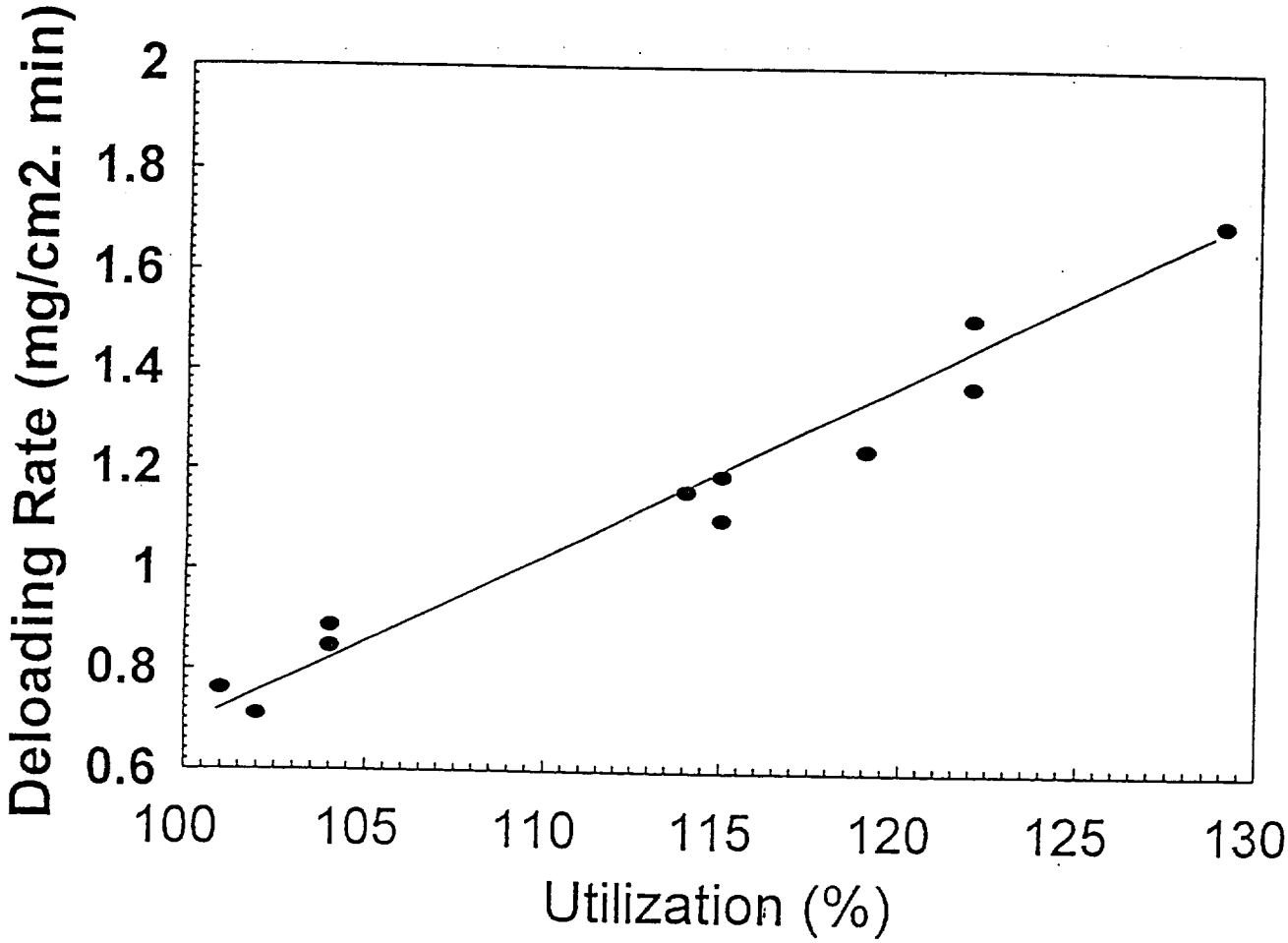


Figure 5. Correlation Between Deloading Rates and Utilization

## IV. SUMMARY AND RECOMMENDATIONS

Currently, we are perfecting the technique and obtaining more data to evaluate method reliability and effectiveness under various conditions. Communication with battery manufacturers will be considered to obtain representative electrodes that are known to be either "good" or "bad" to build up a large database in an attempt to predict electrode utilization as a function of multiple variables which affect plate performance. In the near future this technique, the methods of interpretation, and our results will be forwarded to spacecraft battery manufacturers to allow and encourage them to utilize this new capability.