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PROCEDURE FOR ANALYSIS OF NICKEL-CADMIUM CELL MATERIALS

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GODDARD SPACE FLIGHT CENTER
GREENBELT, MARYLAND

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OF
NICKEL-CADMIUM CELL MATERIALS

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NICKEL-CADMIUM CELL MATERIALS

INTRODUCTION

Nickel-cadmium cells have been substantially improved over the past several years through the implementation of the Specification for Aerospace Nickel-Cadmium Cells[1], several NASA/GSFC contracts[2-5], and general upgrading of the manufacturing processes. The recent improvements were stimulated by the need for high reliability components to meet the long-life aerospace requirements of NASA, DOD, and the communications industry. Because of the improvements through additional and stricter quality controls there has been a significant improvement in uniformity. Therefore, changes in material processes occurring during cycling can now be related with a reasonable degree of confidence to the specific type of operation given the cell. These physical, chemical, or electrochemical changes can be determined using laboratory techniques developed specifically for the analysis of nickel-cadmium cell materials. The procedures for performing analyses on electrolyte, active materials and separator have been coordinated and assembled into this document in a manner such that anyone with a laboratory can perform analyses on nickel-cadmium cell materials (that is, separator, electrolyte, plates).

Several of these procedures have been abstracted from outside sources. Most have been modified to satisfy our analytical requirements. The authors are indebted to those who have supplied information to make this a practical document.

The presentation of the tests includes a brief discussion of each, a list of materials needed, a stepwise procedure, and accompanying data sheets that follow the procedures (Appendixes A and B). A flow diagram of the analytical procedure is given in Figure 1.

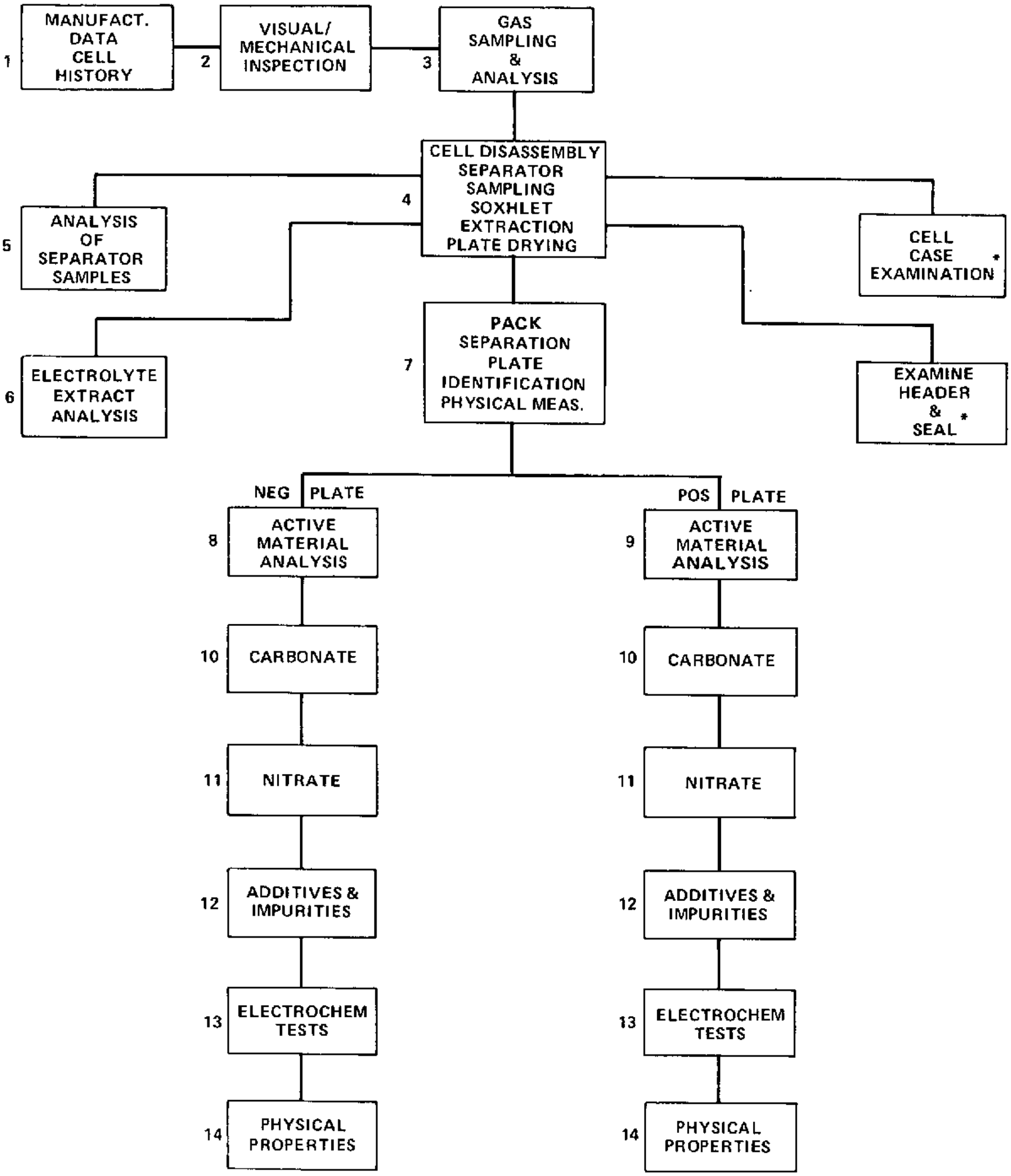
The major steps in the analysis include:

1. Manufacturing data and cell history

2. Visual/mechanical inspection

3. Gas sampling and analysis

4. Cell disassembly, separator sampling; soxhlet extraction and plate drying



*THESE EXAMINATIONS ARE SUGGESTED BUT NOT DESCRIBED IN THIS DOCUMENT.

Figure 1. Flow Diagram of Analytical Procedure

5. Analysis of separator samples
6. Electrolyte extract analysis
7. Plate identification, weight, and thickness
8. Negative plate analysis
9. Positive plate analysis
10. Carbonate in plate
11. Nitrate in plate
12. Additives and impurities
13. Electrochemical tests
14. Physical tests

1. MANUFACTURING DATA, CELL HISTORY

- Record all pertinent data on data sheets provided.

2. VISUAL/MECHANICAL INSPECTION OF CELL

- Visually inspect cell for cracks, leaks, and adherence of material other than original.
- Examine seal and all weld areas.
- Remove all wires and connectors.
- Weigh cell to nearest 0.1 g.
- Record dimensions.
- Record seal type and manufacturer.

3. GAS SAMPLING AND ANALYSIS

It is desirable to determine the composition of gas in those cells that have residual pressure. When a shutoff valve is located on the cell, this is easily accomplished by expanding the gas into an evacuated chamber. The gas in the evacuated chamber is then inserted into a gas chromatograph that has been calibrated for oxygen, nitrogen, and hydrogen. A 92-percent He to 8-percent H_2 carrier gas mixture has been found to distinguish between H_2, N_2, and O_2 in a single sample of gas.

If there is no shutoff valve the can may be punctured using a device developed by GSFC for that purpose.

4. CELL DISASSEMBLY, SEPARATOR SAMPLE REMOVAL, SOXHLET EXTRACTION, AND PLATE DRYING

4.1 EQUIPMENT

- Controlled atmosphere chamber
- 250-ml KOH-inert Erlenmeyer flask with stopper (3)
- Cell case opener
- Long-bladed knife
- Spatula
- Scissors
- Paper towels
- Vise with suction base
- Ruler
- Tweezers
- Metal snips
- Polyethylene bags, self-seal type
- 500 ml of deionized water in flask

- 100-ml graduate
- Balance - readability to 0.01 gm

4.2 PROCEDURE

1. Place cell, GSFC cell case opener*, or similar device capable of cutting open 304L stainless steel case and other tools as required (see list of equipment above) in controlled atmosphere chamber. A vise and snips is needed to remove cell pack from can and disconnect pack from inner terminals. Tweezers and scissors are used to cut and handle separator samples.

2. Cut open cell and remove pack contents from case. Use caution so as not to destroy plates, third electrode, separator or other component. One method is to place cutoff cell header in vise and pull case from pack.

3. Visually inspect pack for unusual construction, color, and any unexpected characteristic.

4. Label and weigh three clean KOH-inert Erlenmeyer flasks (250-ml) with neoprene stoppers (to be weighed before taking them into control atmosphere chamber).

5. Remove three samples of separator from pack by spreading plates and cutting separator material at fold (edge). The samples are to be removed from (a) between second negative and second positive; (b) between fifth negative and fifth positive; and (c) between eighth negative and eighth positive. (In packs where separator bags are used remove entire bag.) Carefully remove separator samples from pack with tweezers.

6. Place each separator sample in a flask and reweigh to determine separator weight. Add 100 cc of deionized water to each flask. Reseal with stopper and swirl occasionally over a 48-hour period.

7. Snip tab connections of plates from comb and place pack in a sealed polyethylene self-seal bag.

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*Drawings available from Electrochemical Power Sources Section (code 76.2), GSFC.

8. Place cell header, cell case, and other cell components in sealed bags for later evaluation.

9. With cell pack in controlled atmosphere, prepare nickel soxhlet extractor* by placing 1000 ml of deionized water in boiler. Bubble nitrogen into boiler through water trap containing deionized water at a high rate for 15 min to remove oxygen from deionized water in boiler and extractor. Attach extraction chamber to boiler. Bubble nitrogen directly into extraction chamber using a temporary cover to minimize oxygen in extractor.

10. Remove polyethylene bags from controlled atmosphere chamber. Place cell pack minus the three separator samples in the extraction chamber of the soxhlet extractor. Open the bag and immediately cover chamber with condenser. Continue nitrogen flow for 15 min at same high rate.

11. Flow cold water through condenser and turn heat on in boiler. Nitrogen gas flow rate is then reduced enough to maintain nitrogen atmosphere in soxhlet.

12. Continue extraction for 48 hrs and terminate heating when a flush has been completed so that most extract will remain in boiler.

13. Disconnect condenser and remove pack. Collect some drippings directly in a flask with phenolphthalein solution. Note color. If pack is completely extracted there will be no change in indicator solution.

14. Pat pack dry to remove excess liquid and place in vacuum chamber or forced nitrogen chamber until dry. An oven with maximum heat at 40° C can be utilized but absence of oxygen must be maintained.

5. ANALYSIS OF SEPARATOR SAMPLES FOR OH^- AND $CO_3^=$

5.1 PRINCIPLE

The double titration method is used to determine potassium hydroxide and potassium carbonate concentrations in the electrolyte. The electrolyte is titrated with acid to a first end point (pH 10 to 8.2) where hydroxide is

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* Model 5010-Ni Soxhlet Extractor, available from Artech Corp., Falls Church, Va.

neutralized and carbonate is converted to bicarbonate (see reactions (1) and (2) below).

$$OH^- + H^+ \longrightarrow H_2O \qquad (1)$$

$$CO_3^= + H^+ \longrightarrow HCO_3^- \qquad (2)$$

A second end point between pH 5.4 to 4.8 is reached during the second titration, when bicarbonate is converted to carbon dioxide (reaction 3).

$$HCO_3^- + H^+ \longrightarrow H_2O + CO_2 \qquad (3)$$

5.2 REAGENTS

- Standard hydrochloric acid solution (0.1 N)
- Standard hydrochloric acid solution (0.01 N)
- Phenolphthalein indicator solution (1 g in 60 ml of ethanol, dilute to 100 ml with H_2O)
- Methyl orange indicator solution (0.1 g in 100 ml of H_2O)

5.3 PROCEDURE

1. To each 250-ml Erlenmeyer flask containing a sample of separator (Section 4, Procedure 6) add four drops of phenolphthalein indicator solution; the solutions will become pink to red.

2. Titrate the first sample to the first end point with 0.1 N HCl (solutions will change from pink to colorless). Record volume of acid used in milliliters. This volume is referred to as $(a + b_1)$.

   a = volume to neutralize all of the KOH
   b_1 = volume to neutralize half the carbonate during titration for OH^-.

3. Add four drops of methyl orange to the flask, and titrate with 0.01 N HCl acid to the methyl orange end point (change from yellow to pale orange). The lower concentration of acid provides greater accuracy in reaching end point. Record the amount of acid used in excess of the phenolphthalein end point. This volume is referred to as b_2

(volume to neutralize the remaining half of the carbonate during titration for HCO_3^-. Therefore, $b_1 = b_2$. Thus, subtracting the volume of acid to reach methyl orange end point from the volume to reach phenolphthalein end point will result in the volume of acid needed to titrate OH^-, that is,

$$\text{Volume to neutralize } OH^- = (a + b_1) - b_2 = a$$

$$\text{Volume to neutralize } CO_3^= = b_1 + b_2 = 2b$$

4. Repeat steps 1 to 3 for the additional two separator samples.

5. Remove each separator sample from the flask and allow them to dry overnight in a clean dry atmosphere.

6. Weigh each and obtain dry weight.

5.4 CALCULATIONS

$$\text{Grams KOH} = \left[(a + b_1) - b_2\right]\left[N_{HCl}\right]\left[56.1\ \frac{g}{1000\ ml}\right] \qquad (1)$$

$$\text{Grams } K_2CO_3 = \left[2b\right]\left[N_{HCl}\right]\left[\frac{138.2}{2}\ \frac{g}{mole}\right]\left[\frac{1\ mole}{1000\ ml}\right] \qquad (2)$$

$$\text{Total as KOH} = \left[g\ KOH\right] + \left[(g\ K_2CO_3)\left(\frac{78.2}{138.2}\right)\left(\frac{56.1}{39.1}\right)\right] \qquad (3)$$

Equivalent milliliters of 31-percent KOH =

$$\frac{\text{Total as KOH}}{0.31\ \frac{g}{g\ solution} \times 1.30\ \frac{g\ solution}{cc}} \qquad (4)$$

6. ELECTROLYTE EXTRACT ANALYSIS

6.1 PRINCIPLE

The analysis for OH^- and $CO_3^=$ in the electrolyte extracted from the cell pack is the same as that for the separator samples. The principle of the double titration method is described in Section 5.

6.2 REAGENTS

See Section 5.

6.3 PROCEDURE

1. Dilute the electrolyte from the soxhlet extractor to 1000 ml (1 liter) in a volumetric flask with deionized water.

2. Pipette 10 ml into a 250-ml beaker and dilute to approximately 100 ml with deionized water and add three to five drops of phenolphthalein indicator solution. Prepare three titration samples in this manner.

3. Titrate solution to end point (change from pink to clear) with 0.1 N HCl and record volume in milliliters.

4. Add four drops of methyl orange to impart a yellow color to the solution, then titrate to the end point (change to pale orange) with 0.01 N HCl solution and record volume of HCl.

6.4 CALCULATIONS

$$\text{Grams KOH} = [(a+b_1)-b_2]\ \text{N HCl}_1 \times 56.1\,\frac{\text{g}}{\text{mole}} \times \frac{1\ \text{mole}}{1000\ \text{ml}} \times \frac{1000\ \text{ml}}{10\ \text{ml}} \tag{5}$$

$$\text{Grams } K_2CO_3 = (2b)\ \text{N HCl} \times \frac{138.2}{2} \times \frac{1\ \text{mole}}{1000\ \text{ml}} \times \frac{1000\ \text{ml}}{10\ \text{ml}} \tag{6}$$

$$\text{Total as KOH} = (\text{g KOH}) + \left[\text{g } K_2CO_3 \times \frac{78.2}{138.2} \times \frac{56.1}{39.1}\right] \tag{7}$$

$$\text{Equivalent milliliters of 31-percent KOH} = \frac{\text{Total as KOH}}{0.31\,\frac{\text{g}}{\text{g solution}} \times 1.30\,\frac{\text{g solution}}{\text{cc}}} \tag{8}$$

7. PLATE IDENTIFICATION, WEIGHT, AND THICKNESS

1. Remove pack from drying chamber; separate positive from negative plates. Label as such in order of removal.

2. Weigh each plate to the nearest 0.01 g and measure thickness at top, middle, and bottom to nearest 0.1 mil.

3. Number plates according to their positions in the cell (negatives odd numbers and positives even). Place each in labeled polyethylene bag and seal. See Table 1 for suggested labeling arrangement.

4. Remove separator, place in separate bag and seal.

8. ANALYSIS OF THE NEGATIVE PLATE

8.1 PRINCIPLE

Cadmium hydroxide ($Cd(OH)_2$) is recognized as the discharged active material and cadmium metal (Cd) as the charged active material in the negative plate of nickel-cadmium cells. The analysis procedure involves the separation of the cadmium ion of the cadmium hydroxide from the cadmium metal in the nickel plaque comprising a nickel or steel substrate. The first step of the separation is accomplished by extraction of the plate with ammonia solution to form the $[Cd\ (NH_3)_6]^{++}$ complex. The complex is broken with the addition of formaldehyde, and the free Cd^{++} is then titrated with standardized disodium ethylene-diamine-tetraacetate (EDTA). The second step of analyzing for the charged cadmium involves dissolving the remainder of the sample from above with nitric acid to form the nitrate. The cadmium ion (Cd^{++}) thus formed is complexed in basic ammonia solution to form the cadmium-ammonia complex as above. However, consideration must be given to the nickel and iron ions also present in solution. After standing overnight, iron hydroxide, which appears as a brown precipitate, is removed by filtration. The nickel and cadmium ions are converted from the basic solution to a cyanide complex by adding sodium cyanide. Then formaldehyde is added to break the cadmium complex without affecting the nickel complex. The free cadmium ion is then titrated with the standardized EDTA solution.

8.2 REAGENTS

- EDTA solution (0.01 m): Dissolve 3.7224 g of dried high purity disodium ethylene-diamine-tetraacetate in deionized water and dilute to 1 liter in a volumetric flask. (EDTA is a primary standard, no further standardization is needed.)

- Concentrated ammonium hydroxide (reagent grade)

- Buffer: Dissolve 54 g of ammonium chloride in approximately 300 ml deionized water. Add 350 ml ammonium hydroxide and dilute to 1 liter with deionized water (pH = 10.00).

Table 1
Plate Identification and Tests

Position in Cell		Test Number	Analysis Performed
Negative	Positive		
1			No test planned
	2	9	Active material analysis - positive
3		8	Active material analysis - negative
	4	10-12, 14	Carbonate nitrate impurities, physical properties
5		10-12, 14	Carbonate, nitrate impurities, physical properties
	6	13	Electrochemical test - positive
7		13	Electrochemical test - negative
	8	9	Active material analysis - positive
9		8	Active material analysis - negative
	10	8	Electrochemical test - positive
11		8	Electrochemical test - negative
	12	9	Active material analysis - positive
13		8	Active material analysis - negative
	14	10-12, 14	Carbonate, nitrate impurities, physical properties
15		10-12, 14	Carbonate, nitrate impurities, physical properties
	16	13	Electrochemical test - positive
17		13	Electrochemical test - negative
	18		No test planned
19			No test planned

- Concentrated nitric acid (reagent grade)
- Ten-percent formaldehyde solution: Dilute 28 ml of 36-percent formaldehyde to 100 ml with deionized water in a volumetric flask.
- Ten-percent sodium cyanide solution: Dissolve 25 g of sodium cyanide in 250 ml of deionized water in a volumetric flask.
- Eriochrome Black T Mix: Dry mix 0.125 g of Eriochrome Black T with 50 g of sodium chloride.
- Extraction solution: Dissolve 232 g of ammonium acetate in 2 liters of concentrated ammonium hydroxide and bring volume to 3 liters with deionized water. (This volume is enough for two disks from each of three plates.)

The following special parts are suggested:

- Erlenmeyer flask—1000-ml wide-mouth (Fisher No. 10090D or equivalent)
- Cold finger (Fisher No. 7-746 or equivalent)—This fits into the mouth of the wide-mouth Erlenmeyer flask.
- A sample holder—To be placed in the bottom of the Erlenmeyer flask to maintain the sample to be analyzed in a vertical position. The sample holder material must be inert to the basic ammonia solutions; pure nickel is suggested (see Figure 2).

8.3 PROCEDURE

1. Each sample is prepared so that the weight of cadmium expected is approximately 1 g (a 1-in. diameter disk is suitable). The sample should be dry and free of electrolyte. Two samples are prepared from each of three plates (#3, #7, and #11, see Table 1).
2. Determine the weight of each plate (Wp) without the tab. Punch a disk from the plate and determine the weight (Ws in grams) of this sample. Measure thickness of each disk.
3. Place sample holder in bottom of wide-mouth Erlenmeyer flask. Add 500 ml of extraction solution to flask and place on hot plate. Place cold finger in mouth of flask and initiate cold water flow. Bring

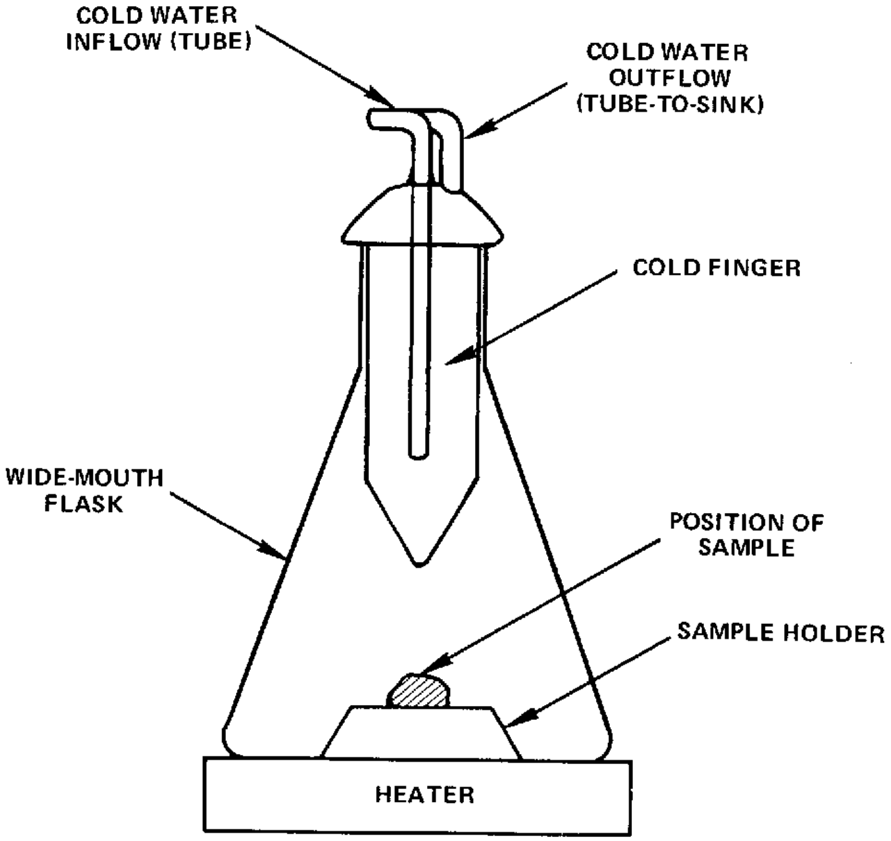

Figure 2. Apparatus for Extraction of Cadmium Hydroxide

temperature of solution to a boil (70 to 80° C). When heating more than one flask, care must be taken to maintain a uniform temperature for all flasks (use hood).

4. Add sample to holder and replace cold finger immediately. Heat for 2 hr (keeping temperature between 70 and 80° C) during which time the Cd^{++} in the sample will be complexed with the ammonia in the solution.

5. Cool and transfer the extract to a 500-ml volumetric flask. Wash disk with deionized water and transfer washings to flask.

6. Dilute to volume with deionized water. Dry and reweigh each disk and store in sealed container. Save the sample disk for the analysis of the cadmium (see section on analysis of cadmium metal).

7. Transfer a 10-ml aliquot to a 250-ml beaker and dilute to approximately 100 ml with deionized water.

8. Place beaker on magnetic stirrer and adjust pH of the solution to 10 by adding concentrated ammonium hydroxide. Add 10 ml of buffer solution. Remove pH electrodes from sample and add 5 ml of 10-percent sodium cyanide.

9. Add enough Eriochrome Black T to impart a medium blue color to the sample solution. Add 7 ml of 10-percent formaldehyde solution. Titrate immediately with EDTA solution (V_{EDTA}) until a permanent blue color is obtained.

10. Calculate the capacity of the active material in the discharged state (C_{DNC}) using Equations (9) to (11) below.

8.4 ANALYSIS FOR CADMIUM METAL—CHARGED STATE

1. Dissolve the nickel plaque in 1:1 volume of nitric acid and deionized water (~10 ml of solution) (use hood).

2. Evaporate to syrupy consistency, cool, and dilute to 100 ml with deionized water in a 250-ml beaker. Adjust pH to 10 with concentrated ammonium hydroxide (NH_4OH). If the substrate contains iron, a brownish precipitate may form, which indicates iron hydroxide.

3. If iron is present, filter the precipitate with suction, then dilute the filtrate to 250 ml with deionized water in a volumetric flask.

4. Transfer a 10-ml aliquot to a 250-ml beaker and dilute to approximately 100 ml with deionized water. Place sample on magnetic stirrer and adjust pH to approximately 10 with concentrated NH_4OH.

5. Add 10 ml of buffer. If the sample remains cloudy, add an additional 10 ml of buffer.

6. Remove pH electrodes from sample and add 5 ml of 10-percent sodium cyanide solution.

7. Add Eriochrome Black T as stated above, 7 ml of 10-percent formaldehyde, and titrate with EDTA as in step 1. The volume required is V'_{EDTA}.

8. Calculate the capacity of the active material C_{CNC} in the charged state in the plate as in Equations (12) to (14) below.

8.5 CALCULATIONS

Grams of discharged negative material in plate

$$W_{DNP} = \frac{W_P}{W_S} \frac{(0.01)}{1000} (V_{EDTA}) \left(\frac{500}{10}\right)^{(2)} \left(\frac{146.4}{2}\right) \tag{9}$$

Ampere-hours equivalent of discharged material in plate

$$C_{DNP} = (W_{DNP}) (26.8) \left(\frac{2}{146.4}\right) \tag{10}$$

Ampere-hours equivalent of discharged material in cell

$$C_{DNC} = (C_{DNP}) \text{ (number of negative plates)} \tag{11}$$

Grams of charged material as cd $(OH)_2$ in plate

$$W_{CNP} = \frac{W_{PL}}{W_S} \left(\frac{0.01}{1000}\right) \left(V'_{EDTA}\right) \left(\frac{250}{10}\right)^{(2)} \left(\frac{146.4}{2}\right) \tag{12}$$

Ampere-hours equivalent of charged material in plate

$$C_{CNP} = (W_{CNP})\ (26.8)\ (\frac{2}{146.4}) \qquad (13)$$

Ampere-hours equivalent of charged material in cell

$$C_{CNC} = (C_{CNP})\ \text{(number of negative plates)} \qquad (14)$$

Ampere-hours of total negative in cell

$$C_{TNC} = C_{DNC} + C_{CNC} \qquad (15)$$

NOTE:

Equations (9) and (12) are of the form

$$\left(\frac{\text{plate wt}}{\text{sample Wt}}\right) \left(\frac{\text{moles EDTA}}{\text{1000 ml}}\right) \text{(ml EDTA)} \left(\frac{\text{Vol Flask}}{\text{Vol aliquot}}\right) \left(\frac{\text{2 equiv. Cd}}{\text{mole}}\right) \left(\frac{\text{146.2 g}}{\text{2 equiv.}}\right)$$

9. CHEMICAL ANALYSIS OF POSITIVE PLATES

9.1 PRINCIPLE

Although providing useful results with regard to the chemical composition of the positive plate, this procedure is known to be somewhat inaccurate, particularly when there is a significant quantity of charged material present. If the plates are in the discharged condition the errors are minimized. The discharged material in the positive plate is accepted as $Ni(OH)_2$ and the charged material as NiOOH or NiO_2. To determine the amount of charged material, the mixture of sintered nickel and active material is treated with excess of ferrous ammonium sulfate in acetic acid solution. This results in a reduction of higher valent (both trivalent and tetravalent) nickel to divalent nickel by the simultaneous oxidation of ferrous ions to ferric ions:

$$NiO\ OH + Fe^{2+} + 3H^{+} \longrightarrow Ni^{2+} + Fe^{+++} + 2\ H_2O$$

$$NiO_2 + 2Fe^{2+} + 4H^{+} \longrightarrow Ni^{2+} + 2Fe^{3+} + 2\ H_2O$$

The excess of ferrous ions in the solution is then titrated with a standard potassium permanganate solution:

$$5\ Fe^{2+} + MnO_4^{-} + 8H^{+} \longrightarrow 5\ Fe^{3+} + Mn^{2+} + 4\ H_2O$$

To determine the total amount of active material, the mixture of sintered nickel and active material is leached in acetic acid; hydrazine sulfate is added as an inhibitor for metallic nickel dissolution. The nickel in the solution is then titrated with a standard EDTA solution. The amount of discharged material is determined by subtracting the charged material from the total. To determine the amount of metallic nickel, nitric acid is used to dissolve the metallic residue from the extraction which was described in the preceding paragraph. The nickel in the solution is then titrated with a standard EDTA solution.

9.2 REAGENTS

These reagents are required for the chemical analysis of positive plates:

- Standard potassium permanganate solution (0.1 N)
- Concentrated sulfuric acid (reagent grade)
- Ferrous ammonium sulfate (ACS purity)
- Acetic acid (10 percent by volume)
- Hydrazine sulfate (ACS purity)
- Concentrated ammonium hydroxide (reagent grade)
- Pyrocatechol violet indicator (0.1-percent solution in water)
- EDTA solution (0.01 M) (see Section 8, first reagent, for directions on preparation).

9.3 CHARGED MATERIAL ANALYSIS

Procedure

1. The weight of plates (W_p) #4, #10, and #14 are determined with tabs removed.

2. Two 1-in. diameter disks are punched from each of the plates. A 1-g sample is used where a punch is unavailable. The weight of each sample disk is determined (W_s).

3. The sinter and the active material is broken off the disk and the weight of the substrate (W_{ss} in grams) is determined.

4. The nickel mixture is prepared by grinding the nickel sinter and active material to a fine powder.

5. The weight of the sinter and the active material in the plate (W_a) is calculated from Equation (16).

6. A sample of the nickel mixture from 4 above (0.3 to 0.5 g) is weighed (W_{psc}).

7. Approximately 150 g of ferrous ammonium sulfate is weighed (W_{fas}).

8. The nickel mixture and the ferrous ammonium sulfate are transferred into a 250-ml flask which has a ground glass stopper; 100 ml of 10-percent acetic acid is added, and the air in the flask is replaced with nitrogen by blowing this gas over the solution. Then the flask is closed and the mixture is allowed to react for 1 hr while it is being stirred with a magnetic stirrer.

9. The metallic nickel residue is collected with the magnetic bar, and 2 ml of concentrated sulfuric acid is added to the solution slowly. (CAUTION, USE HOOD.)

10. The light-green solution is titrated with (0.1 M) standard potassium permanganate solution to the end-point (light-green changes to violet). (V_{perm})

11. The number of ampere-hours (C_{CPP}) of charged material is calculated as in Equations (17) to (19).

9.4 TOTAL ACTIVE MATERIAL AND METALLIC NICKEL ANALYSIS

Procedure

The total active material analysis is done as follows:

1. Weigh a sample (0.3 to 0.4 g) of the nickel mixture from procedure 4 of Section 9.1 (W_{pst}) and approximately 1.5 g of hydrazine sulfate into a 250-ml volumetric flask, add 100 ml of 10-percent acetic acid, stopper loosely and replace air in flask with nitrogen stopper, and stir for 1 hr on a magnetic stirrer.

2. Transfer 10 to 15 ml of concentrated nitric acid (HNO_3) into a 250-ml volumetric flask, decant the solution from procedure 1 into it and dilute to mark with deionized water. Label "sample."

3. Dissolve the metallic residue that has been collected on the stirring bar with nitric acid in 250-ml beaker (heating hastens dissolution). Let solution boil for about 6 to 10 minutes (depending on the amount of HNO_3 used) to expel excess nitric fumes. (Use hood.)

4. Transfer solution from procedure 3 above to a 250-ml volumetric flask and dilute to mark with deionized water. Label "sample."

5. Pipette a 10-ml aliquot of the sample in step 2 into a 250-ml Erlenmeyer flask and dilute to approximately 100 ml with deionized water. Add 5 to 6 ml of concentrated ammonium hydroxide (NH_4OH) and 2 drops of pyrocatechol indicator solution.

6. Titrate solution dropwise with standard EDTA solution till end-point is attained (blue changing to violet). The average amount of EDTA used in titrating the total active material will be identified as V_{EDTA}.

7. Repeat steps 5 and 6 using 10-ml aliquot from solution in step 4. The volume of EDTA used to titrate the nickel sinter sample is identified as V'_{EDTA}.

8. Calculate volume of original sample using thickness (cm) and area of sample disk (cm^2). Calculate porosity using Equation (26).

9.5 CALCULATIONS

Weight of sinter plus weight of active material in plate

$$W_a = W_p \left(1 - \frac{W_{ss}}{W_s}\right) \tag{16}$$

Grams of charged material as Ni $(OH)_2$ in plate

$$W_{cpp} = \frac{Wa}{W_{psc}} \quad (92.73) \quad \left[\frac{W_{fas}}{392.15} - \frac{(V_{perm})(0.1)}{1000}\right] \tag{17}$$

Ampere-hours equivalent of charged positive material in plate

$$C_{cpp} = \frac{(W_{cpp})(26.8)}{92.73} \tag{18}$$

Ampere-hours of charged positive material in cell

$$C_{cpc} = C_{cpp} \text{ (Number of positive plates)} \tag{19}$$

Grams of active material in charged and discharged state as $Ni(OH)_2$

$$W_{tpp} = \frac{W_a}{W_{pst}} \quad \frac{250}{10} \quad \frac{(0.01)}{(1000)} \quad (V_{EDTA}) \; (92.73) \tag{20}$$

Grams of $Ni(OH)_2$ in discharged state in plate

$$W_{dpp} = W_{tpp} - W_{cpp} \tag{21}$$

Ampere-hours equivalent of discharged positive material in plate

$$C_{dpp} = W_{dpp} \quad \left(\frac{26.8}{92.73}\right) \tag{22}$$

Ampere-hours equivalent of discharged positive material in cell

$$C_{dpc} = C_{dpp} \text{ (Number of positive plates)} \tag{23}$$

Total ampere-hours of positive active material in cell

$$C_{tpc} = C_{cpc} + C_{dpc} \tag{24}$$

Grams of metallic nickel in positive plate

$$W_{NPP} = \left[\frac{(W_a)}{(W_{pst})} \quad \left(\frac{250}{10}\right) \quad \frac{(0.01)}{(1000)} \quad (V'_{EDTA}) \quad (58.71)\right] \tag{25}$$

Porosity of positive plate

$$P_p = 1 - \frac{W_{npp}}{\text{(area of plate) (thickness)}(8.90)} \tag{26}$$

10. CARBONATE IN PLATES

10.1 PRINCIPLE

There are two sources of carbonate in the positive and negative plates of nickel-cadmium cells. One source is K_2CO_3 which is an inherent part of plates or electrolyte built into a cell during manufacture or is a result of degradation of separator material during cell operation. A second source is the carbonate associated with the active materials of the plates, namely nickel carbonate ($NiCO_3$) in the positive and cadmium carbonate ($Cd\ CO_3$) in the negative plate. In the procedure suggested, K_2CO_3 is removed from plate samples by dissolution in deionized water. The solution is titrated as in analysis of separator samples (Section 5) and soxhlet extract solution (Section 6) by the double titration method.

The determination of $NiCO_3$ and $CdCO_3$ can be accomplished by dissolving the plate sample above in concentrated acid. The CO_2 gas evolved is collected as a precipitate in $Ba(OH)_2$ as $BaCO_3$ or in KOH solution, as K_2CO_3. However, for small quantities of carbonate this procedure is insensitive and somewhat tedious.

A recently developed technique utilizes the gas chromatograph to detect the small quantities of CO_2 evolved when the acid reacts with the carbonate in the plate. Sensitivities to as low as 1 mg of carbonate can be detected using this technique. It is accomplished by collecting the CO_2 in a liquid nitrogen trap and then expanding it into a gas chromatograph calibrated for CO_2 gas.

10.2 MATERIALS

- H_2SO_4 (1. N)
- K_2CO_3 reagent grade
- Filter flask (250-ml)
- Pressure transducer and digital millivoltmeter
- Hot plate-stirrer
- Drying tube with indicating silica gel
- Hypodermic syringe (5-ml) and long needle

- Gas flowrate controlling valve
- Gas chromatograph (Fisher model 25 or equivalent) with gas sampling valve

  Column #1, 11 in. of 60- to 80-mesh silica gel
  Column #2, 30 in. of molecular sieve
  Sample loop, 7 ft. coiled copper tubing 3 in. O.D.

- mv recorder
- Dewar flask and liquid nitrogen
- Helium gas
- Vacuum pump
- Beaker (250-ml)

10.3 PROCEDURE

1. Calibrate the chromatograph and columns using preweighed quantities of K_2CO_3. When calibration is completed, select plates used in chemical analysis (negative plates #3, #7, and #11; positive plates #4, #10, #14) and punch two 1-in. diameter disks from each. For calibration see step 4. For carbonate analysis in plate continue with step 2.

2. Weigh all disks to nearest 0.001 g; place one of the two sample disks from each plate in a separate beaker containing 25 ml of deionized water. Heat to boiling for 5 min. Remove disk, dry, and reweigh each.

3. Titrate each beaker in step 2 using the double titration method of Sections 5 and 6 above.

4. Arrange instrumental apparatus as shown in Figure 3. Grind each disk from end of step 2 to a fine powder; then weigh. Place the powdered sample and stirring bar in filter flask. Gas sampling valve (V_3) is in position 1, that is, connected from vacuum pump through sample loop to filter flask. Gas flow control valve (V_1) is open. Turn on vacuum pump.

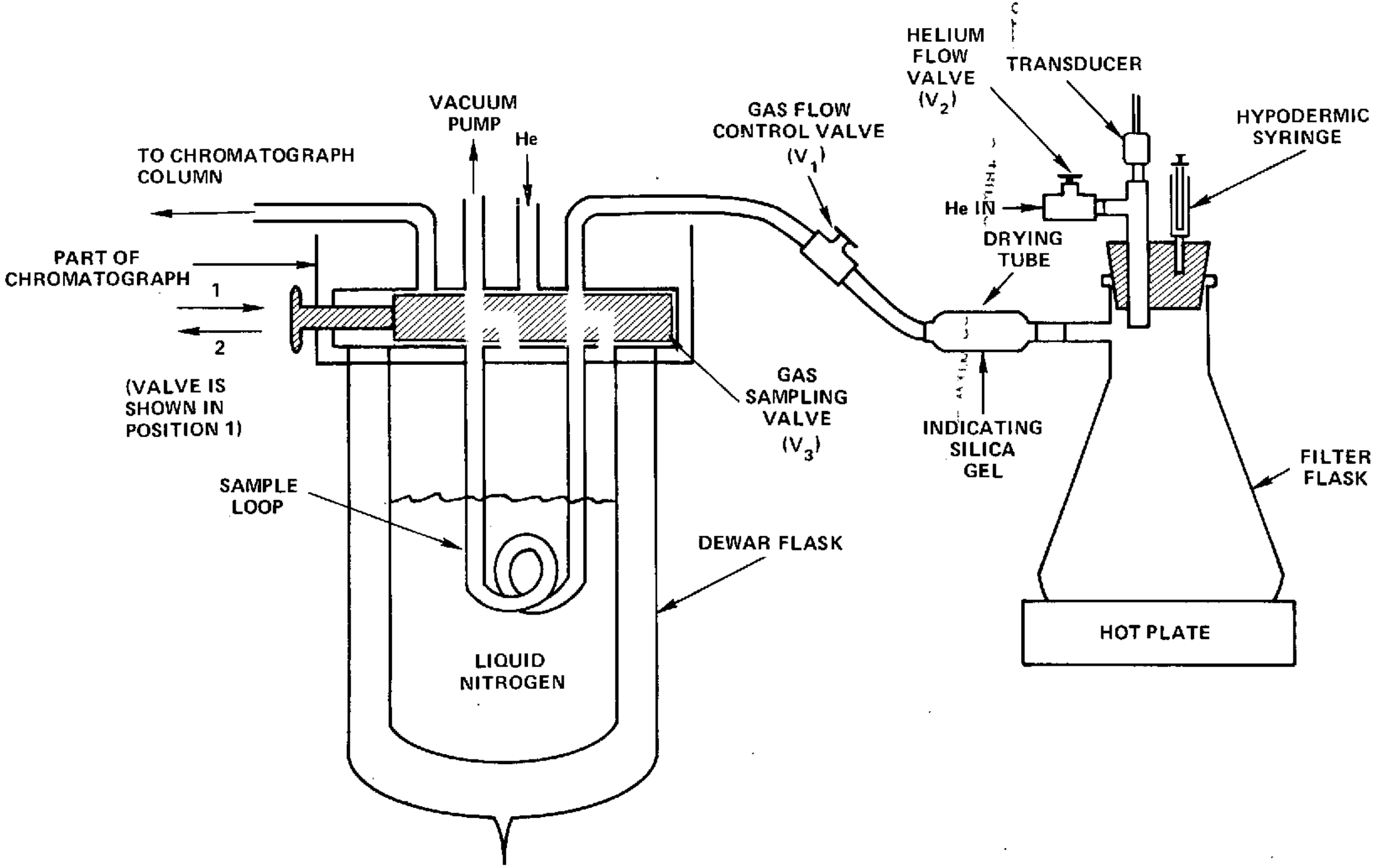

Figure 3. Apparatus for Determination of Carbonate in Plates

5. Fill Dewar flask around sample loop with liquid nitrogen. Flush filter flask with helium gas through helium flow valve (V_2), close V_1, open V_2. Pull vacuum, then open V_1, close V_2, and repeat five times concluding with vacuum.

6. Close V_1 and V_2 with vacuum pump on and still connected. Continue to pull vacuum back to V_1.

7. Inject 4 ml of preheated 0.1 N H_2SO_4 at 50° C into filter flash through rubber stopper with hypodermic syringe. Ensure that sample is well stirred. Continue heating of flask with sample and acid.

8. Open V_1 slowly until pressure in apparatus as indicated by millivoltmeter connected to the pressure transducer has stabilized (~5 min). Open valve further and continue pulling vacuum on filter flask until liquid starts to boil.

9. Close V_1 and remove liquid nitrogen Dewar from around sample loop. Place beaker of room temperature water around sample loop and allow to come to equilibrium (~2 min).

10. Place V_3 in position 2 to allow sample to be swept into gas chromatograph in helium carrier gas as per Figure 3.

11. Compare recorder peak height with calibration curve obtained with K_2CO_3 for determination of carbonate in plate. Because the calibration is in terms of K_2CO_3 the values are converted to $NiCO_3$ and $CdCO_3$ using the appropriate molecular weight ratios.

10.4 CALCULATIONS

Grams of K_2CO_3 in sample

$$\text{Use equations of Section 5} \tag{27}$$

Grams of K_2CO_3 from chromatograph

$$\text{Use calibration curve} \tag{28}$$

Grams of $NiCO_3$ in positive plate

$$W_{pc} = \left(\frac{118.72}{138.21}\right)\left(\frac{W_{plate}}{W_{disk}}\right)\left(\begin{array}{c}K_2CO_3 \text{ in disk} \\ \text{from curve}\end{array}\right) \tag{29}$$

Grams of $CdCO_3$ in negative plate

$$W_{nc} = \left(\frac{172.21}{138.21}\right)\left(\frac{W_{plate}}{W_{disk}}\right)\left(\begin{matrix}K_2CO_3 \text{ in disk} \\ \text{from curve}\end{matrix}\right) \tag{30}$$

11. NITRATE ANALYSIS

Nitrate in nickel and cadmium cell plates has been known to increase the self-discharge rate significantly. It can be found in plates whose active material conversion from the nitrate to the hydroxide has not been complete. The Kjeldahl method for nitrate determination is widely accepted and has been found to be adequate for analysis of battery plates as well.

11.1 MATERIALS

- Pregl-type micro Kjeldahl distillation apparatus
- Boric acid solution 4 percent
- Devarda's Alloy
- NaOH solution 12.5 m
- Indicator: methyl red - methylene blue, prepared by mixing two parts of 0.2-percent methyl red with 1 part 0.2-percent methylene blue both in 95-percent ethanol.
- HCl (0.07 N)

11.2 PROCEDURE

1. Weigh plate, then punch dry electrolyte-free disk and weigh.
2. Boil in 20 ml of deionized water.
3. Rinse quantitatively into distillation apparatus.
4. Transfer 5 ml of 4-percent boric acid solution into a 125-ml Erlenmeyer flask; place flask under the delivery tube of the condenser, and tilt the flask so that the tip of the condenser flask extends below the liquid surface.

5. Add 0.5 to 0.6 g of Devarda's alloy (50 percent copper, 45 percent aluminum, and 5 percent zinc) to the flask together with 25 ml of NaOH (containing 500 g of NaOH/liter).

6. Begin steam distillation. (Care must be exercised in regulation of the reaction with the reducing alloy by careful adjustment of the steam supply. This is a critical point, because if too vigorous a reaction causes any of the NaOH to froth over into the receiving flask, the analysis will be ruined.)

7. Perform distillation for 5 min from the time condensation begins in the condenser.

8. Lower the Erlenmeyer flask so that the end of the tube is out of the liquid and up near the neck of the flask, and distill for another 3 min.

9. Using methyl red-methylene blue indicator, titrate the NH_3 collected in the flask with 0.07 HCl. For smaller than 5 mg of nitrate, dilute the CHl of 0.007 N; the volume required is HCl. The normality used is N_{HCl}. The reaction during titration is:

$$NH_3 + HCl \longrightarrow NH_4\,Cl$$

11.3 .CALCULATIONS

Grams of nickel nitrate ($Ni(NO_3)_2$) in positive plate

$$W_{NNT} = \left(\frac{(V_{HCl})\,(N_{HCl})}{(1000)}\,\frac{182.8}{2}\right)\left(\frac{W_{PL}}{W_{sample}}\right) \qquad (31)$$

Grams of cadmium nitrate ($cd\,(NO_3)_2$) in negative plate

$$W_{CNT} = \left(\frac{(V_{HCl})\,(N_{HCl})}{(1000)}\right)\left(\frac{236.4}{2}\right)\left(\frac{W_{PL}}{W_{sample}}\right) \qquad (32)$$

12. ADDITIVES AND IMPURITIES

Detection and measurement of either purposely or inadvertently added materials can be accomplished using several instrumental and chemical techniques. Those additives of present interest included lithium, cobalt, and iron. These can be detected to 100 ppm, 10 ppm, and 10 ppm respectively, using emission spectroscopy. Once detected, atomic absorption is useful in making quantitative measurements, as is X-ray fluorescence.

13. ELECTROCHEMICAL TEST

13.1 PRINCIPLE

In order to correlate quantity of total active material as determined chemically in Sections 7 and 8 with quantity of useful active material available in the cell during operation, a series of electrochemical tests is required. The difference between the chemical and electrochemical tests is a measure of the quantity of inactive or unavailable active material.

13.2 MATERIALS

- Container—KOH-resistant, that can be placed in a nitrogen or inert gas environment during electrochemical test
- KOH—Thirty-one-percent solution, mercury cell grade or equivalent
- Three pairs of positive plates and three pairs of negative plates of aerospace quality to be used as opposing electrodes
- Reference electrodes—Six strips (0.5 in. by 2 in.) of positive plate with tab
- Open mesh rigid separator material (for example, Vexar) to place between plates to maintain separation
- Charge-discharge circuitry including power supply, ammeter, voltmeters, timer, and associated electrical parts

13.3 PROCEDURE

1. Positive plates (#6, #10, and #16) and negative plates (#7, #11, and #17) are selected from the same pack as those on which chemical analysis was performed (see Sections 7 and 8).
2. Weigh each plate to the nearest 0.01 g and measure thickness of each in three places to nearest 0.1 mil.
3. Prepare a strip 4 to 6 mils thick of pure nickel stock (4 by 3/4 in.), clean with ethanol, and spot weld to the tab of each plate. Stamp tab with designated plate number. Do the same for the six reference electrodes.

4. Place each negative plate between two positive plates serving as opposing electrodes and each positive between two negative plates. Position each three-plate pack in a separate KOH-resistant container with KOH-resistant separator material (Vexar) physically separating the plates. Add one reference electrode to each cell.

5. Fill each cell with 31-percent KOH solution so that all plates are submerged in the electrolyte.

6. Cover containers, place in inert gas environment, and make electrical connections.

7. Electrically connect to charge-discharge circuitry. Capability for monitoring current, time, and voltages of each of the six-plate packs is required. Low and high voltage protection is desirable.

8. Discharge each of the six containers at the C/2* rate based on nominal plate capacity (that is, for a 6-amp cell containing 10 negative plates the rate is 0.3 amps). The positive plate should fail immediately. The negative plate will exhibit some capacity which is a measure of the precharge of the cell. For this reason it is important that the contact time of the negative plates with air be minimized during all handling processes.

<u>NOTE</u>: Ensure that the test plate is failing and not the opposing plates or reference electrode.

9. Perform the following charge-discharge cycles on the test packs. Use rate based on nominal plate capacity.

   (a) Charge at C/10 for 16 hr; discharge at C/2 to failure

   (b) Charge at C/7 for 8 hr; discharge at C/2 to failure of test electrode

   (c) Repeat b

   (d) Repeat b

   Open circuit stand between charge and discharge must be kept to a minimum.

---

* C/2 refers to the current to be used and is found by dividing the nominal cell capacity by 2.

10. Record ampere-hours charge; record ampere-hours discharge.

13.4 CALCULATIONS

Excess negative capacity

= capacity of negative - capacity of positive

Precharge

= ampere-hours of discharge (procedure 8, Section 13)

14. PHYSICAL MEASUREMENTS

The following techniques can be used for analysis of physical properties of positive and negative plates. The physical properties of the materials have been compromised to some extent when extracting in hot water. However, from a practical standpoint it is difficult to make physical measurements in the "as removed" condition. Some experimental work has been done on the plate materials using the following techniques:

(a) Surface area

- BET—gas absorption
- Double layer capacitance

(b) Pore size distribution

- Mercury intrusion
- Volumetric with water or other solvent

(c) Crystal structure

- X-ray diffraction

(d) Mechanical strength

- Four point bend test*

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* Described in final report Tyco Labs, Contract NAS-5-11561, July 1969.

(e) Thermal properties and hydration

- Differential thermal analysis
- Thermogravimetric analysis
- Calorimetry

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APPENDIX A

DATA SHEETS

FOR

ANALYSIS OF NICKEL-CADMIUM CELL MATERIALS

Date of Analysis ____________

Cell Serial Number ____________ Project Name (satellite) ____________

1. Manufacturer Data and Cell History

Manufacturer ____________ Cell Size ____________

Catalog Number ____________ Date of Manufacture ____________

Signal Electrode ____________ Date of Activation ____________

Where Tested ____________ Date of Test ____________

Battery Test Group ____________

2. Visual Mechanical Inspection of Cell

Observations ____________

Cell Dimensions ____________ Weight ____________

Length ____________ Thickness ____________

Height ____________ Seal Type Manufacturer ____________

3. Gas Sampling

Pressure in Cell ____________ Estimated Cell Volume _________

Sample Holder Volume ______________________________________

Hydrogen __________ Oxygen _______ Nitrogen _______________

4. Separator Sampling

Sample Size (1) ________ (2) ________ (3) ________
(L) (W) (L) (W) (L) (W)

Separator Wet Weight (1) ________ (2) ________ (3) ________

Dry Weight (from V-6) (1) ________ (2) ________ (3) ________

Electrolyte Weight (1) ________ (2) ________ (3) ________

5. Analysis of Separator Sample for OH^- and $CO_3^=$

Normality of HCl to first endpoint ______________________________

Volume of acid (1) ________ (2) ________ (3) ________ AVG _____

Normality of HCl solution to M.O. endpoint ______________________

Volume of acid (1) ________ (2) ________ (3) ________ AVG _____

Calculated grams of KOH (Equation (1)) ______________

Calculated grams of K_2CO_3 (Equation (2)) ____________

Total as KOH (Equation (3)) _______________________

Equivalent volume of 31-percent KOH (Equation (4)) _______________

6. Electrolyte Extract Analysis

Normality of HCl solution (to first endpoint) 0.1 ____________

Volume of acid (1) ________ (2) ________ (3) ________ AVG ______

Normality of HCl solution (to second point) 0.01 ____________

Volume of acid (1) ________ (2) ________ (3) ________ AVG ______

Calculated grams of KOH (Equation (5)) ________________

Calculated grams of K_2CO_3 (Equation (6)) ______________

Total as KOH (Equation (7)) ________________________

Equivalent ml of 31-percent KOH (Equation (5)) __________

7. Plate Identification, Weight, and Thickness

Plate Number	Weight	Thickness		
		Top	Middle	Bottom
Neg-1				
Neg-3				
Neg-5				
Neg-7				
Neg-9				
Neg-11				
Neg-13				
Neg-14				
Neg-15				
Neg-17				
Neg-19				
AVG				
Pos-2				
Pos-4				
Pos-6				
Pos-8				
Pos-10				
Pos-12				
Pos-14				
Pos-16				
AVG				

8. Analysis of Negative Plate

Weight of plate without tab (1) ______ (2) ______ (3) ______

Weight of disk (W_s) (1) ______ (2) ______ (3) ______

Thickness of disk (1) ______ (2) ______ (3) ______

Volume of extractor solution 500 ml ______

Aliquot 10 ml ______

Volume of EDTA added (V_{EDTA}) (1) ______ (2) ______ (3) ______

Weight of cadmium hydroxide in plate (Equation (9)) ______

Ampere-hour equivalent of cadmium hydroxide in plate
(Equation (10)) ______

Ampere-hour equivalent of cadmium hydroxide in cell
(Equation (11)) ______

Weight of dry disks (1) ______ (2) ______ (3) ______

Volume of solution 250 ml ______

Aliquot 10 ml ______

Volume of EDTA used (V'_{EDTA}) (1) ______ (2) ______ (3) ______

Grams of charged material in plate (Equation (12)) ______

Ampere-hour equivalent of charged material in plate
(Equation (13)) ______

Ampere-hour equivalent of charged material in cell
(Equation (14)) ______

Ampere-hour equivalent of total negative ______

9(a). Analysis of Positive Plate—Charged Material Analysis

Plate weight without tab (W_p) (1) ______ (2) ______ (3) ______

Plate thickness (1) ______ (2) ______ (3) ______

Weight of sample (W_s) (1) ______ (2) ______ (3) ______

Weight of substrate (W_{ss}) (1) ______ (2) ______ (3) ______

Weight of sinter and active material in plate (W_a) (Equation (16)) (1) ______ (2) ______ (3) ______

Weight of nickel sample (W_{psc}) (1) ______ (2) ______ (3) ______

Volume of 10-percent acetic acid 100 ml ______

Weight of ferrous ammonium filtrate (W_{gas}) (1) ______ (2) ______ (3) ______

Volume of $KMNO_4$ used (V_{perm}) (1) ______ (2) ______ (3) ______

Grams of charged material in plate (Equation (17)) ______

Ampere-hour equivalent of charged positive material in plate (Equation (18)) (1) ______ (2) ______ (3) ______

Ampere-hour equivalent of charged material in cell (Equation (19)) ☐

9(b). Analysis of Positive Plate—Total Active Material

Weight of sample (W_{pst}) ____________

Molarity of EDTA 0.01 ____________

Volume of EDTA used (V_{EDTA}) (1) ______ (2) ______ (3) ______

Grams of active material as $Ni(OH)_2$ (Equation (20)) (1) ______ (2) ______ (3) ______

Grams of $Ni(OH)_2$ discharged in plate (Equation (21)) (1) ______ (2) ______ (3) ______

Ampere-hour equivalent of discharged material in plate (Equation (22)) ____________

Ampere-hour equivalent of discharged material in plate (Equation (23)) ☐

Total capacity of positive material (Equation (24)) ☐

Volume of EDTA for metallic Ni(V'_{DDA}) ____________

Grams of metallic nickel (Equation (25)) ____________

Volume of sample ____________

Porosity (Equation (26)) ____________

10. Carbonate Analysis

Project ID ____________ Plate (N) (P)

Cell lot and serial number ____________

Plate weight ________, ________, ________

TITRATION DATA

Phenolphthalein endpoint ________, ________, ________

Methyl orange endpoint ________, ________, ________

Normality of acid phenol ____________

Normality of acid methyl ____________

Total Volume of sample ________, ________, ________

Aliquot volume ________, ________, ________

Grams of carbonate K_2CO_3 in sample ________, ________, ________

Grams of carbonate K_2CO_3 in plate ________, ________, ________

Grams of carbonate K_2CO_3 in cell ________, ________, ________

GAS PARTITIONER DATA

Peak height ____________ Mv range ____________

Grams of $NiCO_3$ or $CdCO_3$ in sample from standard curve

________, ________, ________

	Cd (-)	Ni (+)
Grams of carbonate in sample	________	________
Grams of carbonate in plate	________	________
Grams of carbonate in cell	________	________
Percent carbonate in plate	________	________

11. Nitrate Analysis

	Cd (-)	Ni (+)
Grams nitrate in sample		
Grams nitrate in plate		
Grams nitrate in cell		
Percent nitrate in plate		

12. Additives and Inpurities

	Cd (-)	Ni (+)
Cobalt		
Lithium		
Iron		
Other		

13. Electrochemical Analysis (flooded)

Residual capacity negative (C/2) (precharge) ____________________

Residual capacity positive (C/2) ____________________

Capacity negative (Chg C/7 Dischg C/2) ____________________

Capacity positive (Chg C/2 Dischg C/2) ____________________

Negative/positive ratio ____________________

14. Physical Measurements

	Cd (-)	Ni (+)
Surface area		
Maximum pore size		
Pore volume		
Mechanical strength		

APPENDIX B

SUMMARY REPORT OF

NICKEL-CADMIUM CELL ANALYSIS

Project ______________ Date of Report ______________

Manufacturer ______________ Catalog Number ______________ S/N ________

Nom. Capacity ______________ Date of Activation ______________

Total Electrolyte and Carbonate in Separator

Grams KOH ______________

Grams $CO_3^=$ ______________

Total Electrolyte and Carbonate Extracted from Cell Pack

Grams KOH ______________

Grams $CO_3^=$ ______________

Total electrolyte volume as 31-percent KOH ______________ ml

Plate Measurements	Cd (-)	Ni (+)
Weight (avg)		
Thickness (avg)		

Chemical Analysis	Cd (-)	Ni (+)
Ampere-hours charged material		
Ampere-hours discharged material		
Total capacity		
Negative/positive ratio		
Percent carbonate in plate		

Electrochemical Analysis	Cd (-)	Ni (+)
Ampere-hours charged material (precharge)		
Ampere-hours discharged material		
Negative/positive ratio		

Comments