

Fabrication Procedure for Lithium-ion Rechargeable Coin Cells

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Disclaimer

The following procedures were developed at LBNL specifically for making electrodes and batteries consisting of LiMn_2O_4 (spinel) and MCMB (meso-carbon micro beads) graphite for high-power applications (HEVs). Electrode performance can be very dependent on the active and inactive materials used. The procedures documented here are continuously being refined; therefore, this publication should be considered a work in progress. More details of these procedures can be found in the lab notebooks of Gao Liu and Honghe Zheng.

I. Electrode Preparation

Cathode and Anode Using an NMP-Based Slurry:

(Differences for the anode appear parenthetically in [blue](#). If possible, the anode and cathode should be made in separate areas with separate pieces of equipment to prevent cross contamination.)

Materials

- Active material: $\text{Li}_{1.14}\text{Mn}_{1.86}\text{O}_4$ [Toda M809] ([Graphite \[MCMB 10-28, Osaka Gas\]](#))
- Conductive carbon: Acetylene black [Denka black]
- Binder: PVdF [Kureha 1100 (powder)]
- Solvent: NMP
- Current collector: Battery-grade aluminum ([copper](#)) foil

Equipment

- Vacuum oven
- Glove box with vacuum oven attached
- Homogenizer: Polytron PT10-35
- 50 ml jar
- Viscometer (optional)
- Doctor blade (Yoshimitsu, model YOA-B)
- Variable-speed, vacuum drawdown coater w/heatable table (optional)
- Napkins: Kimwipes
- Dowel: 20 cm long x 3 cm in diameter
- Calendering machine/roll press (International Rolling Mills)
- Scale: Metler balance
- Micrometer

- Punches: 1/2", 9/16", 11/16" and 15 mm
- Plastic hammer
- Small jars: (> 13/16" dia) for storing punched electrodes

Estimated time: Overnight drying + 2.5 hr + overnight drying + 30 min. + overnight drying

1) Determine Cathode Composition

- The cathode (**anode**) material should contain active material, binder, and conductive carbon. The formulation provided in the table below is specific to the cathode (**anode**) material used and is based on our previous work on electrode optimization.¹

Example Cathode Composition (wt%)		
Active material	Toda Spinel M809	85.6%
Binder	Kureha 1100 PVdF	8%
Conductive carbon	Denka Acetylene Black	6.4%

Example Anode Composition (wt%)		
Active material	MCMB 10-28 (Osaka Gas)	82%
Binder	Kureha 1100 PVdF	15%
Conductive carbon	Denka Acetylene Black	3%

2) Pre-treat Inactive Materials

- Dry binder (powder form) at 120°C under high vacuum in an oven for 12 hours.
- Dry the acetylene black at 160°C under high vacuum for 16 hours.
- Store in glove box.²

3) Prepare Slurry

Perform the following procedures in a glove box with a vacuum oven attached or in a dry room.

- Mix 10 g of the Toda M809 (**MCMB graphite**) active material powder and 0.75 g (0.44 g) of conductive carbon in 10 g (15 g) NMP solvent.
- Mix the slurry using the homognizer at a rate of 6000 rpm for 15 minutes.
- Add 0.94 g (1.76 g) of PVdF into the well-mixed slurry of Step ii.
- Homogenize the slurry again at a rate of 4000 rpm for 20 minutes to completely dissolve the PVdF into the NMP and produce a uniform, viscous slurry.³

Example Cathode Slurry Composition (wt)		
Active material	Toda Spinel M809	10.0 g
Conductive carbon	Danka Acetylene Black	0.75 g
Solvent	NMP, anhydrous	10.0 g
Binder	Kureha 1100 PVdF	0.94 g

¹ Effects of inactive material composition on the discharge and cycling performance of MCMB/Li[Ni_{1/3}Mn_{1/3}Co_{1/3}]O₂ cells, 215th ECS meeting, San Francisco, USA, May 24, 2009.

² Exclusion of water in these early stages is critical as the polymer gels if water is present.

³ The order of mixing is critical for distributing acetylene black in the electrode matrix, as can be observed in a paper to be published and slides from the 2010 DOE Annual Merit Review.

Example Anode Slurry Composition (wt)		
Active material	MCMB10-28 (Osaka Gas,	10.0 g
Conductive carbon	Denka Acetylene Black	0.44 g
Solvent	NMP, anhydrous	15 g
Binder	Kureha 1100 PVdF (5%)	1.76 g

- v. Observe/measure the viscosity of the slurry using a viscometer. Make sure the viscosity of the slurry falls in the range of 1200 to 500 (1400 to 600) cPa-s at a shear rate of 3 to 8 s⁻¹. (Adjust the viscosity of the slurry if it is too thick or too thin by, respectively, adding more NMP or evaporating out the NMP by putting it under vacuum).
- vi. After adding or removing the NMP, stir the slurry for an additional 5 minutes at 6000 rpm.
- vii. Remove bubbles in the slurry by putting it under vacuum for 2 min.

4) Prepare Current Collector

Perform the following procedures in a glove box or a dry room.

- i. Set the casting speed of the drawdown coater between 20 and 80 cm per minute.⁴
 - a) A casting speed of 30 (20) cm/min has been adopted for the *spinel* (MCMB) slurry of 700 (900) cPa-s.

Example Current Collector Parameters for Cathode	
Mitutato blade height	180 μm
Casting machine speed	30 cm/min
Current collector dimensions (Al)	1 m x 30 cm x 28 μm

Example Current Collector Parameters for Anode	
Mitutato blade height	120 μm
Casting machine speed	20 cm/min
Current collector dimensions (Cu)	1 m x 30 cm x 18 μm

- ii. With a napkin, spread a small amount of NMP solvent on the perforated glass table top of the casting machine.⁵
- iii. Spread Al (Cu) foil over the slightly dampened table. Turn on the vacuum. The Al (Cu) foil should be tightly held to the glass by the vacuum and trace NMP.
- iv. Using a smooth dowel of approx. 3 cm in diameter and 20 cm in length, smooth out the foil on the casting table. This may take approximately 5 minutes.
- v. Wipe the foil down with NMP to remove any residue from the manufacturing of the foil.⁶
- vi. Allow the NMP to evaporate before casting the electrode.

⁴ Casting speed is adjusted according to the slurry viscosity. Too high of a casting speed can result in an uneven slurry.

⁵ NMP helps in spreading the foil smoothly onto the casting table and for keeping the foil in place.

⁶ NMP also seems to improve the wettability of the slurry with the Al (Cu) foil.

Points of interest:

1. The current collector must be spread extremely flat onto the glass surface as you are trying to cast films on the foil that are around 0.1 mm thick.
2. Roughening the Al (Cu) foil surface improves the adhesion by increasing the contact area between the laminate and the current collector. Roughening of the metal foil surface can occur through sanding, chemical etching, or electrolytically depositing Al (Cu) onto the foil before casting the electrode. [Work in this area will soon be published.]

5) *Cast and Dry Electrode*

Perform the following procedures in a glove box or a dry room.

- i. Set the doctor blade to a height of 180 (120) μm .⁷
- ii. Before casting, perform one final mixing of the slurry for 1 minute using the homogenizer set at 1000 rpm.
- iii. Immediately pour the slurry uniformly across the front of the blade.⁸
- iv. Begin casting at a constant speed of 30 (20) cm/min for the slurry of ca. 700 (900) cPa-s viscosity.⁹
- v. After the casting arm has stopped, turn on the heater (if the table has this capability) and set it at 70°C to bake out the NMP.¹⁰
- vi. Further dry the laminate at 120°C for 12 hours under vacuum.¹¹

6) *Calender Electrode*

After the electrode is completely dried, it is compressed to a designated porosity, which is done according to the following procedures.

- i. Punch 3 representative 1/2" (9/16") circular cathodes. Record the weight and thickness of each.

Example Size Parameters	
Diameter	1/2 (9/16) inch
Loading	11.8 (4.4) mg/cm ²

- ii. *Optional:* Calculate the initial porosity of the laminate using the average electrode thickness, electrode weight per square centimeter, and the raw materials' densities.

⁷ The ratio of the height of the blade and final electrode thickness (dry state) usually comes out to be ca. 2.5:1 (2:1) for this cathode (anode) mixture.

⁸ The doctor blade height and the amount of slurry poured will dictate how much of the foil will be covered in slurry. For a doctor blade height of 0.018 (0.012) cm and width of 8 cm and a laminate that is 30 cm long, you'll need ca. 4.3 (2.9) ml of slurry.

⁹ The speed is adjusted according to the slurry viscosity. Too high of a casting speed may result in a laminate of sinusoidal thickness from beginning to end.

¹⁰ If the casting table does not have this feature, then allow the laminate to dry overnight. However, our experience has shown that direct heating leads to laminates of improved mechanical properties as compared with drying overnight.

¹¹ This process is to remove trace water and NMP solvent. Residual water can be detrimental to battery life; residual NMP can lead to delamination from the current collector.

- (The initial porosity of cathode typically varies from 40% to 60% depending on the active material used and the slurry composition, particularly, the amount of NMP.)
- iii. Define the target porosity of the electrode (typically 30 to 40% (25 to 35%) porosity for cathode (anode) materials). Determining the best thickness and porosity are part of the electrode design optimization process that is based on the application.) The target thickness of the laminate is calculated from the desired porosity, the weight of the laminate (minus the current collector), and the densities of the raw material components. The thickness of the electrode includes the current collector. (Typically, the target thickness of the laminate, without current collector, is 70 to 80% of its initial thickness).

Example Targets	
Target porosity	40% (35%) porosity
Electrode thickness	91 (60) μm

Electrode pressing is done in a glove box or a dry room using a calendering machine (International Rolling Mills). Temperature was not controlled during the following steps.¹²

- iv. Set the distance between the two rollers of the calendering machine to the thickness calculated in Step (iii).
- v. Set the rolling speed to 5 (equivalent to 15 cm/min).
- vi. Feed the electrodes through the rollers twice. After passing the laminate through the first time, rotate it from front to back before passing it through the second time to offset differences across the gap between the rollers.
- vii. Measure the thickness of the electrode using a micrometer. Repeat the process until the desired thickness is achieved.
- viii. Store the calendered laminate in an inert atmosphere glove box.

7) Punch Electrode

- i. In a glove box, cut out a strip of laminate to punch.
- ii. Transfer the following into the glove box:
 - a) hammer
 - b) punching tool
 - (a) 1/2" (9/16") for cathodes (anodes) for half-cell testing
 - (b) 9/16" (15 mm) for cathodes (anodes) for full-cell testing
 - c) labeled jar(s) for punched electrodes (make sure they are open in the antechamber to prevent release of air into the glove box).
- iii. Align the punch over the area that you want to punch out, then strike the top of the punch with the hammer.
- iv. Place the punched electrode in the jar.
- v. Continue punching electrodes until the entire strip is punched.
- vi. Dry the electrodes for 16 hours at 120 to 150°C under vacuum in the antechamber attached to the glove box.¹³

¹² High temperature calendering was found to increase the electronic resistance of the cathode yet improve the performance of the anode. Further research is underway.

- vii. After drying is complete, store the electrodes in the labeled jars back inside the glovebox.

Anode Using a Water-Based Slurry (SBR-CMC binder):

Substituted materials

- Active material: graphite (CGP - G8, Conoco Phillips)
- Binder: SBR (received in water at 40% conc.) and CMC powder
- Solvent: De-ionized water

Additional equipment

- Magnetic stirrer
- Stir bar

Note: a glove box should not be used.

Estimated time: Overnight stirring + 2 hr + overnight drying + 30 min. + overnight drying

1) *Define the Anode Composition*

- i. The anode should contain active material, CMC binder, and SBR binder. The following recipe is based on previous work and does not contain acetylene black.

Example Anode Composition (wt%)		
Active Material	CGP-G8 (Philips)	94%
SBR	Hydro Quebec	2%
CMC	Hydro Quebec	4%

2) *Pre-treat Inactive Materials*

- i. Dissolve 2 g of CMC powder into 98 g of deionized H₂O and thoroughly stir overnight with a magnetic stir bar so that the polyelectrolyte completely dissolves in the water. Store excess 2% CMC solution in a refrigerator at around 4°C for long-term storage.
- ii. Use SBR emulsion (40 wt%) as received.

Steps 3 to 5 below should be performed in a lab under ambient conditions.

3) *Prepare Slurry*

- i. Mix 5 g of the CGP-G8 graphite into 10.63 g of the CMC solution using a homogenizer set at 4000 rpm for 30 minutes.
- ii. Observe/measure the viscosity of the slurry; make sure the viscosity of the slurry falls in the range of 1200 to 500 cPa-s at a shear rate of 3 to 8 s⁻¹. (Adjust the viscosity of

¹³ Lower temperatures cannot remove the trace water while higher temperatures may bring about a conformational transition of the PVdF (its melting temperature is *ca.* 170°C.)

- the slurry if it is too thick or too thin by, respectively, adding more water or evaporating out some water under vacuum).
- iii. Add 0.26 g of the SBR emulsion into the mixture.
 - iv. Mix the slurry using the homogenizer at a rate of 4000 rpm for 10 minutes. The SBR should be well-distributed in the slurry.
 - v. Remove bubbles within the slurry by placing under vacuum for 1 min.

Example Slurry Composition (wt)		
Active Material	CGP-G8 (Philips)	5.0 g
SBR	Hydro Quebec (40% emulsion)	0.26 g
CMC	Hydro Quebec (2% solution)	10.63 g

4) Prepare Current Collector¹⁴

- i. Set the casting speed to 1 (around 100 cm per minute) for the slurry of around 600 cPa-s at 8 s⁻¹ shear rate.
- ii. With a napkin, spread a small amount of NMP solvent on the perforated table top of the casting machine.
- iii. Spread the copper foil over the slightly dampened table. Turn on the table vacuum.
- iv. Using a smooth dowel of approx. 3 cm in diameter, smooth out the foil on the casting table. This may take approximately 5 minutes.
- v. Wipe the foil down with 0.1 M HCl solution to remove any residue from the manufacturing of the foil. Then, remove the acid solution by rinsing with de-ionized water 5 times.¹⁵
- vi. Allow the foil to dry under ambient conditions before casting the slurry.

5) Cast and Dry Electrode

- i. Set the doctor blade at a height of 280 μm.¹⁶

Example Casting Parameters	
Mitutato blade height	280 μm
Casting machine speed	100 cm/min
Current collector dimensions (Cu)	1 m x 30 cm x 18 μm
Slurry volume (CGP-G8, SBR-CMC)	20 ml

- ii. Before casting, perform one final mixing of the slurry using the homogenizer set at 1000 rpm for 1 minute.
- iii. After stopping the homogenization, pour the slurry adjacent to the doctor blade holder and allow it to spread across the doctor blade.
- iv. Begin casting at a constant speed of 100 cm/min.

¹⁴ Explanations can be found in the section on “Cathode and Anode Using an NMP-based Slurry (PVdF)”, footnotes 4-6.

¹⁵ The water also seems to improve the wettability of the slurry with the Cu foil.

¹⁶ The ratio of the height of the blade and final electrode thickness (dry state) is *ca.* 3.5:1 for this anode mixture.

- v. After the casting arm has stopped, turn on the heater to 45°C to slowly bake out the water. If the coating table is not heatable, then allow the laminate to dry in place.
- vi. Once the laminate is visually dry, put it in a vacuum oven at 100°C for 12 hours under high vacuum to completely remove any trace water.

6) Calender Electrode

- i. Punch out three representative 9/16” circular anodes. Record the weight and thickness of each.

Example Size Parameters	
Diameter	9/16 inch
Active material loading	6.5 mg/cm ²

- ii. *Optional:* Calculate the initial porosity of the graphite laminate using the electrode thickness and weight per square centimeter, and the raw materials’ densities. Initial porosity of the graphite electrode typically varies from 45% to 60% depending on the active material and overall electrode composition.
- iii. Define a desired porosity for the electrode (typically 20 to 30% porosity for graphite anodes). The target thickness is calculated from the desired porosity, mass loading, and the densities of the starting materials. Typically, the target thickness of the laminate (without current collector) will be 60 to 70% of its initial thickness.

Example Targets	
CGP-G8 target porosity	25%
Total thickness with Cu foil	66 μm

Electrode calendering is done in a glove box or a dry room using a calendering machine.

Temperature was not controlled during the following steps.¹⁷

- iv. Set the distance between two rollers of the calendering machine to the distance calculated in Step (iii).
- v. Set the rolling speed to 5 (equivalent to 15 cm/min).
- vi. Feed the electrodes through the rollers twice.
- vii. Measure the thickness of the electrode using a micrometer. Repeat the process until the desired thickness is achieved.
- viii. Store the calendered laminate in an inert atmosphere glove box.

7) Punch Electrodes

- i. In a glove box, cut out a strip of laminate to punch.
- ii. Transfer the following into the glovebox:
 - a) plastic hammer
 - b) punching tool
 - (a) 9/16” for anodes in half cells

¹⁷ Research is under way to determine a calendering temperature that results in optimal electrochemical performance for the CMC/SBR-based electrode.

- (b) 15 mm diameter for anodes in full cells
 - (c) Labeled jar(s) for punched electrodes.
 - iii. Align the tip of the punch over the area that you want to punch out, and then hit the top with the hammer.
 - iv. Place the punched electrode in the jar.
 - v. Continue punching electrodes until the entire strip is punched.
 - vi. Dry the electrodes for 16 hours at 120°C (no more than 130°C) under vacuum in the antechamber attached to the glove box.¹⁸
 - vii. After heating is complete, store the electrodes in labeled jars inside the glove box.
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II. Coin Cell Assembly

Full-cells and Half-cells¹⁹:

(Differences in the manufacture of half-cells appear in parenthesis in green. Half-cells are distinguished from full-cells as they contain a lithium metal electrode.)

Materials

- Coin cell hardware: includes bottom cell casing, 11/16" stainless steel disc, spring, top enclosure, and insulating ring
- Anodes: punched graphite laminates (lithium foil for half-cells.)
- Cathodes: punched laminates
- Separator
- Electrolyte

Equipment

- Coin cell press
- Glovebox
- Latex gloves
- Balance
- Punches: 11/16" and 13/16"
- Pipette
- Plastic or electrically insulated tweezers.

Estimated time: 20 minutes (there is a 2 hr wait at the end to allow for wetting.)

¹⁸ A lower temperature cannot adequately remove the trace water while a higher temperature may bring about decomposition of the CMC and SBR binder.

¹⁹ There are two general rules for making lithium ion cells that must be followed:

1. Electrode area: The area of the anode (lithium) must be larger than that of the cathode by around 5 to 10%. Otherwise, alignment issues will result in premature cell failure.
2. Electrode capacity: The capacity of the anode should be greater than that of the cathode by 5 to 10% in full cells (this can be ignored for half-cells). Otherwise, the cell may be vulnerable to lithium deposition if over charged.

1) Prepare Equipment and Materials

- i. Open valve outside of the glove box that is used for pressurizing the coin cell press.²⁰
- ii. Place arms into the glove box. Use latex gloves over glovebox gloves when handling materials to avoid contamination.
- iii. Select (and prepare) electrodes
 - a) Use 9/16" for cathode and 15 mm dia. for anode.²¹
 - b) For half-cells, use 1/2" cathode or 9/16" for anode; use 11/16" lithium foil for counter electrode.
 - (a) Punch circular lithium electrodes out of lithium foil using the 11/16" punching tool.
 - (b) Flatten the lithium after punching.
 - (c) Center the lithium on top of a flat 11/16" stainless steel disc. The disc is part of the coin cell hardware. Press the lithium onto the disc.
- iv. Weigh each electrode on the balance.
- v. After weighing electrodes, calculate the capacity of each electrode to verify that the anode has 5 to 10% more capacity than the cathode.²²
- vi. Prepare the separator by using the 13/16" punch.

2) Assemble Cell

When placing components in the cell casing, avoid any contact with the cell wall, as this will short the cell, and attempt to place the components in the center of the assembly without having to move them once in the cell. Otherwise, this may adjust the relative position of the anode and cathode, resulting in incomplete overlap.

- i. Place cathode (or anode) into the bottom casing (larger of the two casings) of the coin cell.²³
 - a) Remove any dust by spraying the electrodes with a pressurized gas (argon) line.
 - b) Place the electrode into the can with the current collector facing down.
 - c) Using plastic or electrically insulated tweezers, center electrode within the metal casing.
- ii. Center the separator on top of the electrode.
- iii. Drop electrolyte onto the separator using a pipette.
 - a) Use 5 (4) drops (<100 μm thick electrodes) or 6 (5) drops (>100 μm electrodes).²⁴
 - b) Rotate and tilt the cell to get the electrolyte to fully wet the underside of the separator. Wetted parts will appear transparent.
- iv. Center the anode (lithium-covered stainless steel disc) facing downward and making contact with the separator.
- v. Center the disc on top of the anode (an additional disc is not necessary).

²⁰ This pneumatic press was purchased from the NRC of Canada for the sole purpose of doing battery research.

²¹ It is impossible to exactly line up two circular electrodes. To avoid issues of misalignment, the counter electrode is physically larger than the working electrode.

²² The weight is also used to estimate the cell capacity.

²³ The bottom casing consists of a more corrosion resistant steel designed specifically for 4 V cathodes. Do not use the top enclosure as the cathode, especially cathodes > 3V.

²⁴ Each drop contains *ca.* 0.025 ml of electrolyte.

- vi. Center the spring onto the stainless steel disc. The widest part of the spring should be in contact the stainless steel disc.
- vii. Press the insulating ring around the edge of the top enclosure of the cell.
- viii. Place the top enclosure (with O ring) into coin cell. Insulator should be separating the top enclosure from the bottom casing. Efforts should be made to assure that all parts of the cell remain centered, including the top enclosure.
- ix. Press the coin cell.
 - a) Open the valve above the press.
 - b) Place cell in press assembly without jarring the inner workings.
 - c) Make sure the press plate can rotate freely before pressing.
 - d) Open the valve on the press and press for 30 seconds.
 - e) Close the valve on the press.
 - f) Close the valve above the press.
- x. Close the argon valve outside of the glove box.
- xi. Begin cycling formation 2 hours after constructing the cell.²⁵

Gel Cells:

Additional materials

- o Cross-linking catalyst: Perkadox (stored in a refrigerator)
- o Polymer: PEO

Additional equipment

- o Small beakers
- o Refrigerator

Estimated time: 1.5 hours (goes directly to formation)

1) *Prepare Materials*

- i. Transfer a small amount of Perkadox from the refrigerator to a glove box.
- ii. Make a solution of 95 % wt. EC/DEC 1:2 and 5% wt. PEO polymer (Solution A). Make only a small amount because the solution has a short lifetime.
- iii. Mix Perkadox (1000 ppm of the polymer weight in Solution A) with Solution A. Stir carefully to prevent the solution from overheating. This new solution (Solution B) has an even shorter life and must be used immediately.

Example Solution B Composition		
Electrolyte	EC/DEC 1:2	1.9 g
Polymer	PEO (Hydro Quebec)	0.1 g
Cross-linking catalyst	Perkadox	0.1 mg

²⁵ Two hours allows for complete wetting by the electrolyte of each part in the cell. Longer wait times may result in corrosion of the copper current collector.

2) *Assemble Cell*

- i. Construct the cell according to the standard procedures for coin cells, using Solution B as the electrolyte.
 - ii. After pressing the cell, heat at 60°C for 1 hr under vacuum.
 - a) Add a few remaining drops of Solution B into a couple of coin-cell bottom casings.
 - b) Place the casings in the oven with the fabricated coin cell to assure one's self that a gel film forms.
 - c) Place the rest of Solution B into the oven to see if it polymerizes.
 - iii. Begin the formation process immediately after the 1 hour heating step.
 - iv. Return any extra Perkadox to a refrigerator, kept in a separate container.
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III. Coin Cell Formation and Characterization

Materials

- Coin cells

Equipment

- Environmental chamber
- Battery cycler: Maccor
- Impedance analyzer: Solartron 1260
- Potentiostat: Schlumberger 1286
- Computer

Formation:

Formation is performed to get the cell to a steady-state condition from where it starts when first assembled to where it will typically be operated.

Estimated time: 24 days

1) *Prepare Formation Procedure*

- i. Prior to testing a coin cell, prepare a formation procedure using the BuildTest program in the Maccor battery cycler.
 - (a) Our standard formation procedure is to perform two C/24 charge/discharge cycles followed by ten C/10 charge/discharge cycles.
- ii. Define appropriate current ranges within your procedure to make sure that no excess current passes through the cell.

2) *Run Formation Procedure*

- i. Select the appropriate environmental chamber at the appropriate formation temperature. Due to a limited number of environmental chambers and USABC testing requirements, it is probably best to do formation at 30°C.²⁶
- ii. Insert coin cell into coin cell fixture that is connected to leads for cycling.
- iii. Run the formation procedure on the cell's channel using the Maccor software. The procedure takes approximately 24 days.

3) *Collect & Analyze Data*

- i. Open the MIMS data viewing program from the Maccor software interface.
- ii. On the taskbar of the screen that appears, click "Options" then "View Data from File".
- iii. On the taskbar of the screen that appears, click "File" then "Save as Text".
- iv. Save the data in the appropriate location on the hard drive, flash drive, etc.
- v. Open the text file on a computer with the Excel program.
- vi. Copy the text into Excel.
- vii. Analyze the data, making calculations and graphs.

Example Coin Cell Results	
Cathode	Toda Spinel
Anode	Lithium
Electrolyte	LP40 (Ferro, America)
C-Rate	C/10
1 st Charge Capacity	1.23 mAh/cm ²
1 st Discharge Capacity	1.20 mAh/cm ²
1 st Cycle Efficiency	97.5%
Specific capacity	91 mAh/g

EV Characterization Test:

This test is designed to determine the highest, constant current a cell can withstand to full discharge without being limited by mass transport properties. This test is performed after formation.

Estimated time: 15 days

1) *Prepare Test Procedure*

- i. Prepare a procedure using the Maccor BuildTest program.
 - (a) The procedure should vary the C-rate on discharge, but keep it constant on charge. The standard procedure varies the discharge rate between C/10 and 20C and always charges at C/10.
 - (b) Define appropriate upper and lower cut-off voltages.

²⁶ One could have an environmental chamber dedicated to formation if a more optimum temperature were identified.

- (c) Decide whether to use a constant voltage charge (trickle charge) at the top of charge.
 - (d) Decide on an appropriate time to wait at top of charge at open circuit before discharging.
 - (e) Define appropriate current ranges within your procedure to make sure that the cell will not be exposed to excessive currents.
- ii. The discharge capacity obtained from the last formation cycle is used as the nominal capacity of the cell.

Example EV Cycling Procedure		
Cycle 1	C/10 Charge, C/10 Discharge	3 cycles
Cycle 2	C/10 Charge, C/5 Discharge	3 cycles
Cycle 3	C/10 Charge, C/2 Discharge	3 cycles
Cycle 4	C/10 Charge, C/1 Discharge	3 cycles
Cycle 5	C/10 Charge, 2C Discharge	3 cycles
Cycle 6	C/10 Charge, 5C Discharge	3 cycles
Cycle 7	C/10 Charge, 10C Discharge	3 cycles
Cycle 8	C/10 Charge, 20C Discharge	3 cycles
Cycle 9	C/10 Charge, C/10 Discharge	3 cycles

2) Run EV Procedure

- i. If the coin cell is not already in the appropriate oven and Maccor channel after formation, then correct the situation.
- ii. Run the procedure on the cell's channel using the Maccor software.
- iii. Collect and analyze data as described under the Formation section.

AC Impedance Test:

This test is designed to help determine the different contributions to impedance in a cell. AC impedance is typically performed sometime after Formation.

Estimated time: 1.25 hours

1) Set Cell State of Charge

- i. Determine the state of charge you want the cell to be at during the impedance test.
- ii. Using the Maccor software, equipment, and interface, prepare and run a procedure that sets the coin cell to the desired state of charge.
- iii. Let the coin cell rest for at least a few hours before running the impedance test.

2) Prepare Equipment

- i. Turn on the impedance analyzer (Solartron 1260) and potentiostat (Schlumberger 1286), as well as the related desktop computer.
- ii. Let the equipment rest for 30 minutes prior to running any tests.
- iii. Open the ZPlot 2 program.

- iv. Under the “Ctrl E: Sweep Freq” tab, choose the following parameters:
 - a) DC Potential = 0 V vs. Open Circuit
 - b) AC Amplitude = 10 mV
 - c) Initial Frequency = 100000
 - d) Final Frequency = 0.01
 - e) Logarithmic Steps
 - f) 10 Steps/Decade

3) *Run Impedance Test*

- i. Connect the cables from the Schlumberger 1286 and Solartron 1260 to the coin cell - use Anderson connectors, if possible.
- ii. On the ZPlot screen, click “Monitor Cell Potential”. Make sure the cell is at an appropriate voltage for the test.
- iii. If the cell is ready for testing, click “Measure” on the taskbar, then “Sweep”. The test should take about 10 minutes.
- iv. Once the test is complete, save the data to the proper location.

4) *View Data*

- i. During or after the test, view the data using the ZView 2 program.
- ii. Open ZView 2 and click on “File”, then “Data Files”.
- iii. Select all the files you would like to view, then click “OK”.
- iv. Collect and analyze data as described under the Formation section.

HPPC Test:

The HPPC test is designed to measure the resistance of the cell in increments of 10% DOD from a direct current applied for 10 seconds. This data can be used to calculate the energy available for delivering or receiving a pulse of power of a given magnitude. This data is used to size batteries for HEV applications. It should be performed after Formation.

Estimated time: 3 days

1) *Prepare Procedure*

- i. Prepare a procedure using the Maccor BuildTest program. See procedure file “scHPPC” for an example.
- ii. Define appropriate current ranges within your procedure to make sure that no excess current passes through the cell. It is especially important that the current be at the right level for this test because it will significantly impact the results of the test, since resistance is typically current dependent in an electrochemical cell.

2) *Run Procedure*

- i. Check that the cell is in the appropriate environmental chamber, connected to the selected channel.
- ii. Run the procedure on the cell’s channel using the Maccor software. The procedure should take about 3 days.
- iii. Collect and analyze data as described under the Formation section.