

## **Procedure for cathode coatings:**

-PVDF (Sigma-Aldrich 427152-100G) is dissolved in anhydrous 1-methyl—2-pyrrolidinone (Sigma-Aldrich 328634-1L) to obtain a concentration of 7.5 wt% NMP.

-Carbon (TIMCAL Super C65 Carbon Black, BET area: 62 m<sup>2</sup>/g) and LNMO is dispersed in the PVDF solution in the following ratio:

- 84wt% active material (LNMO).
- 8 wt% carbon.
- 8 wt% binder.

NMP should amount to around 70 wt% of the solid parts.

-The cathode slurry is shaken thoroughly for 10 minutes, and any bubbles are removed by suction.

-The slurry is then coated on double-sided carbon coated aluminium foil (MTI, 2+15+2µm) with a 100µm thickness.

-The coatings are dried at 80 °C under vacuum for 2 hours.

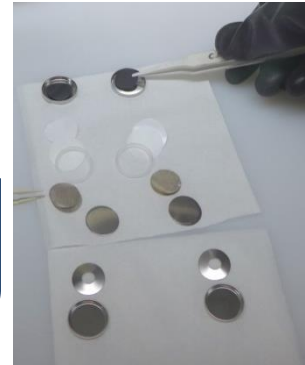
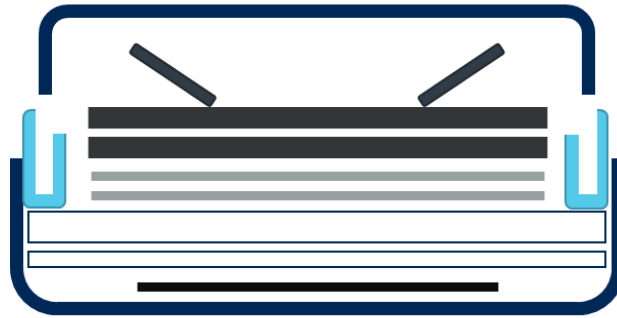
-14mm diameter electrodes are cut from coatings and weighed.

-The electrodes are pressed using 3 tonnes in a 20mm diameter die.

-Finally, the electrodes are dried under vacuum at 120 °C for 10 hours.

## Cell assembly

Casing (-)  
Spring  
2 spacers  
2 thin lithium  
O-ring  
100  $\mu$ L electrolyte  
Viledon separator  
Toray separator  
Cathode  
Casing (+)



Anode: Lithium metal

Electrolyte: 1 M LiPF<sub>6</sub> in 1:1 vol EC:DMC

Polymer separator: Viledon FS2192-11SG and Toray or Celgard.

Casing: Hohsen

Disc spring: MTI

## Constant current cycling:

Cycle	1-3	4-6	7-11	12-16	17-21	22-26	27-31	32-1031
Charge	0.2c	0.5c	0.5c	0.5c	0.5c	0.5c	0.5c	0.2c/0.5c
Limit	5 V, 10h	5 V, 3h	5 V, 3h	5 V, 3h	5 V, 3h	5 V, 3h	5 V, 3h	5 V, 8h/3h
Discharge	0.2c	0.2c	0.5c	1.0c	2.0c	5.0c	10c	0.2c/1.0c
Limit	3.5 V	3.5 V	3.5 V	3.5 V	3.5 V	3.5 V	3.5 V	3.5 V

## Comments

Rest 15 min between each discharge and charge.

Cycles 1-6 are formation cycles used to make a suited SEI layer on the anode and cathode surface.

Cycles 7-31 are the power test.

Cycles 32-6031 are the cycling test (1 cycle at 0.2c/0.2c and 19 cycles at 0.5c/1.0c), repeated 50 times. If this is complicated to set up with the tester, the 0.2c/0.2c cycles can be left out in the cycling test.

If the tester only allows one limit to the charge and discharge, the time limit can be left out.

## **Cyclic voltammetry:**

Run cycles 1-6 from the constant current cycling to ensure correct formation of the cells.

After this, cyclic voltammetry should be started within 30 minutes.

Use a scan rate of 20  $\mu\text{V/s}$ . This is slow a slow measurement (40h for 1 cycle), but it is needed to obtain good resolution of the two high voltage peaks. Charge to 5V and discharge to 3.5V

## **Electrochemical impedance spectroscopy:**

If you have a three-electrode cell, this is preferred, but it will be very difficult, if you have not tried it before!

Run cycles 1-6 from the constant current cycling to ensure correct formation of the cells.

Relax the cell until the voltage changes less than 2mV/h. If you cannot set this value, use a rest time of 5h.

Measure the impedance

The spectrum should be measured at OCV

Frequency: 0.1 Hz - 100 kHz

Amplitude: 10 mV

No. of frequencies: 15 pts/decade (3 measurements per frequency is recommended)

Charge at 0.5h at 1C to approximately 50% SOC and relax the cell as before

Measure the impedance

Charge at 0.5h at 1C to approximately 50% SOC and relax the cell as before

Measure the impedance

Charge to 4.9V at 1C and relax the cell as before

Measure the impedance

Charge to 4.9V at 1C again to remove even more lithium from the cathode and relax the cell as before

Measure the impedance

If you have time, try to cycle the battery 20 or 100 times at 1C and do the EIS measurement again.