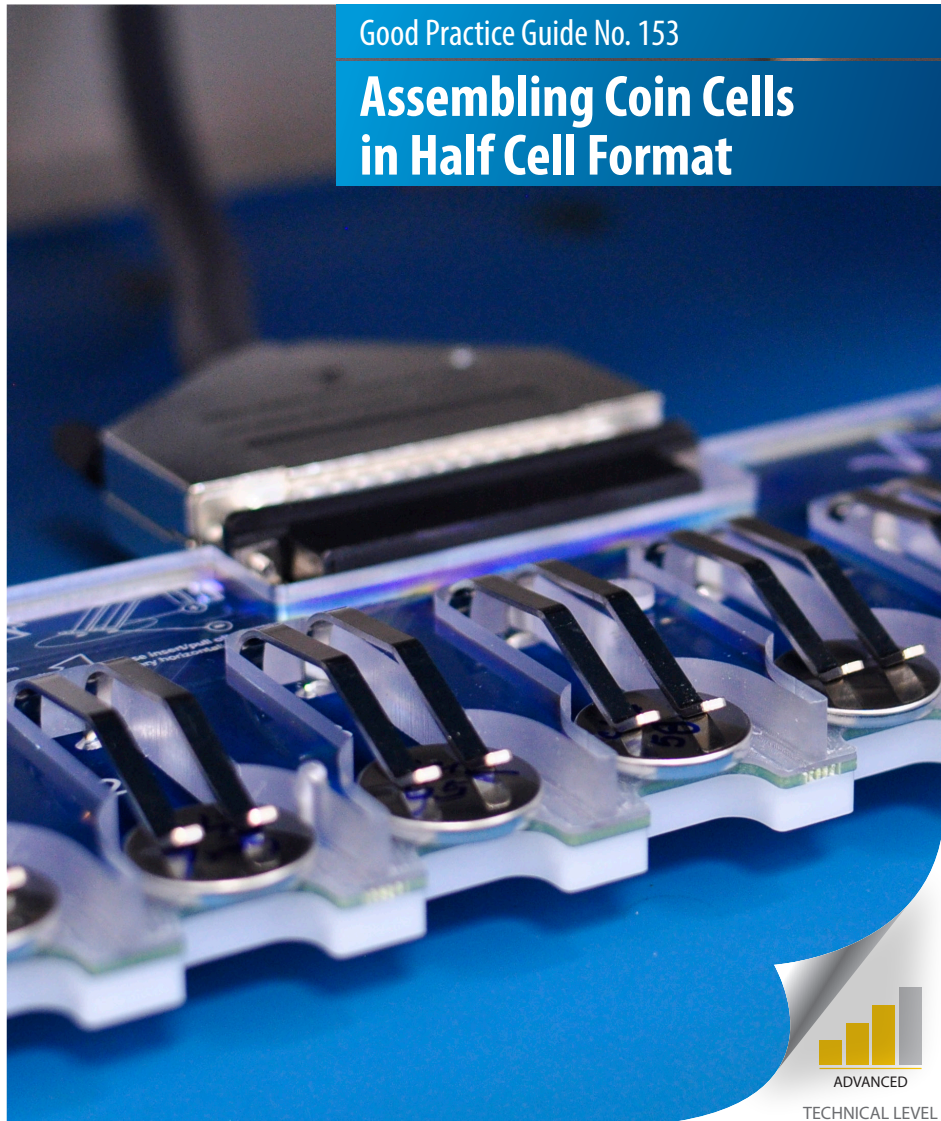




National Physical Laboratory

Good Practice Guide No. 153

Assembling Coin Cells in Half Cell Format



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Assembling Coin Cells in Half Cell Format

NPL Good Practice Guide

This guide outlines best practice in the assembly of coin cells in half cell format. It is intended as an introductory text for researchers working on battery science, whether entering the field for the first time or those with more experience.



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1 Introduction

- Scope
- Introductory Remarks

1.1 Scope

The coin cell phase form is the most used format for the electrochemical evaluation of materials and components for rechargeable batteries. It has many advantages; however, there is a misconception that its compact form and its ubiquity make it easy to assemble and test and many newcomers to the field struggle with the generation of reproducible data, especially when attempting interlaboratory comparisons.

To address this issue, this good practice guide offers a comprehensive description of all the necessary steps for the repeatable and reproducible production of coin cells in half-cell configuration. It assumes that the reader has, as a minimum, an introductory level of knowledge in battery science and has taken university courses in general chemistry, general physics, and basic algebra. The overarching purpose of this guide is to facilitate the production of uniform batches of cells, and to create awareness of the main sources of variability and the pitfalls encountered when assembling coin cells.

We have chosen to limit this guide to the half-cell configuration. In a lithium half-cell, the negative electrode is in the metallic form (zero oxidation state); this means that this electrode, compared to the mass of the positive electrode, provides a *de facto* infinite source of active cation, specifically lithium ion (Li^+). This detail is particularly important when evaluating the initial practical capacity of newly synthesised electrodes, and the main reason why materials discovery relies heavily on the half-cell format. Additionally, the electrochemical behaviour of lithium metal, especially in liquid electrolytes, is well understood, which simplifies the process of elucidating the causes of the electrochemical response of new materials. ^(1-3, 7)

The content herein covers several aspects, from detailed technical instructions to the more mundane logistics and organisation of the various steps of the process. In that way, this guide can serve either as a teaching aid to train new users, or as a desk instruction that can be consulted by more experienced users, when necessary, to maintain a uniform method of cell assembly.

In addition to detailed instructions for planning and performing coin cell assembly work, this guide contains a glossary of terms (Section 5.1), extensive instructional graphics such as photographs, diagrams, and videos, a few key references that can serve as additional information for readers (Section 7), and a downloadable Excel file containing an example of a data and metadata spreadsheet (Section 3.1.1).

Please notice that this good practice guide can be cited as: *Lopez et al., NPL Good Practice Guide 153, DOI: <https://doi.org/10.47120/npl.mgpg153>, 2024*

1.2 Introductory Remarks

A well-prepared electrochemical cell can yield a plethora of results that can support further device and materials development. The only absolute requirements are having a good understanding of what constitutes acceptable cell to cell variability, and a systematic way to compare results between tests.

Coin cells, also known as button cells, are a compact, circular cell format usually available as primary (non-rechargeable) cells; they are commonly used in portable electronics such as calculators, hearing aids, and wearable devices. Coin cells made with high-quality components are also suitable to be used as secondary (rechargeable) cells for powering portable electronics and for research purposes.

As a widely used commercial product, coin cells have advantages over other lab cells such as Swagelok, beaker-type cells, or other in-house made cells, mainly in their reproducibility, in the relative ease of maintaining internal cell pressure, in providing a good replica of the electrode-electrolyte interface and electrode separation of larger cell formats, and in the availability of high-quality, relatively inexpensive components. Compared to larger cell formats such as cylindrical and pouch, the advantages lie mostly in sustainability and efficiency gains due to the ability to carry out a large number of tests with much lower use of materials and chemicals, reduced electricity usage, much smaller footprint required for testing, and less waste when disposing of the cells after cycling. These aspects make coin cells especially useful for the electrochemical evaluation of materials in the early stages of development.

With the rising worldwide interest in the discovery of more sustainable battery materials, a high number of new players have entered the battery components market. Unfortunately, a standard for coin cell battery components has not been adopted by many manufacturers, creating issues with the quality of supplies. At the same time, new generations of researchers coming from diverse areas of science are attempting to work with coin cells, often without rigorous and systematic training. Furthermore, every research laboratory has their own bespoke way of assembling cells, which usually results in moderate to low repeatability of measurement and poor lab to lab reproducibility.

The popularity of coin cells in the research community can be ascertained from the many publications that routinely report using them, but also by the availability of peer-reviewed papers reporting best practice on the assembly, optimisation, and cycling parameters for this cell format.^(4-6, 9) Although these works are highly useful, they are mostly focused on repeatability within the same laboratory. Others report round robins in which the participants measure the same cells or batches of the cells with the same chemistries in different labs. However, there is still an unaddressed need for process refinement and to go beyond the best possible outcomes to describe specific sources of variability. Furthermore, the importance of systematic recording of observational metadata that can help identify these sources is not

sufficiently emphasised in the scientific literature. This good practice guide aims to address that gap.

2 Preliminaries

- Preparation
- Equipment

A high-level procedure for the good practice of assembling coin cells in the half-cell format is described schematically in figure 1, section 2.1. In devising this procedure, we have assumed a wide range of hands-on experience for different users, and we have, therefore, included all the possible steps that are necessary for producing the cells.

We describe the process of cell assembly referring to the coin cell format CR 2032, but this process can also be used for the CR 2016 format, which is a similar but thinner coin cell format. Unless otherwise specified, whenever we mention coin cell, we are referring to the CR 2032 format. Taller button cells with a distinctive cylindrical shape and no jelly roll are out of the scope of this guide.

This guide assumes that the cells will be prepared in a glove box with argon (Ar) atmosphere. In principle, cells could also be prepared in a dry room; however, as that would only protect the electrodes from moisture and not oxygen or nitrogen, an Ar glove box is preferred. While many materials are stable in dry room conditions, metallic lithium electrodes used in the half cell configuration have a high rate of reaction with nitrogen; hence, preparation of half cells in a glove box is preferred to avoid excessive contamination. Whenever possible, we also recommend working in a glove box with a nitrogen gas sensor and a nitrogen filter to provide even better control of native surface layers.

It is also assumed that the equipment necessary for the process is shared between various users, and that the users have been fully trained in the proper operation, safety, and maintenance of an Ar glove box and other equipment.

2.1 Preparation

The efficiency in performing any complex process is determined by adequate and timely preparation. This requires an explicit work plan that tells users when, with what resources, why, and in what order the different steps should be performed.

In this guide we chose to organise these steps considering the relative amount of time that each takes and what is the most productive way to perform them. A high level, four step sequence, as illustrated in figure 1, is described below.



Figure 1: High-level procedure for the good practice of assembling coin cells in the half-cell format. Note the organisation of steps, with planning at the top of the hierarchy, moving from top left to bottom right.

- **STEP 1: PLANNING** It is important that, at least one week, sometimes several weeks before the actual assembly, users make sure they reserve the equipment that they will use including vacuum ovens, the glove box, vacuum chambers, and any other shared equipment. Additionally, users should verify that all the components and chemicals needed for the process are in the glove box, and that the data and metadata spreadsheet is set up and ready to use. Verify as well that there is an adequate supply of gas for maintaining atmosphere but also for purging the glove box after the work, in case of a solvent spill, or if there is a need to use a large transfer chamber.
- **STEP 2: ASSEMBLING** With proper preparation this stage should go smoothly but always allow extra time to account for mistakes, unexpected occurrences, or user fatigue.
- **STEP 3: CLEANING** Allow enough time to clean up after cell assembly is completed. It is polite to keep a tidy work area, but it is also important for health and safety reasons.
- **STEP 4: CONNECTING** Even the most perfect cell can exhibit errors in its electrochemical response if the electrical connections to the cyclers are faulty. Always check electrical connections and the state of coin cell holders immediately before cycling, and keep a regular maintenance schedule.

2.2 Equipment

Table A provides a list of all the usual materials and equipment required to assemble coin cells. In most cases we have provided photos of preferred equipment, but these are intended as examples and are not prescriptive (e.g., figures 2, 3 and 5). When a photo is available for the preferred equipment, the panel number in figures 2, 3 and 5 is indexed to the corresponding item number in Table A.

With the increase in the interest in battery technology there has been an increase of suppliers and equipment, especially for cell fabrication. It should be noted that although specifications appear similar at first glance, not all providers have the same quality requirements.

Before buying equipment and cell hardware, the user should ask the providers for the tolerances and/or errors associated with their specifications. For coin cell hardware and coin cell crimpers, it is of the utmost importance **NOT TO MIX AND MATCH PARTS** from different manufacturers.

Please see section 3.1.1 “To do at least several weeks before assembly” for more details.

Please note that, whenever possible, all equipment should be calibrated to traceable standards.

Table A: Equipment and Specifications

Item Number	Equipment Description	Preferred specifications and/or manufacturer	Figure/Panel
1	Glove box with Ar atmosphere	O ₂ < 0.1 ppm, H ₂ O < 0.1 ppm, N ₂ filter, solvent trap	2/1
2	Coin cell crimper	---	---
2.1	Automatic	Hohsen or similar	2/2.1
2.2	Manual	Hohsen or similar	2/2.2
3	Coin cell hardware (CR 2032)	Hohsen or similar	2/3
3.1	Can (base)	Al-coated inside can	2/3.1
3.2	Wave washer (Ring)	---	2/3.2,3,6
3.3	Spacers	16 mm diameter, 0.3 mm thick, or with a soldered wave washer	2/3.3a, b
3.4	Gasket	Hohsen, separate	2/3.4
3.5	Cap	Hohsen, gasket free	2/3.5
4	Electrodes	---	2/4
4.1	Positive Electrode (Cathode)	commercial laminate sheet	2/4 (right side)
4.2	Negative Electrode (Anode) - Lithium metal (aka metallic lithium)	battery-grade foil, with smooth surface finish	2/4 (left side)
5	Separator	trilayer, PP:PE:PP, Celgard 2325 or similar, if drying use < 50 °C	2/5 (insert)
6	Electrolyte	battery-grade, Solvay, Ube Corp., Solvionics or similar	2/6
7	Micropipette	auto pipette, calibrated	2/7
7.1	Micropipette tips	when possible, from same manufacturer as the pipette, auto pipette, calibrated	2/7.1
8	Disposable gloves	---	---
9	Lint-free wipes	Kimwipe or similar	3/9
10	Electrode punch to use only for Lithium	---	3/10
10.1	Electrode punch for positive electrodes and separator, manual + mallet	---	3/10.1, 10.11
10.2	Hand operated die cutter	---	3/10.2
11	Plastic base to punch electrodes	PEEK or POM preferred, avoid PTFE	3/10.11
12	Coin cell assembly mould	---	3/12, 12.1
13	Small plastic bags or boxes	---	3/13, 13.1, 13.2
14	Multimeter	Fluke or similar	5/14
15	Non-metal tweezers	---	5/15

Item Number	Equipment Description	Preferred specifications and/or manufacturer	Figure/Panel
16	Acetone or Dimethyl Carbonate (DMC)	dry solvent packed in Ar	---
17	Balance (readable to at least 0.1 mg)	readable to at least 0.01 mg	---
17.1	Base for balance	marble or granite, anti-vibration preferred	---
18	Drying oven	Buchi or similar	5/18, 18.1
18.1	Vacuum pump	dry vacuum pump	---
19	Temperature chamber	battery-rated, outside vented chamber, Spec or Maccor preferred	5/19 (Spec)



Figure 2: Examples of equipment needed for the assembly of coin cells. The numbers in the different panels of the figure are indexed in Table A.

3 The Four Step Process in Action

- STEP 1: Planning
- STEP 2: Assembling
- STEP 3: Cleaning
- STEP 4: Connecting

3.1 Planning

In the previous sections we provided a general description of the various preliminary aspects to be considered before attempting to assemble coin cells. To be more efficient, this procedure needs to be considered a multi-step, time-bound process in which independent stages have an optimal order that should be planned well in advance. The instructions and recommendations below follow that optimal order.

But before we move on to that, we ask you to consider the following, real-life scenario:

A young and smart researcher has spent several days shopping online for the parts needed to assemble coin cells and feels confident they have found everything necessary. They are really happy to have saved £2000 by sourcing equipment and parts from different suppliers.

“After all,” they think, “the specifications are almost the same.”

To prepare for making the cells, they read several papers in which the authors used coin cells to make their electrochemical measurements; they all had similar diagrams in the supporting information and the materials and methods sections seemed quite simple. Just to make sure, they spend a whole afternoon watching videos online; some of these have really cool music.

Assembly day arrives, so hands into gloves and then into the glove box, and full speed ahead. Then they remember that there is an SOP for the coin cells somewhere, but they forgot to bring it to the lab. They shrug and continue work. Who wants to read boring stuff when you can watch videos?

They lay their components in a tidy row and begin to put the cells together. But wait, now they cannot remember what electrode goes first. And why are the wave washers so pointy? They decide it doesn't matter; they are in a hurry because they reserved the shared glove box for only an hour. Surely an hour is plenty of time to make ten cells?

They hesitate for a couple of minutes and finally decide that the order of the electrodes is not that important, they can always turn the cells around.

After standing on their tiptoes to reach the vial containing the separators, they succeed in putting all the components together for the first cell; they pick it up with metal tweezers and transfer it to the crimper. The crimper is a manual one, so they have to pump the lever in order to crimp the cell.

Up and up the base of the crimper goes, until the pressure gauge sits just below 500 psi. But they need 500 psi, and they decide that it is better to have too much than too little pressure; they pump hard and the gauge needle goes above 700 psi. Should they write that down? Maybe next time.

When they unlock the crimper and the base comes down, no coin cell comes down with it. None of the videos they watched or the papers they read mentioned cells disappearing.

That's weird, they think, but they have no time to panic.

Carefully probing with one of their hands they find that the cell is stuck to the top well of the crimper. They also find that it won't budge. They call one of their colleagues for help. She tells them to use a plastic pen to push the cell out through the aperture in the top of the crimper, carefully and with one of their hands ready to catch it. They position the pen and push with all their strength; the cell is still not moving. Half an hour has passed, and they start to panic. In a frantic attempt, they use both their hands to push down on the pen. With a sudden pop, the cell flies out of the crimper to the back of the glove box, where it lands among the various tools, jars, and plastic containers, never to be seen again.

That cell is, presumably, still in the glove box, but nobody knows for sure.

The moral of the story? Every little detail counts, so plan ahead, be prepared, and take your time.

3.1.1 To do at least several weeks before assembly

It is recommended that the following sub-steps are performed well in advance of the actual assembly of the cells in order to mitigate any problems that may arise.

- **TRAINING:** Make sure that all users are properly trained before carrying out any procedure. An example of proper training should have at least three stages: (a) Observing the process and reading high-quality teaching aids vetted by the most experienced researcher in the laboratory, (b) Carrying out the process with the help of a more experienced user, (c) Carrying out the process under audit and comparing the results with those of a more experienced user, and/or curated data set obtained under the same conditions.
- **STANDARD OPERATING PROCEDURES (SOPs):** Every lab should have written documentation of the steps of their processes. The term SOP is used differently across laboratories, but in this guide, we refer to a pared-down set of instructions to remind users of the most common steps and the order in which they should be performed. Reading a SOP is not a substitute for proper training, however. Please see the glossary for more information.
- **EQUIPMENT SPECIFICATIONS:** Make sure that all coin cell hardware matches the specifications claimed by the supplier and that there is a perfect match between the dimensions of the can and cap and the dimensions of the well of the crimper.
 - The scenario described above in which there was a serious mismatch between the coin cell diameter and the crimper well diameter has been observed by us in three different laboratories, including ours.
 - When procuring equipment with sensitive dimensions, be sure to ask the manufacturer for the tolerances associated with their specifications. Half a millimetre in diameter might not seem like much but it can significantly affect the quality of your cells. In our case, we ended up sending our

crimper to be retooled; this improved the quality of our cells, but we still had to buy an even higher-quality crimper to meet our exacting standards of work.

- Don't mix and match; always obtain your spare parts from the same supplier and document it if you need to change suppliers for any reason. Also, always record part numbers; a verbal description is not enough. For coin cells, even CR 2032, parts can have different diameters and thicknesses. There are different types of wave washers, the casing can be made from different types of stainless steel, the interior can be Al-coated to reduce internal corrosion, spacers can have different thickness, and the position of the sealing gasket can vary by manufacturer.
- In your cyclor and/or potentiostat, verify that you have the right coin cell holder for your cells. Gold-plated coin cell holders are more resistant to corrosion and are preferred whenever possible. Also make sure that you choose holders that are more resistant to metal fatigue.
- Please see section 3.2 for more details.
- CALIBRATION and MAINTENANCE: Make sure that all equipment is maintained and calibrated properly.
 - Keep calibration and maintenance schedules and certificates in a place accessible to all users.
- CHEMICALS: Check the quality and purity of all chemicals to be used and buy fresh ones if necessary.
- ELECTROLYTE: Please read and carefully follow all supplier recommendations for storing and using your electrolyte. We recommend using battery-grade electrolyte from reputable suppliers, for example Ube Corporation, Solvionics, or Solvay. How do you know if your supplier can provide you with battery-grade electrolyte? Simple, they will provide a good list of product specifications and very detailed instructions for using and storing it.
 - If your electrolyte needs to be in cold storage, let it get to room temperature inside the glove box before using it or transferring it to secondary containers.

Other things to consider:

- Do not use electrolyte directly from your primary container. Inside the glove box, transfer small portions of electrolyte (around 5-10 ml is enough for more than a month of intense use) to secondary aluminium bottles. Label your bottles with the formulation and chemical composition, your name, and the date.
- The most commonly used lithium-ion electrolytes are colourless; discard your portion if you observe any yellowish or pink colour. You could try to

filter it, but it is usually not worth the trouble as you might not get reliable results.

- As much as possible, keep your electrolyte bottles closed. Do not leave bottles uncapped between cells. Commonly used electrolytes are mixtures of solvents with different vapour pressures, which results in different rates of evaporation. If you leave your bottle open, the ratio of solvents today will probably not be the same tomorrow. That is also why using small portions in secondary bottles is important. Additionally, we recommend changing to fresh electrolyte portions every 2-4 weeks depending on usage.
- METALLIC LITHIUM: We recommend to always use the highest purity of lithium metal available. Some suppliers sell battery-grade lithium; make sure that they explicitly describe the purity and the amount and chemical identity of trace contaminants, as well as the surface finish. Foil is preferred but pre-cut lithium chips are also acceptable.
 - Only use lithium that is delivered sealed in pouches or in glass ampules, make sure these are packed under argon, and open and store them only in the glove box.
 - Do not use ingots and do not use lithium packed in oil.
 - High-purity lithium metal that has been managed and stored properly should be a shiny grey, with a smooth finish. Do not use lithium that shows a black, white, or reddish surface.
 - Please see section 3.2 “assembly” for more information.

3.1.2 To do at least 24 hours before assembly

How to formulate and laminate slurries to obtain uniform electrodes, free of defects and with adequate adhesion to the current collector, is out of the scope of this guide. We assume that readers are proficient in electrode fabrication and have prepared them before assembly, or use high-quality and well-characterised commercial products.

SETTING UP DATA AND METADATA SPREADSHEET: Before assembly begins, the first step is to set-up a spreadsheet for collecting all the data and observational metadata generated in every step of the process.

- Your spreadsheet should include, as a minimum, a unique control number for your electrodes and/or your cells, the date of assembly, the name of the user/assembler, the mass of the current collector, mass of the positive electrode, formulation of the electrode, calculation of the active mass, identification of test performed, open circuit voltage (OCV) of the cell as assembled, diameter of the positive electrode, diameter of the negative electrode, diameter and composition of the separator, chemical composition of the electrolyte, and any observational notes.

- We strongly recommend using the same control number for the positive electrode and the cell to reduce the chance of mistakes in indexing cell to electrode properties.
- In appendix 5.2 we provide a downloadable Excel spreadsheet as an example of what our battery team uses at NPL at the time of writing this guide. Feel free to adopt it in your own laboratory if you find it useful.

Note: Always wear gloves to protect yourself from chemicals and to protect your electrodes from grease from your hands.

PUNCHING POSITIVE ELECTRODES to the desired diameter. For this coin cell size, a good rule of thumb is to use a diameter of 14 mm.

- Two types of tools that are relatively cheap and easy to procure could be used: manual hole punch and mallet (figure 3, items 10, 10.1 and 11) or a hand-operated die cutter (figure 3, item 10.2). A good quality die cutter will produce electrodes with smoother borders, with fewer indentations and burrs, but a hole punch used properly can also provide good results.

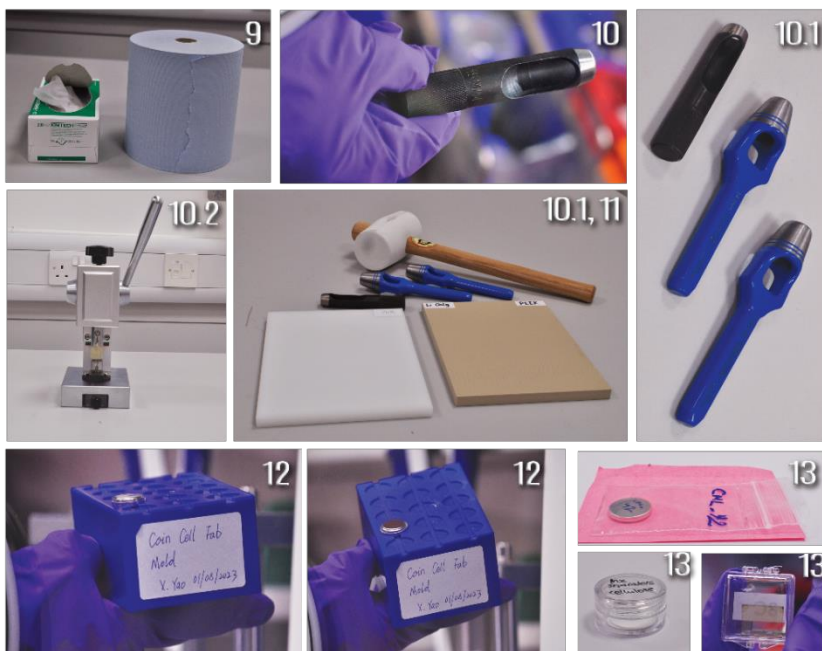


Figure 3: Examples of equipment needed for the assembly of coin cells. The numbers in the different panels of the figure are indexed to Table A.

- Prior to punching, place your electrode laminate in between two pieces of paper. Unwaxed printer paper works well. Waxed weighing paper can be used as well but there is always a chance that wax residues can transfer to the edge of the electrode, which can introduce variability that is difficult to account for. Always use paper that doesn't produce particles or dust while cutting, and make sure to gently remove any particles or dust from your electrodes after cutting and prior to drying.
- If using a hole punch and mallet, always make sure to hold the mallet close to its head, and the hole punch close to the base (figure 4), wear safety glasses, and position yourself away from the mallet (figure 4a). Also, avoid excessive distance between the mallet and the top of the hole punch; the movement should come from your elbow, NOT from your upper arm (figure 4d, 4e, 4f).
- Whether using punch and mallet or die cutters, check for oil residues or dirt in the cutters and make sure to clean them before use. Especially with new equipment, residues of oil and chemicals from the manufacturing process can contaminate electrodes during this step.

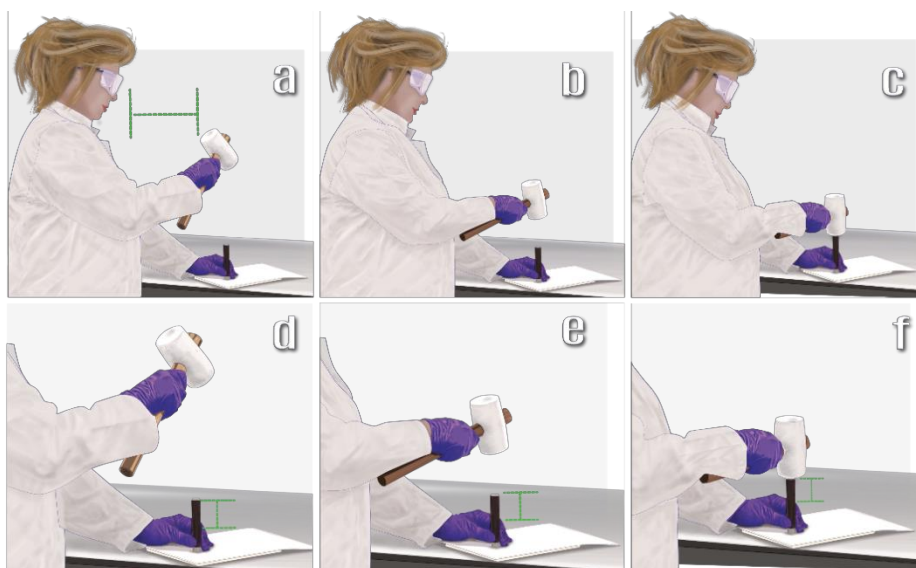


Figure 4: The process of punching positive electrodes to use in the coin cell assembly. Top row indicates the correct body position, bottom row shows a magnified view of the punching action. Notice the green dotted lines indicating the minimum distance between face and mallet (top row) and the hand position relative to the top of the punch (bottom row).

VACUUM DRY POSITIVE ELECTRODES for at least twelve (12) hours; this is usually best done overnight.

Note: When handling vacuum oven parts, always make sure that you wear appropriate oven mitts after drying and in between different users; do not assume that the oven is cool just because it is off.

- If your vacuum oven is programmable, you can set the required time. If it is not, make sure you allow enough time for the oven to cool before you start your assembly.
- We recommend using a tabletop oven with a tube that can be closed, removed, and transferred into the glove box, Buchi or similar (figure 5, items 18, and 18.1, figure 6).



Figure 5: Examples of equipment needed for the assembly of coin cells. The numbers in the different panels of the figure are indexed to Table A. (Panel 14, voltmeter; panel 15, plastic tweezers; panel 18, 18.1 vacuum ovens and pump, respectively; panel 19, battery-rated temperature chamber).

- Place your electrodes in a secondary container such as a glass vial, cover the mouth of the vial with aluminium foil, and use a tweezers or similar to puncture small holes into the aluminium foil.
- Make sure the tube valve is placed in the open position before turning on the heat (figure 6 c).
- Use a temperature of 120 °C for electrodes prepared with pVdF (polyvinylidene difluoride) binder.
- When done drying, make sure the tube valve is placed in the closed position before turning off the vacuum, (figure 6 b) and never expose your electrodes to air once they have been dried.
- We strongly recommend the use of dry pumps in all your vacuum equipment. Oil from vacuum pumps can condense inside vacuum tube ovens and create a contamination issue (figure 6e, 6f).

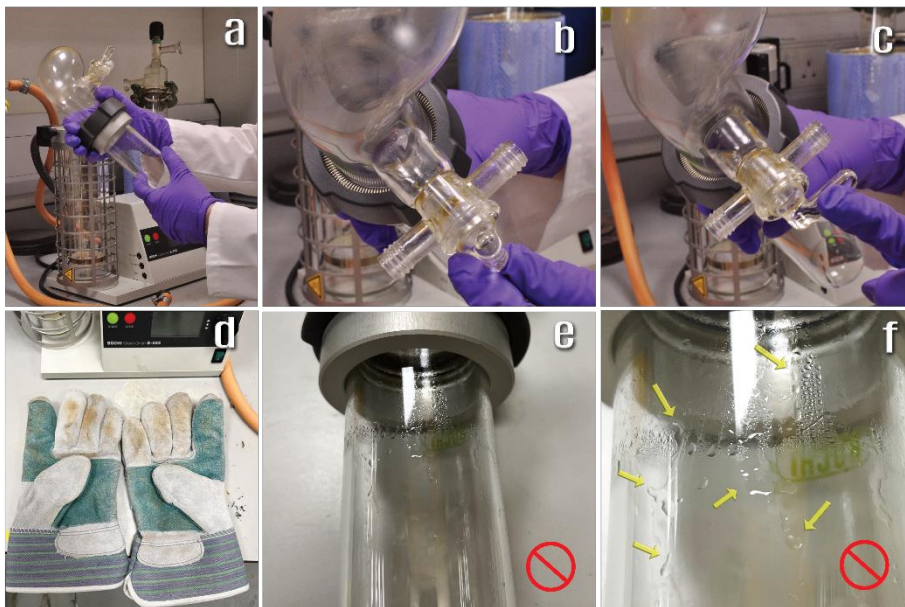


Figure 6: Some tips for handling the vacuum oven. (a) How to hold the glass tube of the Buchi oven, (b) valve closed (c) valve open, (d) a pair of well-used oven mitts, (e-f) what happens when one doesn't follow the correct procedure. Note that oven mitts were not worn when the photos were taken because the oven was not in use. See text for more details.

- Some glove boxes have a vacuum antechamber with heating mantle that can be used for drying equipment and electrodes. If using this, make sure that the temperature of the chamber is stable and reproducible.

TO WASH OR NOT TO WASH the coin cell hardware before assembling. Some workers in the literature report washing, some don't.

- First, see what the manufacturer of the coin cell hardware you use recommends. Some cell parts have obvious residues or stains from the manufacturing process, others don't, and if you have been handling them with gloves, then they should not have grease or other residues.
- If the hardware arrives clean and is handled properly, we recommend not to wash in order to minimise the use of extra chemicals. Otherwise, rinse with one of the solvents from the electrolyte (for example, dimethyl carbonate or ethyl methyl)

carbonate) and make sure the parts are thoroughly dry before bringing them into the glove box.

- We do not recommend baking or heat treating the coin cell parts. However, whatever procedure you use, we strongly recommend recording it in the notes section of your data and metadata spreadsheet for future reference.

TO HEAT-DRY OR NOT the separator. This depends on which type of separator you use.

- Always follow the instructions of the manufacturer/supplier and avoid purchasing material that doesn't have detailed specifications.
- For lithium-based chemistries we recommend using a three-layer polypropylene-polyethylene-polypropylene separator (PP:PE:PP, Celgard 2325 or equivalent).
- These separators should not be dried at temperatures higher than 50 °C in order to avoid significant changes in porosity. ⁽⁸⁾
- If stored under inert atmosphere, we recommend NOT to heat dry at all.
- We do NOT recommend the use of filter papers (Whatman or similar) as separators due to their susceptibility to mechanical deformation when wetted with electrolyte, which increases the risk of shorting during cell assembly.
- Another problem arises if performing post-mortem/post-test analysis, as residues from this type of separator often transfer to the active electrode materials.
 - However, if you must use this, record your choice, and dry overnight to at least 100 °C, using the same vacuum oven and transfer procedure into the glove box as the positive electrodes.
 - Furthermore, be careful not to confuse separator fibres with dendrites when using SEM or other imaging techniques to characterise the electrode materials.

OTHER THINGS to consider:

- Most research laboratories have only one fabrication glove box for coin cells. Reserve enough time in the glove box. Whatever time you think you need, double it to allow for mistakes, fatigue, missing equipment, and enough time to cycle from vacuum to Ar atmosphere in transfer chambers.

3.2 Assembling

3.2.1 Before assembly starts

3.2.1.1 Initial checks

If the planning stage was followed carefully, you should have all the necessary parts and equipment inside the glove box; however, there are often things that are forgotten or used up. We recommend allowing between twenty (20) minutes to one (1) hour to bring things inside the glove box if necessary.

- Check that you have enough argon pressure to operate the glove box.
- Always wear cotton or disposable gloves.
- Additionally, you should wear a pair of disposable gloves on top of the gloves of the glove box to protect them from chemicals.
- We strongly recommend working with a colleague to record all data and observational metadata directly on the spreadsheet.
- We strongly discourage writing on the walls of the glove box, on gloves, or assorted pieces of paper.
 - The only writing done inside the glove box at this stage should be the unique identifier number in the bag/box for the positive electrodes and cells (see section 3.1.1, table A, figure 3, item 13), and on the can of the cell, as described below.
- Use only plastic tweezers to manipulate all the parts, electrodes, and the finished cells. Never touch the finished cells with any metallic parts including the bottom of the glove box, tweezers, aluminium bottles, etc.

3.2.1.2 Recording the mass of the positive electrode

A battery electrode is a layered sheet that includes a current collector (aluminium for the positive electrode) onto which a laminate composite is applied. This laminate composite includes the active material, a conductive additive (usually some sort of amorphous carbon), and a polymeric binder that holds everything together.

The formulation of this electrode is usually indicated in weight percent and written with the name of the active material followed by percentage of active: percentage of conductive additive: percentage of binder, for example NMC 811 (90:5:5). This is important because the initial evaluation of materials is usually reported as gravimetric capacity in units of mAhg^{-1} . By knowing the formulation of the electrode and the mass of the current collector the mass of active material in a certain geometric area can be calculated.

Manipulating weighing boats or weighing paper inside the glove box can be challenging. Whenever possible, we recommend weighing each individual electrode directly on the surface of the balance, assuming that this surface is clean and free of particles.

Weighing the electrodes in a glove box can be challenging and requires patience. Below is a step-by-step procedure.

- Make sure the balance is stable and within comfortable reach of your arms.
- If your balance has a level indicator, use it to verify that the balance is well levelled. If it is not, do not weigh your electrodes. Follow manufacturer recommendations to level the balance before proceeding further.
- Tare the balance and wait for the number on its display to stabilise.
- Using a pair of plastic tweezers (like the ones shown in figure 4, item 15), gently pick up one positive electrode and place it in the middle of the weighing plate of the balance.
- Try to stay very still.
- When the number in the balance display stabilises, have your colleague record this number in the spreadsheet.
- Always double check that this mass is recorded correctly.
- Place the electrode in the small plastic bag and close it.
- Write the unique identifier number on the bag. This number will also be used to identify the corresponding cell and should be used to index all properties and other observations and data in the spreadsheet.
- Repeat the previous steps for all the positive electrodes that you plan to use for this batch of cells.

Note: Most battery electrode materials are usually air sensitive and will either react irreversibly with atmospheric oxygen and moisture, or absorb it easily, which may introduce errors in mass or in performance. Always dry your electrodes as described above before using, and store them in the glove box.

- Never expose your electrodes to air after drying.
- Be aware that storing under inert atmosphere is NOT a substitution for drying in a vacuum oven.

3.2.1.3 Recording the mass of the current collector

Note: This step can be done in advance or the same day of assembly, whenever is most convenient for the user.

To be able to calculate the mass of active material you need to know the mass of the current collector.

To do this, punch at least 10 pieces of aluminium from the same foil used to prepare the positive electrodes (if prepared in house), or from the clean borders of the electrodes (if procured elsewhere, figure 7a).

- Always use the same punching method, for example use the same hole punch you used cutting the positive electrode, or the same die cutter.
- Always use the same balance used to weigh the positive electrode.

Record the mass of the ten pieces as you did in the previous step and calculate the average. We recommend rounding this average to the same number of decimal places that you have in your balance.

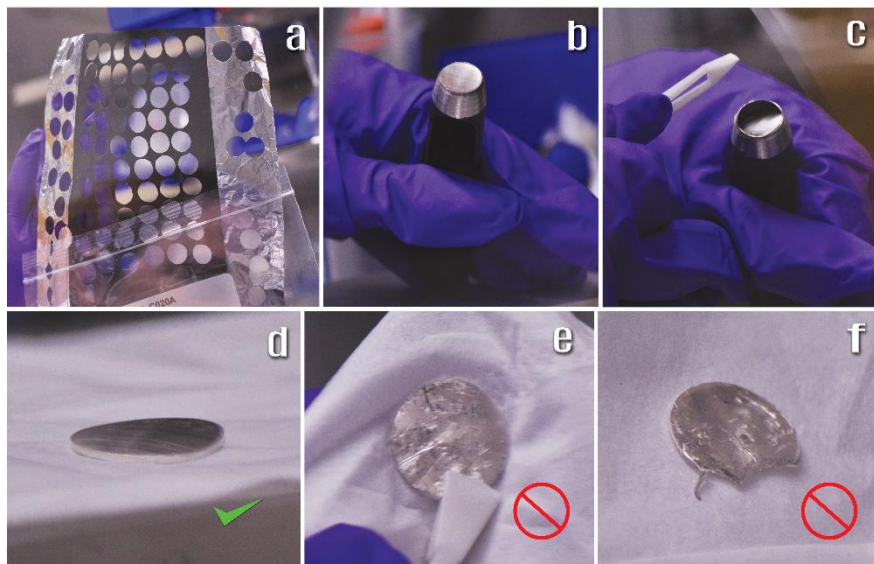


Figure 7: About the current collector and the negative electrode. (a) How to punch current collectors to calculate their mass, (b-c) punching metallic lithium, (d) good metallic lithium electrode, (e-f) bad, and very bad metallic lithium electrode.

3.2.1.4 Prepare metallic lithium electrodes

See section 3.1.1 for additional information on purchasing and storing metallic lithium.

Metallic lithium is soft and can be easily smeared onto other surfaces; waxes and grease stick to its surface and are difficult to remove, and lithium reacts readily with fluorinated compounds such as PTFE.

Use only shiny and clean lithium. We strongly advise against cleaning or preparing lithium in-house as a homogeneous surface quality cannot be obtained; this affects the growth of the primary SEI layer in lithium and can introduce irreproducibility in the electrochemical response of the cell. (1-3, 7)

- Always use the same thickness of lithium. Cycling consumes active lithium; therefore, having the same thickness is important, especially for cycling life evaluation longer than 25 cycles.
- For this type of coin cell, we recommend using a 15 mm diameter hole punch, and a lithium thickness of 200 μm . Higher thicknesses could be used but lithium is expensive and, in most circumstances, thicker lithium electrodes are an unnecessary waste of material. At any rate, make sure that lithium electrode thickness and diameter are the same from cell to cell.
- When punching metallic lithium make sure that you use the hole punch exclusively for this, and do not use the punch for other materials to avoid cross contamination. Place your lithium on a clean plastic surface; PEEK or POM are preferred. If none of these are available, you can use a clean metal surface such as that from a lab jack.
 - NEVER use PTFE.
- Press firmly on the top of the hole punch. Remove your punched electrode with plastic tweezers (figure 7b-d) and set aside. Try not to press, pinch, or otherwise deform your lithium electrodes. Discard any that show obvious signs of mechanical damage (figure 7e-f).

3.2.2 Putting it all together

At this point the average user would have spent close to an hour working in the glove box, and their arms are probably sweaty.

Before proceeding further, we recommend taking a break; drink water, check your emails, move around, whatever, for approximately 10-15 minutes. There is a very good reason for this.

At the time of writing this guide, we have spent more than six months gathering observational metadata and there is strong evidence that the number of bad cells produced during assembly increases with tiredness of the user, even for highly experienced researchers. Very often, the

user is not aware of their level of fatigue until they make several mistakes in a row, or worse, after they have set to cycle cells that should have been scrapped in the first place.

We strongly recommend taking frequent breaks when assembling cells in a glove box.

- Do not rush your work, and do not attempt to make large numbers of cells per batch.
- A good rule of thumb is to take a 10–15-minute break at the following intervals:
 - in between preparation and the actual cell assembly
 - after making four (4) to five (5) cells

3.2.2.1 Now we can finally assemble the cells

Schematic illustrations of the components of the coin cell and the order in which they should be assembled are shown in figure 8. We strongly recommend printing these figures and keeping them by the glove box. The illustrations are rather self-explanatory; however, a few extra tips follow.

- There are several hardware designs depending on the manufacturer of the coin cells, these vary in subtle but important ways. The two most common for CR 2032 are shown in the figure 8; (a) the gasket as a separate piece, and (b) the gasket integrated in the cap. This slightly changes the order of assembly as illustrated in the figure.
- The two gaskets are shown in figure 8, c-d, and figure f, respectively.
 - Notice that in figure 8, c, and d, the top and bottom of the separate gasket have slightly different diameters. This gasket should always be positioned with the smaller diameter aperture facing down and in direct contact with the can.
- The volume of electrolyte should be dispensed using a calibrated auto pipette similar to that shown in figure 2, items 7 and 7.1.
 - We recommend using 100 microlitres of electrolyte dispensed directly on top of the separator once you have put it into the cell.
 - You can refine this quantity to adjust for your own electrode materials and if you use other lithium metal thicknesses. Always remember to record any modifications in the data and metadata spreadsheet.
 - The effect of the volume of electrolyte used when assembling coin cells has been investigated by other researchers, and is out of the scope of this guide. ⁽⁹⁻¹⁰⁾
- ALIGNING THE POSITIVE AND NEGATIVE ELECTRODES as perfectly as possible is important to mitigate edge effects that can promote the premature growth of dendrites.

- If you use a trilayer separator like the ones recommended in this guide (Celgard 2325 or similar), you will be able to see the positive electrode through the separator which can help with electrode alignment.
- SHOULD YOU USE ONE OR TWO SPACERS? This depends on the thicknesses of your electrodes and separator. If you have the dimensions and products recommended in this guide, you should use two spacers as shown in figure 8, each with a diameter of 16 mm and a thickness of 0.3 mm.
 - You also have the choice of purchasing a wave washer soldered to a spacer, as shown in Table A, and figure 2, item 3.3, and using it as the top spacer. At the time of writing, we have some preliminary data indicating that using a soldered wave washer makes it easier to transfer the cell to the crimper without having the components move around.
 - If you choose this option, make sure that you wash and dry this component before using it (as described in the section on coin cell hardware above) as it may have residues of the soldering process.

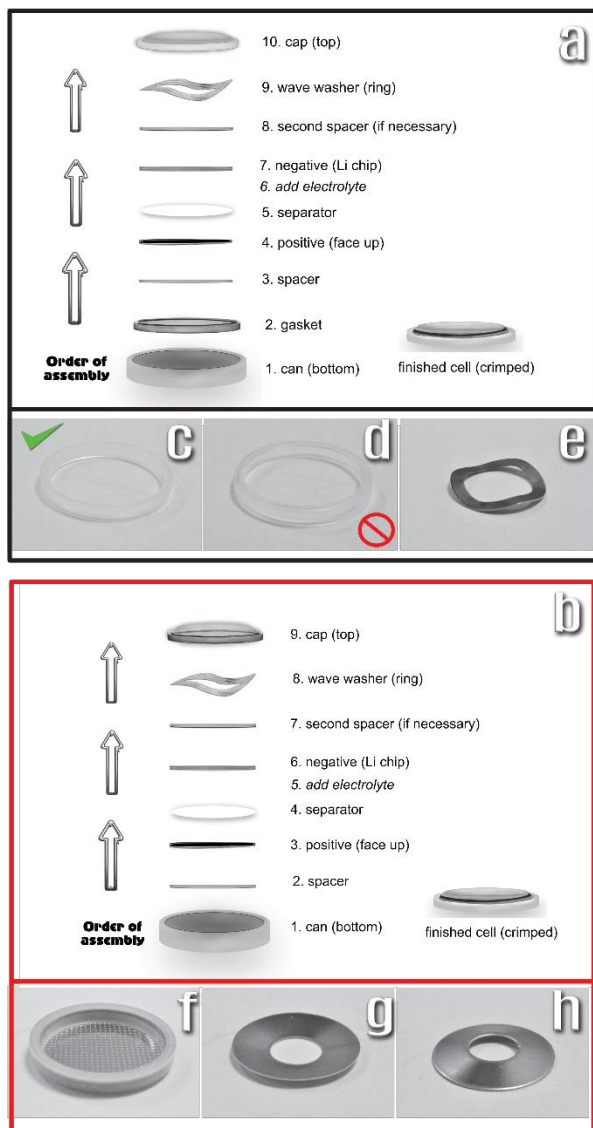


Figure 8: Diagram on how to assemble coin cells with different hardware. (a) Hohsen CR2032 with separate gasket, (b) Unknown supplier with integrated gasket and cone wave washer. The circle with a diagonal indicates the least favourable option, the green checkmark the preferred. (c) The right orientation to place the gasket inside the can in assembly a.

When you have stacked your cell components as in figure 8, it is time to crimp the cell.

Figure 9 shows the steps for this part of the process using a manual coin cell crimper that had been retooled to fit the coin cell better.

- With the cell still in its mould, use your thumb to press firmly on the cap (figure 9a, b).
- If your internal components are well aligned, you should feel the cap go down slightly.
- Use a pair of non-metallic tweezers to lift the cell from the mould and transfer it to the coin cell crimper (figure 9c, d).
- The cell should sit comfortably in the well of the crimper, with no obvious imbalance, and completely parallel to the plane of the crimper well.
 - Gently adjust as necessary to achieve this.
- Once you are satisfied that the cell is positioned properly:
 - Turn the spoked knob towards the lock position (figure 9e, f).
 - Pump the lever up and down until you feel increased resistance.
 - Stop and closely observe the circular pressure gauge, then resume pumping the lever until you reach 500 psi.
 - If it looks that you are not quite at the 500 psi mark, (e.g., you think you are closer to 450 than to 500 psi) stop there and document your estimated pressure in the data and metadata spreadsheet.
 - Do NOT keep pumping; it is preferable to undershoot than to overshoot the pressure. Overshooting the pressure often leads to complete or partial shorting of the cells.
 - Turn the spoked knob towards the open position. The base of your crimper should automatically come down with your cell.
- If your cell gets stuck on the top well, use a plastic pen or rod of the appropriate diameter to dislodge the cell as shown (figure 9i, j).
- Always make sure you are ready to catch your cell.
- As soon as you finish crimping your cell, dip a low or lint free (Kimwipe or similar) tissue in acetone or a solvent such as EMC or DMC and WIPE ALL ELECTROLYTE RESIDUES from the cell.
 - Dry solvents packed under argon are preferred, but common chemistry lab solvents will work well enough.
 - This is a tricky thing to do in the glove box and trickier still to describe in writing. Please see the video in the supplementary information for this document (found here <https://doi.org/10.47120/npl.mgpg153>) for a visual guide. DO NOT immerse the cell in liquid.
 - Wiping the cell clean is a VERY IMPORTANT step and it MUST be performed inside the glove box.

- Residues of the electrolyte salt can react with atmospheric moisture to generate HF. This, in turn, can corrode the cell casing introducing variability in the electrochemical response (figure 10).
- With time and use, residues from poorly cleaned cells build up in the coin cell holder and contribute to degradation of the metallic parts of this part of the equipment.

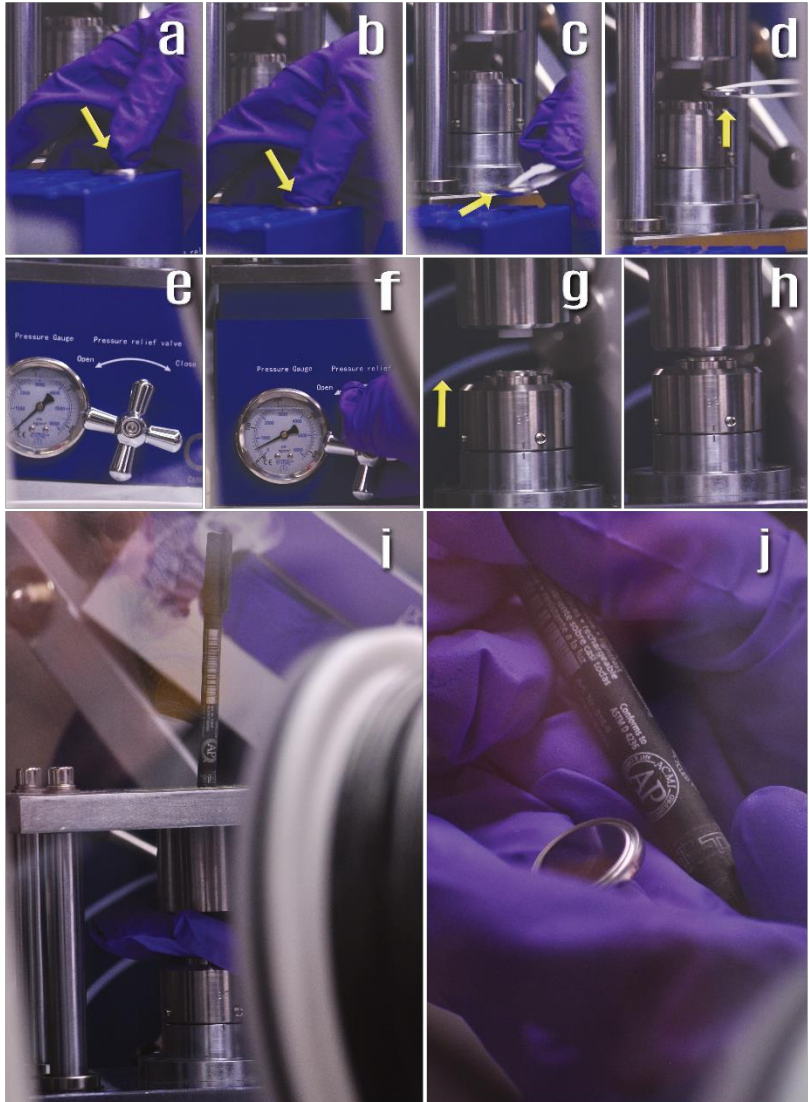


Figure 9: Crimping the cell. (a-b) Pressing down with thumb, (c-d) transfer cell to the crimper well, (e-f) locking the crimper, (g-h) well moves up as the user pumps, (i-j) technique for releasing a stuck cell. Always put a hand below the cell to be able to catch it as it pops out.



Figure 10: The importance of cleaning the cells after crimping them. (a-b) A cell that has been cleaned properly, (a) top (b) bottom (b); a cell that has not been cleaned properly before taking it outside the glove box, (c) top (d) bottom.

Note: From this point on, it is crucial that your crimped cell doesn't touch any metal, except the leads of the voltmeter, until it is connected into the coin cell holder for electrochemical testing.

- Put your cell down onto a piece of clean paper and measure the OCV with a handheld voltmeter and record the value on the data and metadata spreadsheet. A nifty way of measuring OCV is shown in figure 11 in which we use a wooden clothes peg to hold the cell in position.
- OCV values from 3.18 V to 3.45 V vs. Li/Li⁺ are considered within range (for the NMC 811 half-cell, carbonate-based electrolyte, Celgard 2325, cell chemistries).
- Cells at 2.00 V and below are to be discarded.
- Cells with OCV values in between the ones mentioned still cycle but often their electrochemical performance is not reproducible for no other obvious reason.

- You cycle them at your own risk.

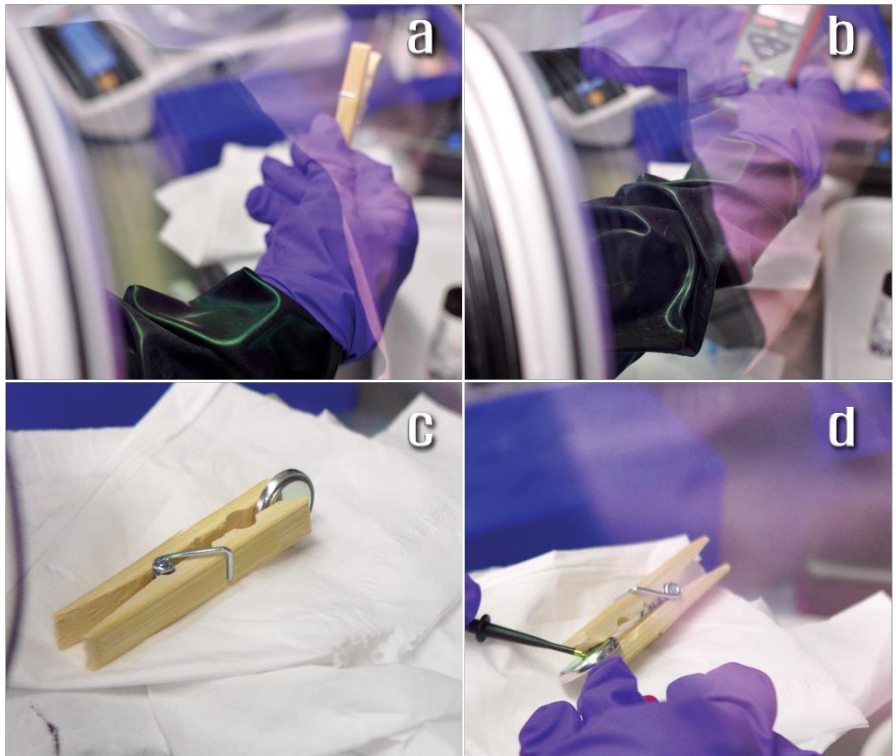


Figure 11: A good way to measure OCV inside the glove box. (a) wooden clothes peg, (b) hand-held multimeter, (c) the cell clamped in the clothes peg, (d) how to measure OCV. Notice that the two probes of the multimeter are held roughly perpendicular to the plane of the cell and directly opposing each other.

Using a marker (Sharpie or similar), write the UNIQUE IDENTIFIER NUMBER on the can of the cell. This is the number that you assigned to the individual positive electrodes when you weighed them and put them in individual plastic bags/boxes, and that is recorded with all the other information in the data and metadata spreadsheet.

- We strongly recommend that you use this number to label every data file associated with each individual cell or samples extracted from it, for the purpose of traceability and to avoid sample identification errors.
 - Do not use wax pencils or graphite to write on the cells as these can introduce variation in the contact resistance of the cell casing.

Put your cell inside the plastic bag/box in which you originally kept the positive electrode. Close it firmly and put aside.

Repeat the steps above until you arrive at the desired number of cells, or it is time to take a break, whichever happens first.

If you need a large batch of cells, it is better to divide your work in two or more days to ensure the quality and sustainability of the process. It is normal to make mistakes and to produce a certain number of bad cells, but this number can be reduced if you take your time and lessen errors arising from fatigue or poor planning.

3.3 Cleaning

“Cleanliness is next to godliness” according to the common adage. And the next step is making sure that all the equipment and materials that were used are cleaned, disposed of, and/or stored properly.

The only waste materials to be treated differently are METALLIC LITHIUM residues, which should be collected in an appropriate separate container and kept inside the glove box until ready for disposal.

- Please consult your lab manager or lab owner as to the proper way to dispose of metallic lithium.
- NEVER dispose of lithium metal residues if you are not fully trained to do so.
 - Asking your colleagues how to dispose of it or watching videos online does NOT count as appropriate training.
- NEVER accumulate large quantities of lithium metal in the glove box.
 - A good rule of thumb is to have a wide mouth jar of 500 mL in volume. If this jar is $\frac{1}{2}$ full without compacting the residues, it is time to arrange for disposal.
 - Never compact residues in a jar to create more usable volume; if the jar is $\frac{3}{4}$ full and hasn't been collected yet for disposal, start a new one.

When cleaning tools and equipment, pay special attention to all parts that have been in contact with electrolyte, including the well of the crimper. Even inside the glove box, electrolyte residues can accumulate and create sticky crusts around joints and moving parts.

- Use a low lint paper (Kimwipe or similar) soaked in acetone to wipe the well of the crimper to remove electrolyte residues.
- Remove and discard the plastic tip from the micropipette used to dispense electrolyte.
 - Rinse with acetone and let the solvent evaporate in the glove box before bringing it out.

- Collect all waste, used gloves, and used paper inside a plastic bag or inside one of the disposable gloves to bring outside and dispose of properly.
- Consult the health and safety guidelines of your institution for specific instructions.

Purge your glove box as per the manufacturer's instructions to remove any solvent residues.

Note: Glove boxes in which battery work is conducted should never vent directly into the laboratory; appropriate exhaust has to be provided prior to the work.

3.4 Connecting

How to perform electrochemical cycling of the coin cells is out of the scope of this guide; however, there is one additional step that goes before and which is often overlooked because of its apparent simplicity: connecting the cells to the cycler.

Before cycling, it is important to make sure that the coin cell holders and interconnects are in good condition. Even small irregularities in connection can introduce significant errors or short the cells. Figure 12 shows some common problems that can be encountered when connecting cells.

- Metal fatigue can affect the points of contact with the coin cell as well as contact pressure (figure 12 a).
- Exposed metal or bad fit between male and female interconnects can short a cell (figure 12 b, c).

Unfortunately, at the time of writing, there is no good method to control for these problems other than allowing enough time to check all interconnects before running your cells every single time. Another alternative is using higher quality equipment like gold-plated coin cell holders with springs to qualitatively control the pressure, and to have electronics that interface directly with the main frame of the cycler which helps by reducing or removing the number of connections between cell and cycler.

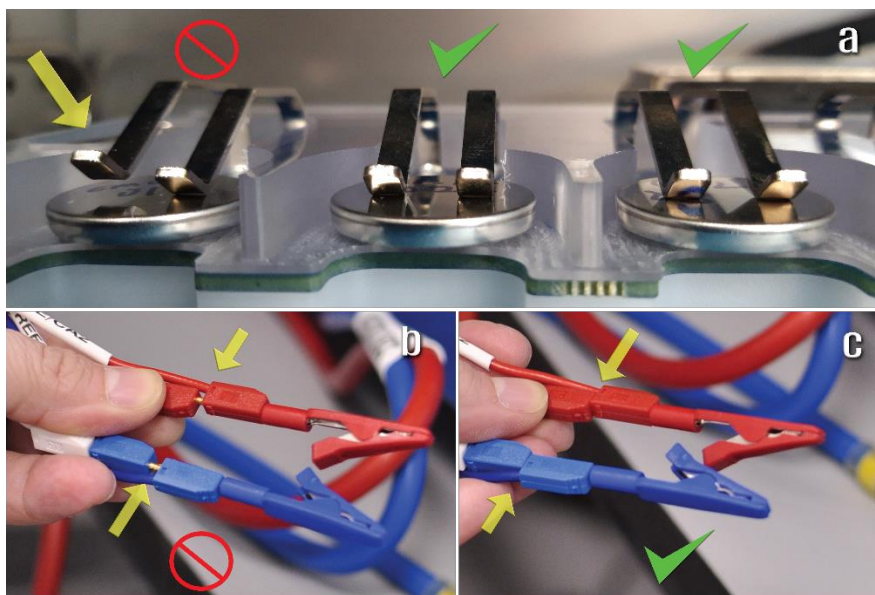


Figure 12: Common problems that can be found when connecting cells to the potentiostat. (a) Metal fatigue in coin cell holder, (b-c) banana plug connection to cables: (b) good, (c) bad. Notice that the bad connections are very difficult to detect and require careful observation.

4 Final Thoughts

- A few final recommendations
- To the users

This good practice guide has been developed based on a combination of more than fifteen years of experience assembling coin cells in more than five countries across the world, systematic audits conducted with our partner academic institutions in the UK, and additional experiments specifically designed to confirm those audits in our own laboratory at NPL.

The primary goal of our recommendations is improving the repeatability and reproducibility of electrochemical measurements in a sustainable and accessible way, the first step towards which is producing good cells reliably. No less important, we aim to serve the Faraday Institution and the wider research community by making the knowledge and associated resources freely available.

4.1 A few final recommendations:

- View the assembly of coin cells as a process and follow each step to the best of your ability.
- SOPs are not optional; they must be followed as written. SOPs can be changed but changes must be well documented and there have to be good technical reasons for the changes.
 - Validation data should be collected if/when changes are made.
- Calibration, regular inspection, and maintenance of every piece of equipment is very important.
- Metadata is as valuable as data.
 - Observational metadata is important to improve processes and helps identify sources of variability.

4.2 To the users

We hope that you enjoyed reading this good practice guide and that, from now on, it helps to facilitate your research. This document was made primarily with the practical needs of researchers in mind and, in particular, those entering the field of battery science for the first time.

We want this guide not only to showcase what we do well, but more importantly, we hope it helps to pinpoint common hurdles that all battery scientists may experience regardless of access to equipment or level of expertise.

Please notice that this good practice guide can be cited as: *Lopez et al., NPL Good Practice Guide 153, DOI: <https://doi.org/10.47120/npl.mgpg153>, 2024*

5 Appendices

- Glossary
- Example of a Data and Metadata Spreadsheet

5.1 Glossary

- **Cell Crimper:** Machine used to seal coin cells after putting all the components together. Before crimping, the cell is not functional.
- **Coin cell formats CR 2032 and CR 2016:** Common coin cell formats, the numbers indicate the cell dimensions in millimetres; CR 2032 is 20 mm in diameter and 32 mm in thickness, while CR 2016 is 20 mm in diameter and 16 mm in thickness.
- **Crimping a cell:** The action of sealing all the components of a coin cell using pressure to render it a functional electrochemical device.
- **Dendrites:** Branching structures with high surface areas that limit the cycle life of batteries and significantly contribute to electrode and cell ageing.
- **DMC:** Dimethyl carbonate. A solvent commonly used in lithium battery electrolytes.
- **Dry room:** A facility in which the level of moisture is carefully controlled to allow for the processing and assembly of water-sensitive materials and devices. Most advanced rechargeable batteries are assembled in this type of facility.
- **EMC:** Ethyl methyl carbonate. A solvent commonly used in lithium battery electrolytes.
- **Full-cell format:** A full Li-ion battery as defined by the electrodes. The positive electrode is usually a transition metal oxide, and the negative electrode is graphitic carbon or another intercalation or conversion compound. This means that the main reactions in the negative electrode are intercalation/insertion and deintercalation/deinsertion, or depending on the chemical identity of the negative electrode, reversible conversion and/or alloying. Plating of metallic forms can happen when the cell ages or under cycling conditions known as “abuse”.
- **Gravimetric capacity:** The cell capacity normalised to the active mass of the capacity-limiting electrode, which is usually the positive electrode.
- **Half-cell format:** Contains a transition metal oxide as the positive electrode and a metallic electrode (i.e. in the zero-oxidation state) as the negative electrode. This means that the main reactions in the negative electrode are stripping and plating.
- **Jelly roll:** In cylindrical cells, the stack of electrodes and separators rolled together that form the center of the cell. They can be rolled onto themselves or onto a central piece of metal called a mandrel.
- **Native surface layers:** Layers which form because of surface defects of materials and their intrinsic interactions with environmental contaminants, and which can affect interfacial properties of electrodes.
- **PEEK:** Polyetheretherketone, a plastic material.
- **POM:** Polyoxymethylene, a thermoplastic polymer.
- **Primary SEI layer in lithium:** The solid electrolyte interface first formed when lithium metal is in contact with a solution of its ions.

- **psi** “pounds per square inch” a unit of pressure used by some equipment manufacturers. Note that the SI unit of pressure is the pascal (Pa), which is defined as 1 Nm⁻². 1 psi approximately equals 6895 Pa.
- **PTFE**: Polytetrafluoroethylene, a synthetic polymer commonly used to make parts.

5.2 Example of a Data and Metadata Spreadsheet

Cell ID	Date	Cathode composition	Cathode (wet electrode) mass / mg	Current Collector mass / mg	Active material %	Anode Mass	Electrolyte Composition	Separation	Anode Composition / Ref	Voltage Initial	Notes	Test	
INTALS_01	Name Surname	DD/MM/YYYY	Material Abbreviated Name - Provider Name - diameter	20.00	14.00	90.00%	5.40	Conc. LPE, in example ##-## (anode used as)	Brand name, diameter mm	Anode Material - Thickness mm -LOT- EXAMPLE, diameter mm	OCV inside the glovebox	Note or observations that may affect the cell during fabrication	Controlled conditions - Channel & Cycling result
BRB_01	William B. Scientist	08/10/2022	NMC 811 - ADNOCHEMS lab Cathode A 023A, 14 mm	21.10	9.72	90.00%	10.24	1.0 M LPE, in EC/PC 50:50 (100 µl)	Original 2325, 10 mm	Lithium Metal - Alfa Aesar - 0.75 mm - LOT: N058044, 15 mm	3.15	Cell fill and may have touched metal prior to cycling, traces of electrolyte, user was sleepy while working, etc.	Controlled conditions - Channel 9, Cell shorted
							0.00						
							0.00						
							0.00						

A fully usable Excel file can be accessed in the supplementary information for this guide (found here <https://doi.org/10.47120/npl.mgpg153>).

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