CHADA

Characterisation Data and description of a characterisation experiment

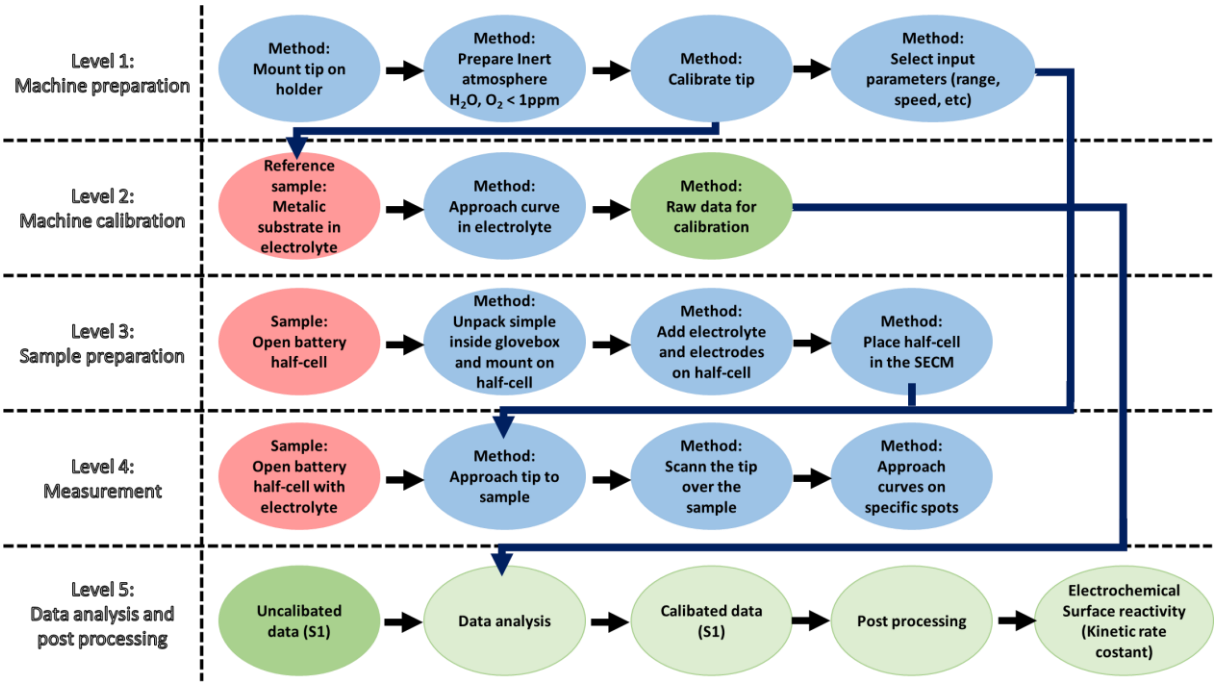
For Scanning Electrochemical Microscopy (SECM) in liquid

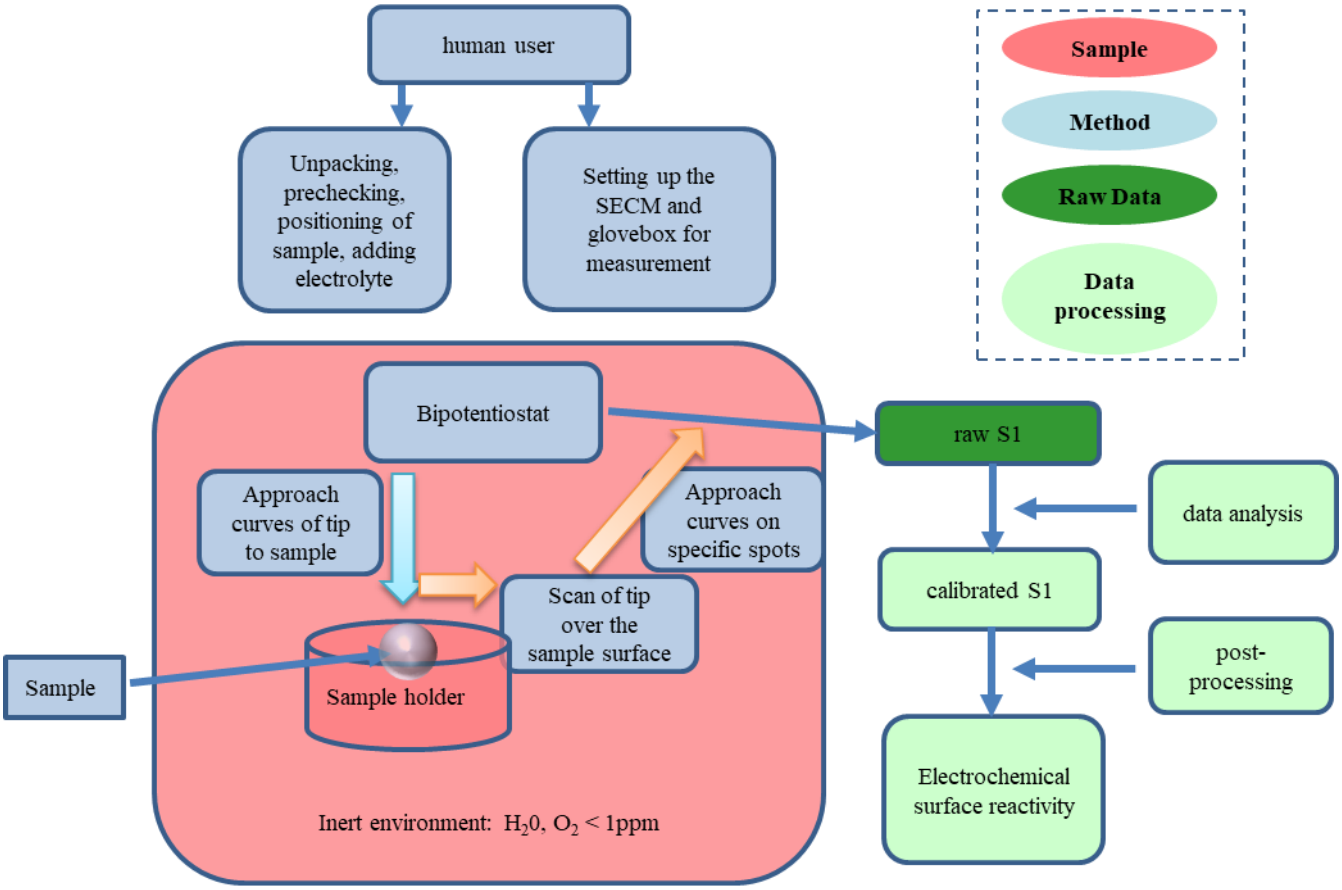
Used in NanoBat (H2020)

Overview of the Characterisation

1	Sample	Open Li-Ion battery half-cells in inert, dry atmosphere (argon)
2	Chain of methods Method	SECM prep 1: Microelectrode tip is mounted onto the tip holder
		Glovebox prep: Argon filled glovebox is prepared to guarantee dry, inert atmosphere
		SECM prep 2: The response of the tip is calibrated
		SECM prep 3: Selection of all input parameters
		Sample prep 1: Half-cell is transferred to the glovebox, unpacked and fix onto the sample holder
		Sample prep 2: Electrolyte, counter- and reference- electrodes are added in the half-cell
		Sample prep 3: The half-cell is placed inside the SECM
		Approach: Tip is brought in contact with the sample
		Measurement: 2D Scan according to the input parameters
		Measurement: Approach curves on specific spots of the sample
		Retraction: Tip is retracted from the sample
3	Data publication	Open innovation environment (established during this project) and Zenodo (https://zenodo.org) Add direct link after publication
4	Access conditions	Open access for raw data. Files are ASCII data, either in .dat or .txt format. They can be read using any kind of data reader, as Microsoft Editor, Notepad, MS Excel or comparable programs.
5	Workflow of the characterisation	Raw data is acquired by following the chain of methods described in point 2. The measured S1 parameter is then calibrated using fitting of the raw data using a reference sample. Finally, the calibrated S1 is post-processed to obtain electrochemical surface reactivity (kinetic rate constant related to electrically conducting character of the passivating SEI film).

Workflow picture





1. Sample		
1.1	USER	Human users (some to various years of expertise in scanning microscopy), no automation of the test
1.2	User case (sample specifications)	Open Lithium-Ion battery half-cells. Sample dimensions approx. 30x30x1mm^3. The battery electrode is fixed onto a sample holder and brought to an argon filled glovebox. The electrolyte is applied onto the electrode within the glovebox.
1.3	Specimen	Battery heterogeneous material (solid electrode and liquid electrolyte)
1.4	Testing environment	Inert gas environment, ambient pressure, H_2O and O_2 < 1ppm. Low noise and vibrations.
1.5	Material	Battery electrode consisting of porous film fixed on a metal foil. The porous film contains ceramic particles, carbon particles and polymeric binder

2. Methods		
2.1	Sample/probe physics of interaction	A redox species (redox mediator) dissolved in the electrolyte undergoes redox reaction at the tip, then diffuse to the sample where it interacts with the sample surface and returns to the tip. The interaction between redox mediator and sample determines the electrochemical response recorded at the tip of the SECM.
2.2	Volume of interaction	A hemisphere with a radius of approx. 1-12 µm (depending on the tip quality)
2.3	Equipment setup	Coarse X,Y,Z positioner, nano-positioner for scanning in X,Y. Nano-positioner in Z for tip-sample-distance control. A bipotentiostal control the potential of the sample as well as the tip, and record the response generated at the tip.
2.4	Calibration	S1 calibration: Approach curve from a metal pad (reference sample) inside the electrolyte.
2.5	Probe	Microelectrode tips produced by RUB
2.6	Detector	The bi-potentiostat measures the current intensity generated at the tip
2.7	Signal	Current flow of electron, signal S1, recorded at the tip when its potential is held at constant value
2.8	Time lapse	Prep: Sample preparation and machine setup 1-2 hrs Measurement: 15min – 5hrs, depending on the resolution, scan size and probed frequency bandwidth
2.9	Testing Input parameters	Probing radio frequency signal: power, frequency, signal bandwidth, number of RF pulses to average per pixel Scan: size and resolution Tip-Sample control: feedback-loop gains and setpoint
2.10	Main acquired channels	Current intensity (S1) at the tip

3. Raw Data		
3.1	Raw Data	2D maps in binary (.dat/.txt) format of the S1 (current intensity)

		Impedance spectra at any lateral grid point also in binary (.dat/.txt) files
3.2	Data acquisition rate	Up to 10 points/s for normal scans, high data acquisition rates up to kHz could be obtained using special modules for pulsing.

4. Data Processing		
4.1	Main data filtering processes	The intermediate frequency bandwidth (IFBW) defines the pulse width of the probing signal. A high IFBW probes the sample over a wider frequency range and the measured response is therefore an average (convolution) over this range. Additionally, the raw data is an average over the number of pulses specified in the input parameters.
4.2	Main data analysis procedures	Calibrated S1 is obtained via fitting the experimental signal to the simulated one.
4.3	Main processed channels	Current intensity (S1) at the tip
4.4	Data processing through calibrations	The calibrated S1 parameter is used to calculate the kinetic rate constant (electrochemical reactivity of the surface´s sample) using the results from the retraction curve described in 2.4
4.5	Properties (elaborated data)	Electrically passivating character of the solid electrolyte interface formed on the surface of the anode.