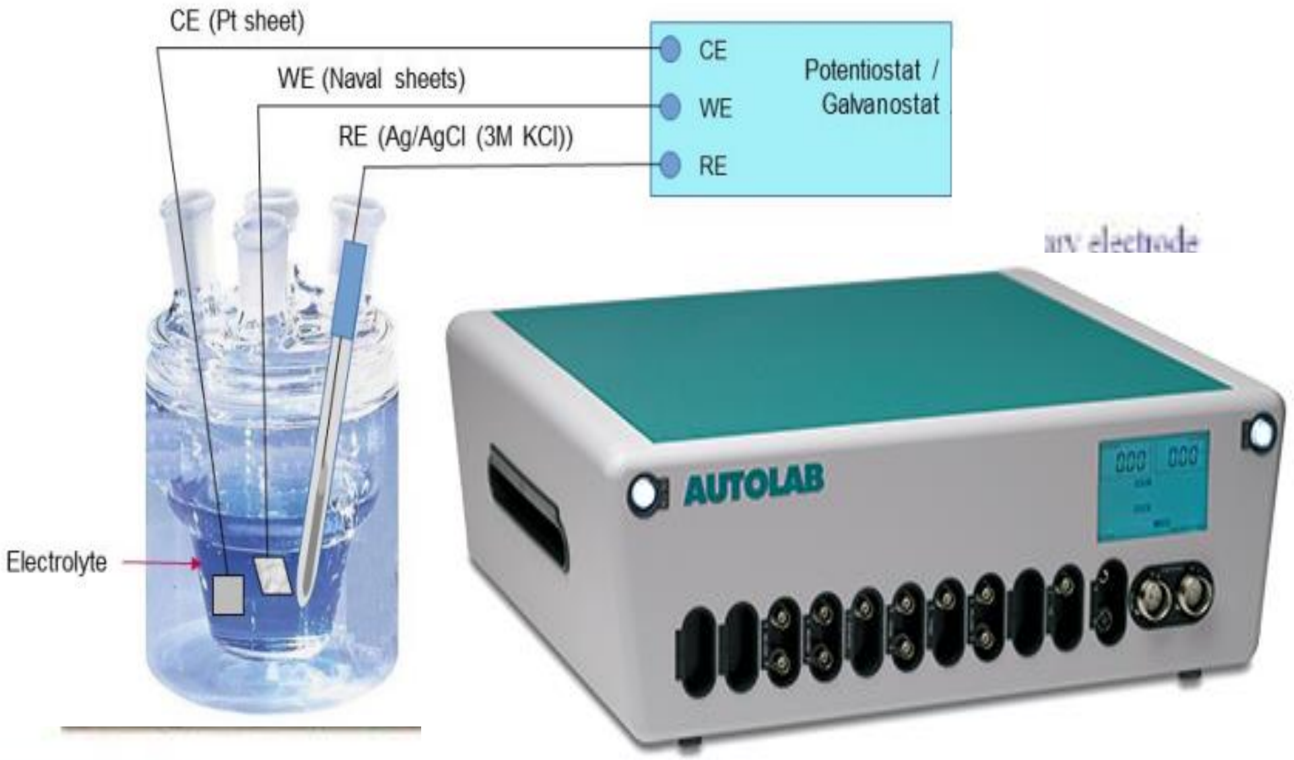
AUTOLAB

Presented by: Roghayeh Samadianfard

12/7/2022

Description



[redacted] Autolab Potentiostat/Galvanostat equipped with an Autolab PGSTAT-302N, general purpose electrochemical system (GPES) and frequency response analysis (FRA) software [A] and three-electrode electrochemical cell system, consisting of a working electrode (Enzymes/c-MWCNT/PANI/Pt), a silver/silver chloride (Ag/AgCl) as reference electrode and Pt wire as auxillary electrode [B]

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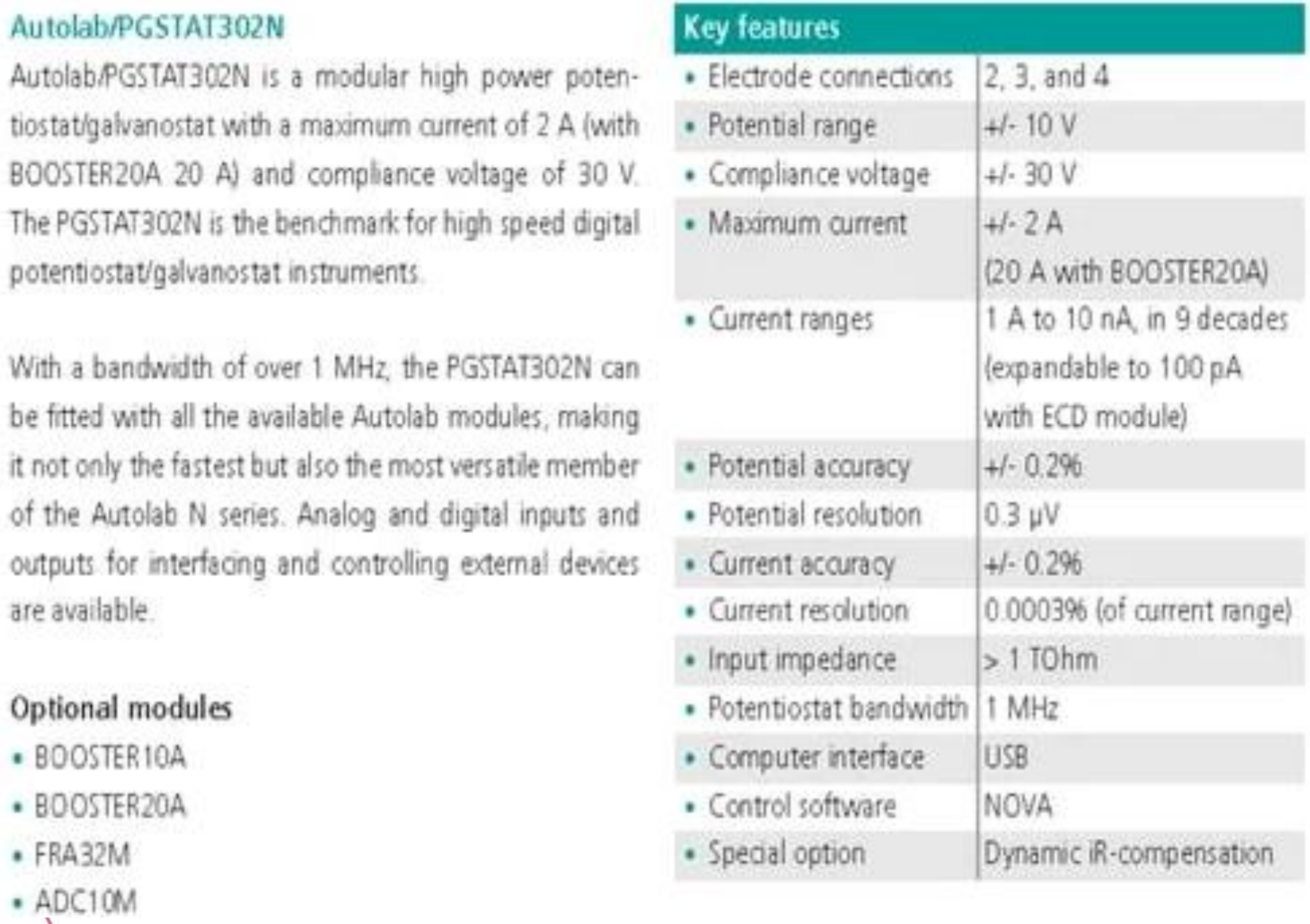


Autolab/PGSTAT302N

Autolab/PGSTAT302N is a modular high power potentiostat/galvanostat with a maximum current of 2 A (with BOOSTER20A 20 A) and compliance voltage of 30 V. The PGSTAT302N is the benchmark for high speed digital potentiostat/galvanostat instruments.

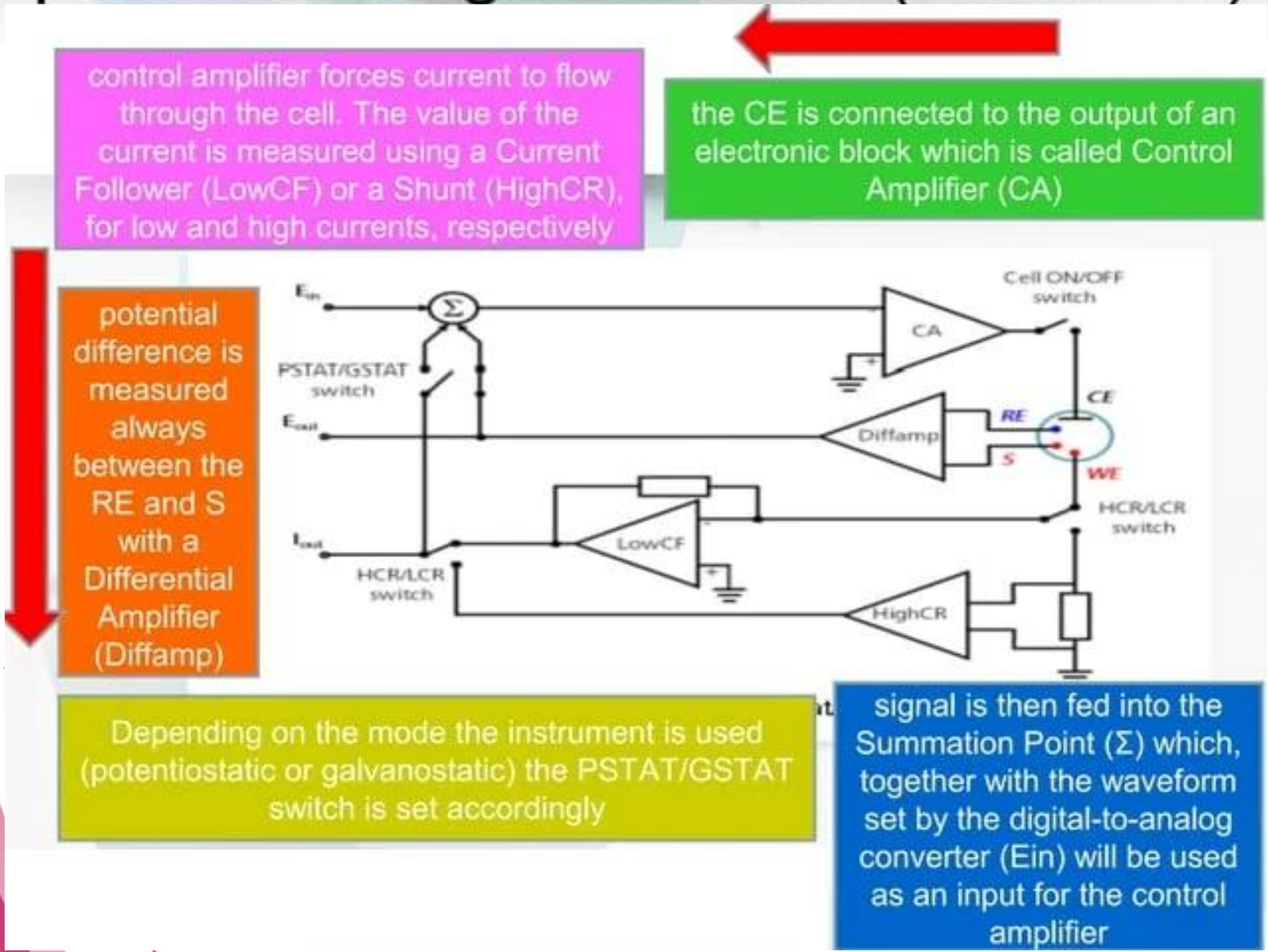
With a bandwidth of over 1 MHz, the PGSTAT302N can be fitted with all the available Autolab modules, making it not only the fastest but also the most versatile member of the Autolab N series. Analog and digital inputs and outputs for interfacing and controlling external devices are available.

Optional modules

- BOOSTER10A
- BOOSTER20A
- FRA32M
- ADC10M

Key features	
• Electrode connections	2, 3, and 4
• Potential range	+/- 10 V
• Compliance voltage	+/- 30 V
• Maximum current	+/- 2 A (20 A with BOOSTER20A)
• Current ranges	1 A to 10 nA, in 9 decades (expandable to 100 pA with ECD module)
• Potential accuracy	+/- 0.2%
• Potential resolution	0.3 µV
• Current accuracy	+/- 0.2%
• Current resolution	0.0003% (of current range)
• Input impedance	> 1 TOhm
• Potentiostat bandwidth	1 MHz
• Computer interface	USB
• Control software	NOVA
• Special option	Dynamic iR-compensation

Basic principle of a potentiostat/galvanostat (PGSTAT)



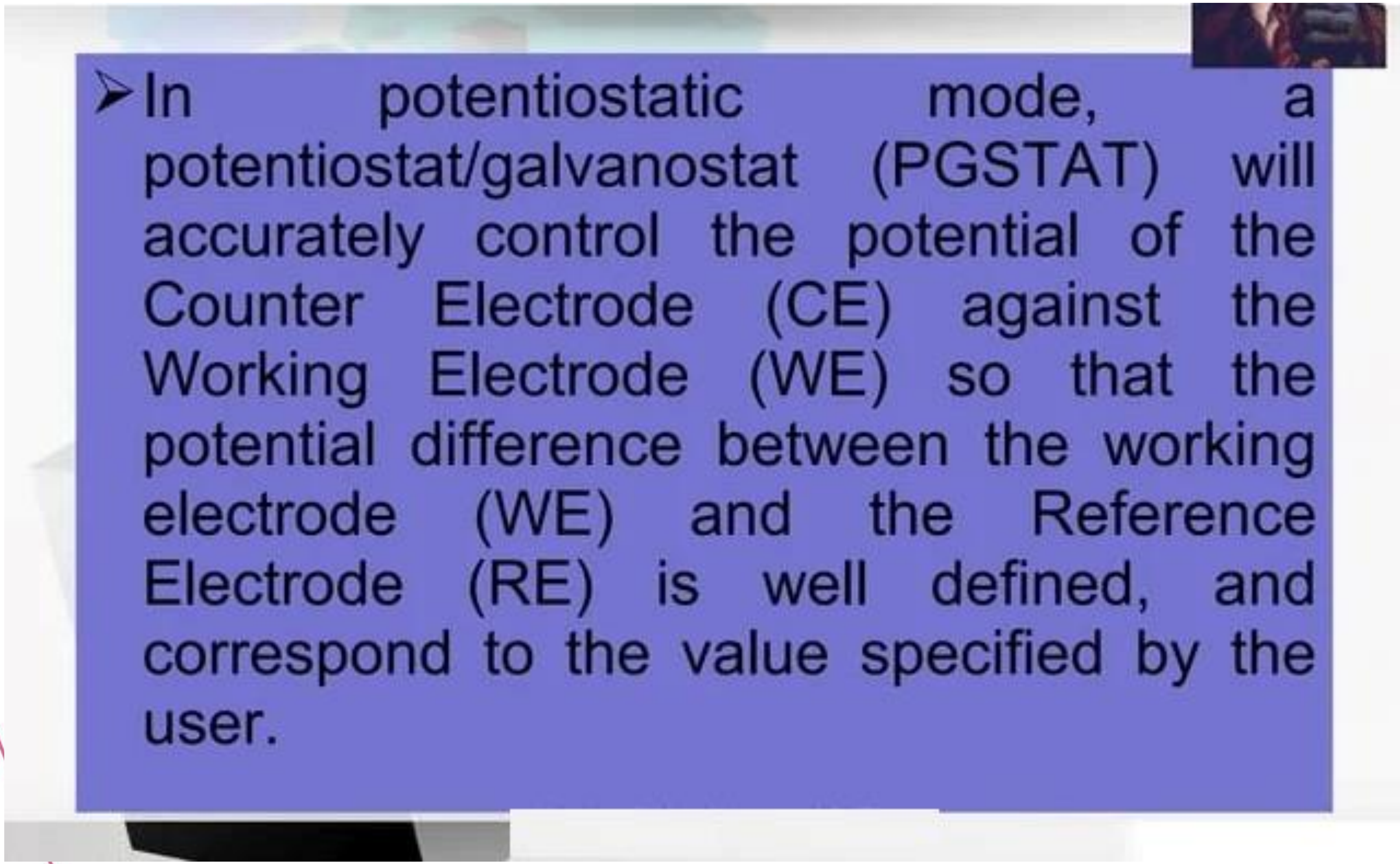
control amplifier forces current to flow through the cell. The value of the current is measured using a Current Follower (LowCF) or a Shunt (HighCR), for low and high currents, respectively

the CE is connected to the output of an electronic block which is called Control Amplifier (CA)

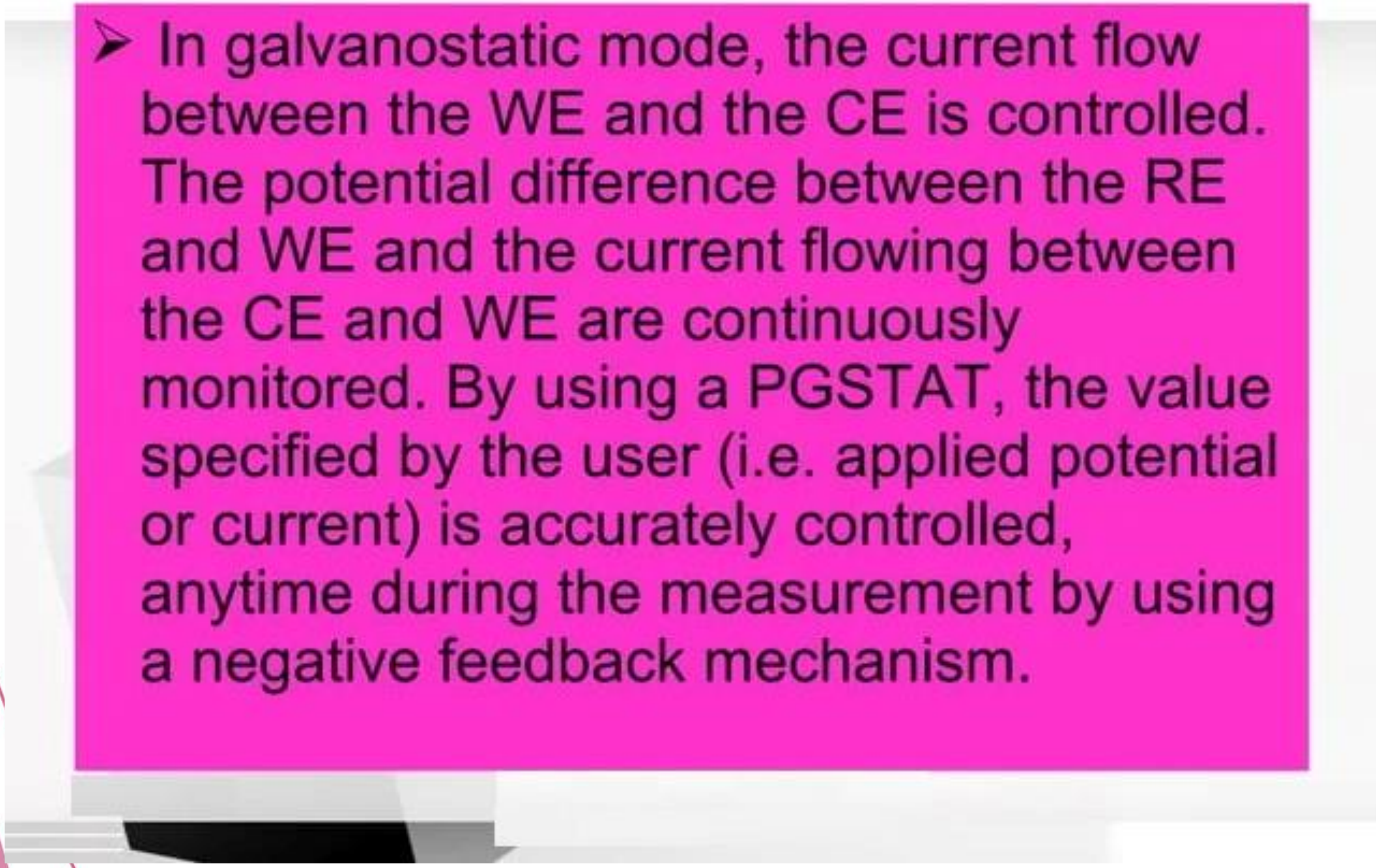
potential difference is measured always between the RE and S with a Differential Amplifier (Diffamp)

Depending on the mode the instrument is used (potentiostatic or galvanostatic) the PSTAT/GSTAT switch is set accordingly

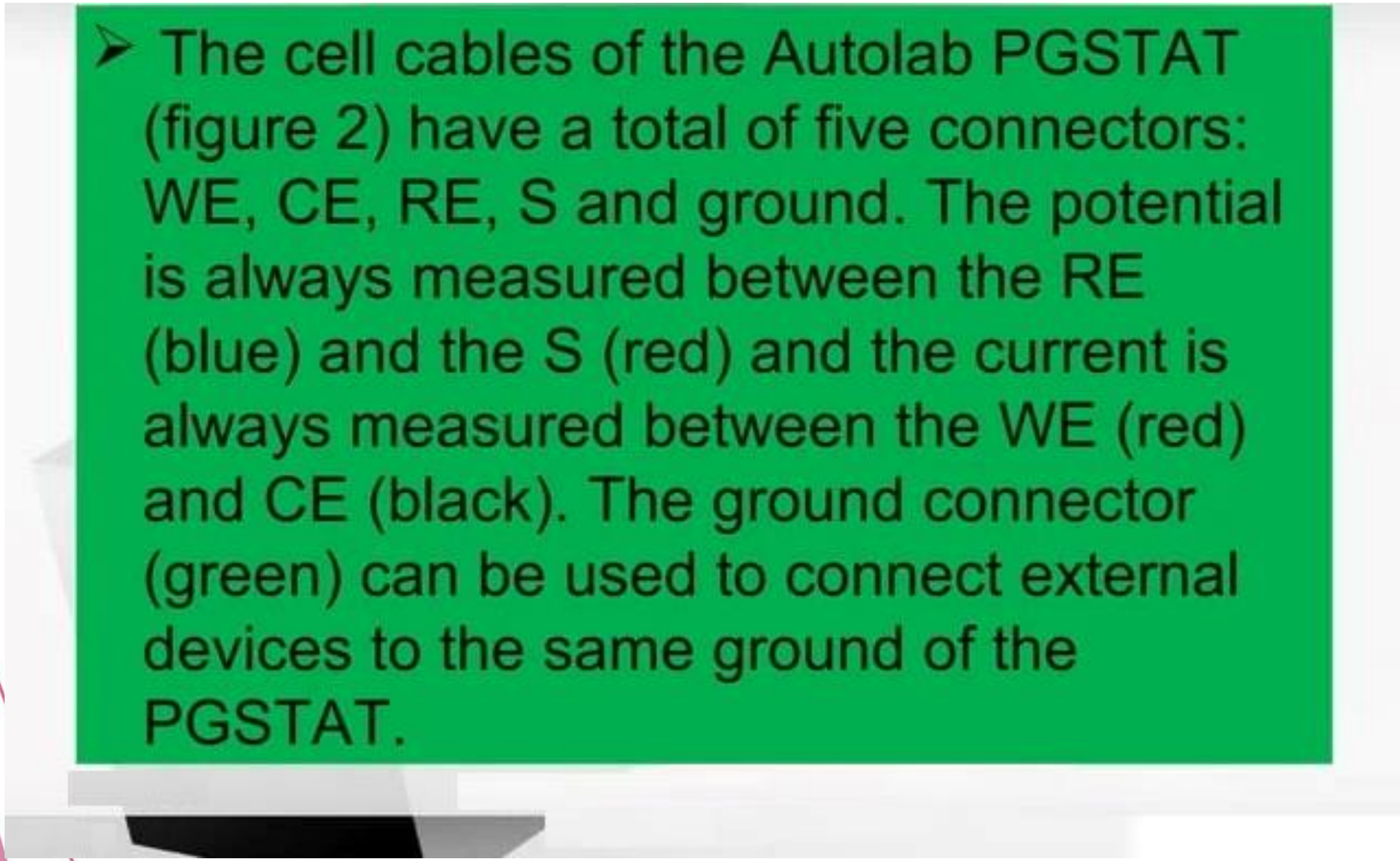
signal is then fed into the Summation Point (Σ) which, together with the waveform set by the digital-to-analog converter (Ein) will be used as an input for the control amplifier



- In potentiostatic mode, a potentiostat/galvanostat (PGSTAT) will accurately control the potential of the Counter Electrode (CE) against the Working Electrode (WE) so that the potential difference between the working electrode (WE) and the Reference Electrode (RE) is well defined, and correspond to the value specified by the user.



- In galvanostatic mode, the current flow between the WE and the CE is controlled. The potential difference between the RE and WE and the current flowing between the CE and WE are continuously monitored. By using a PGSTAT, the value specified by the user (i.e. applied potential or current) is accurately controlled, anytime during the measurement by using a negative feedback mechanism.



- The cell cables of the Autolab PGSTAT (figure 2) have a total of five connectors: WE, CE, RE, S and ground. The potential is always measured between the RE (blue) and the S (red) and the current is always measured between the WE (red) and CE (black). The ground connector (green) can be used to connect external devices to the same ground of the PGSTAT.

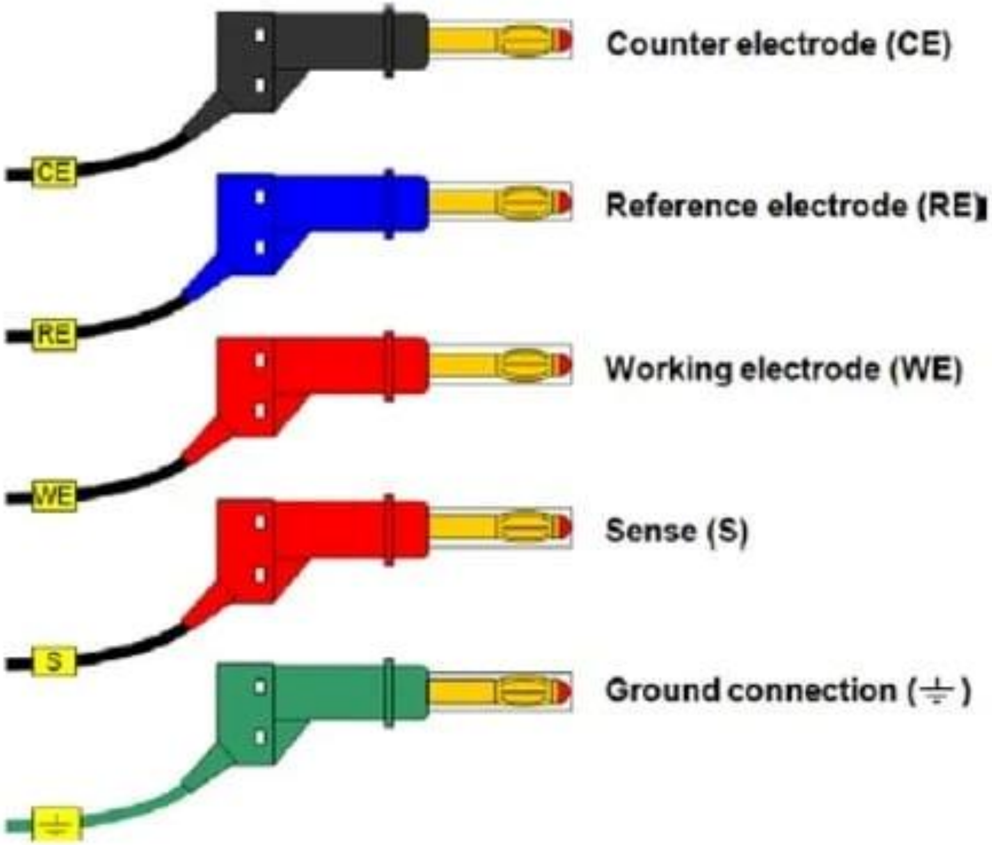
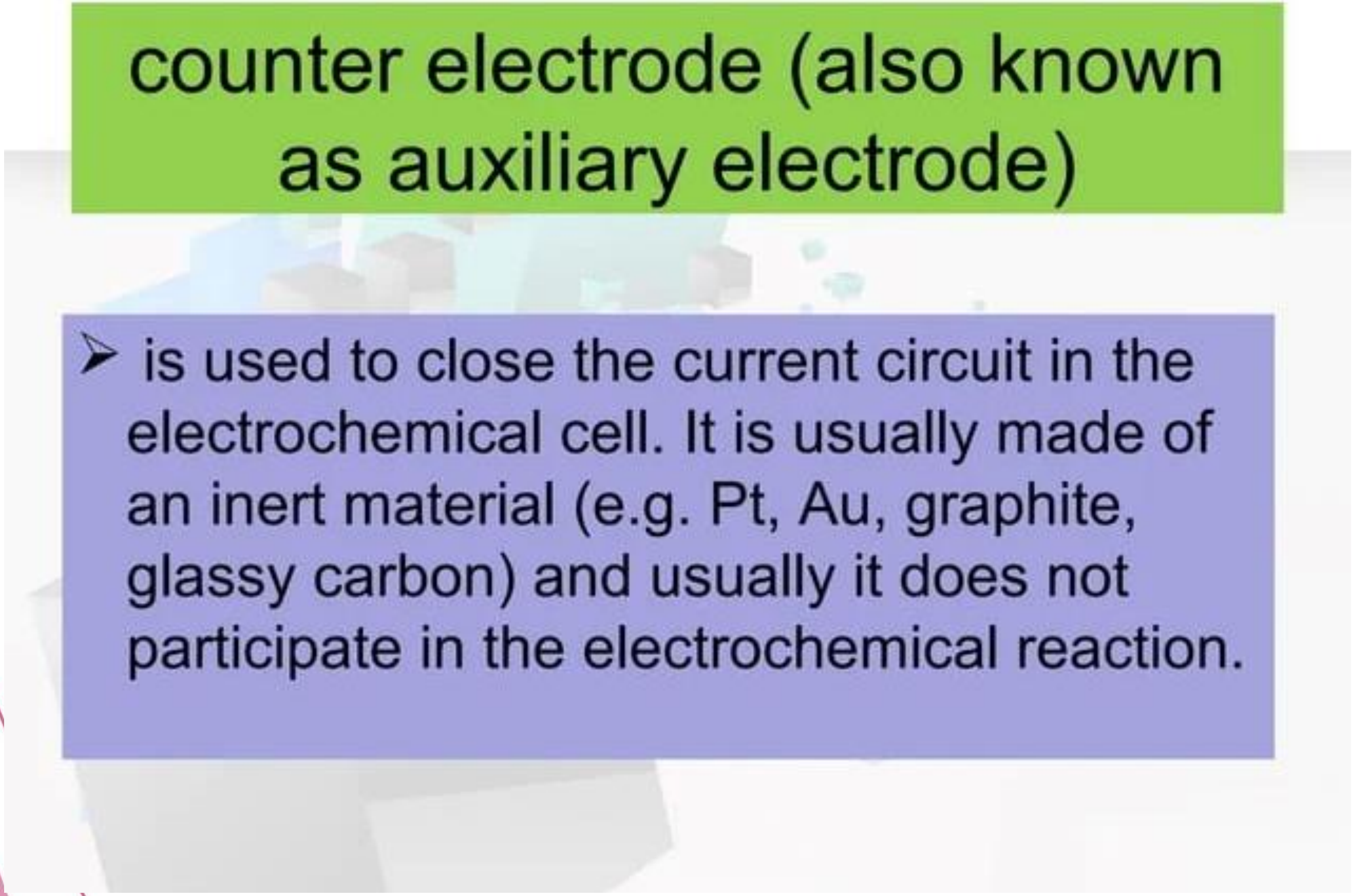
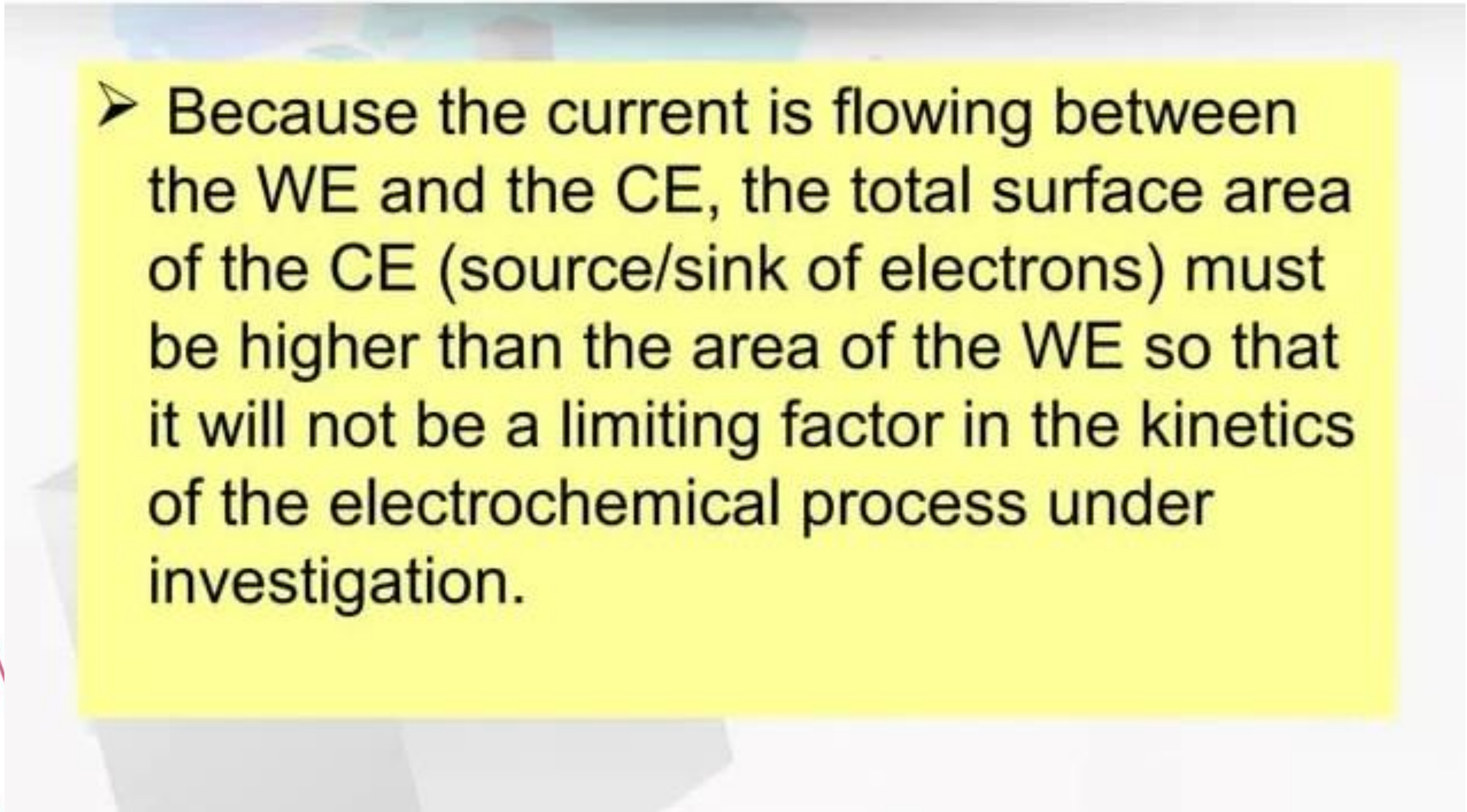


Figure 2 - The connections available on the cell cables of the Autolab PGSTAT and the color code used

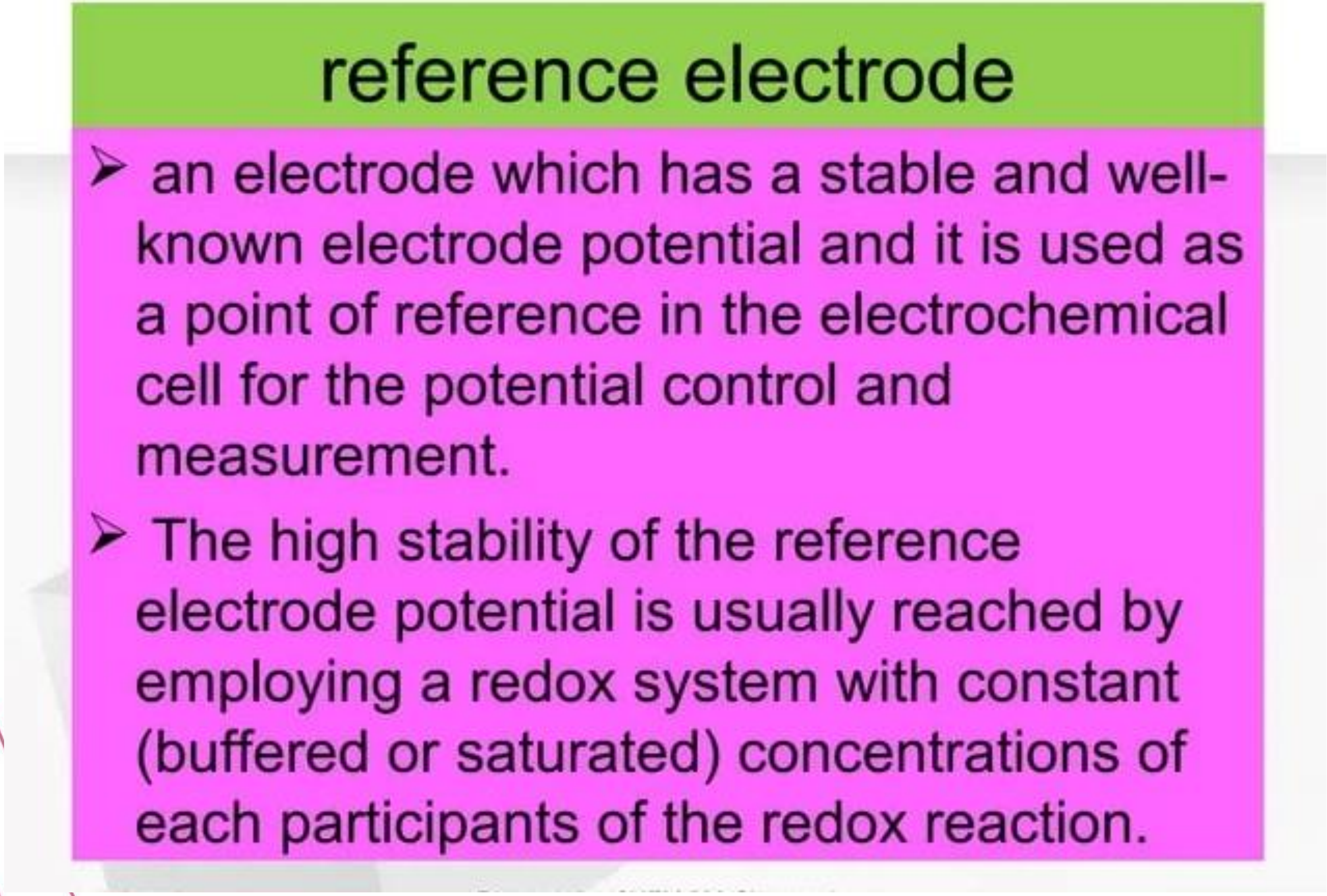


counter electrode (also known as auxiliary electrode)

- is used to close the current circuit in the electrochemical cell. It is usually made of an inert material (e.g. Pt, Au, graphite, glassy carbon) and usually it does not participate in the electrochemical reaction.

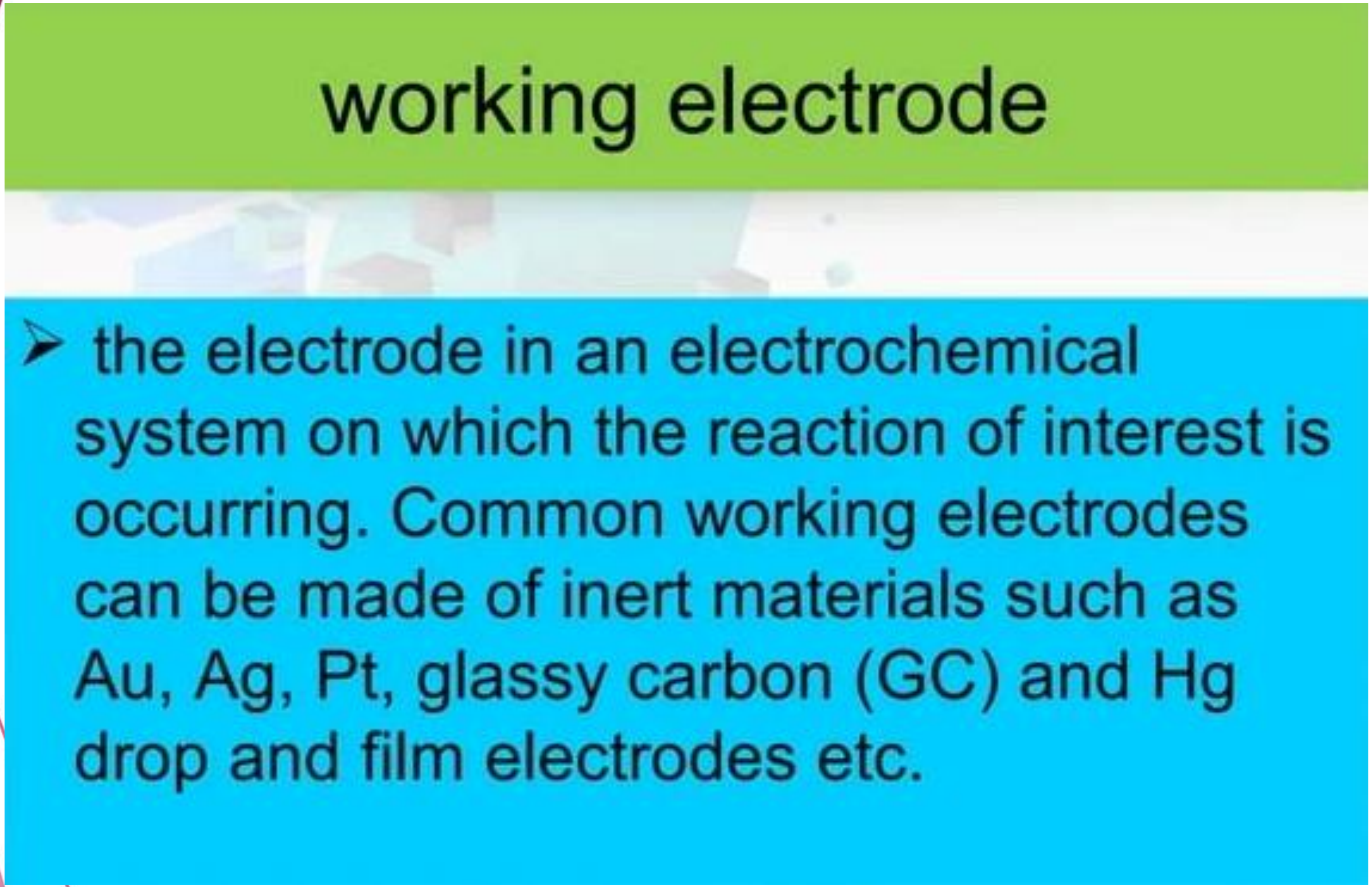


- Because the current is flowing between the WE and the CE, the total surface area of the CE (source/sink of electrons) must be higher than the area of the WE so that it will not be a limiting factor in the kinetics of the electrochemical process under investigation.



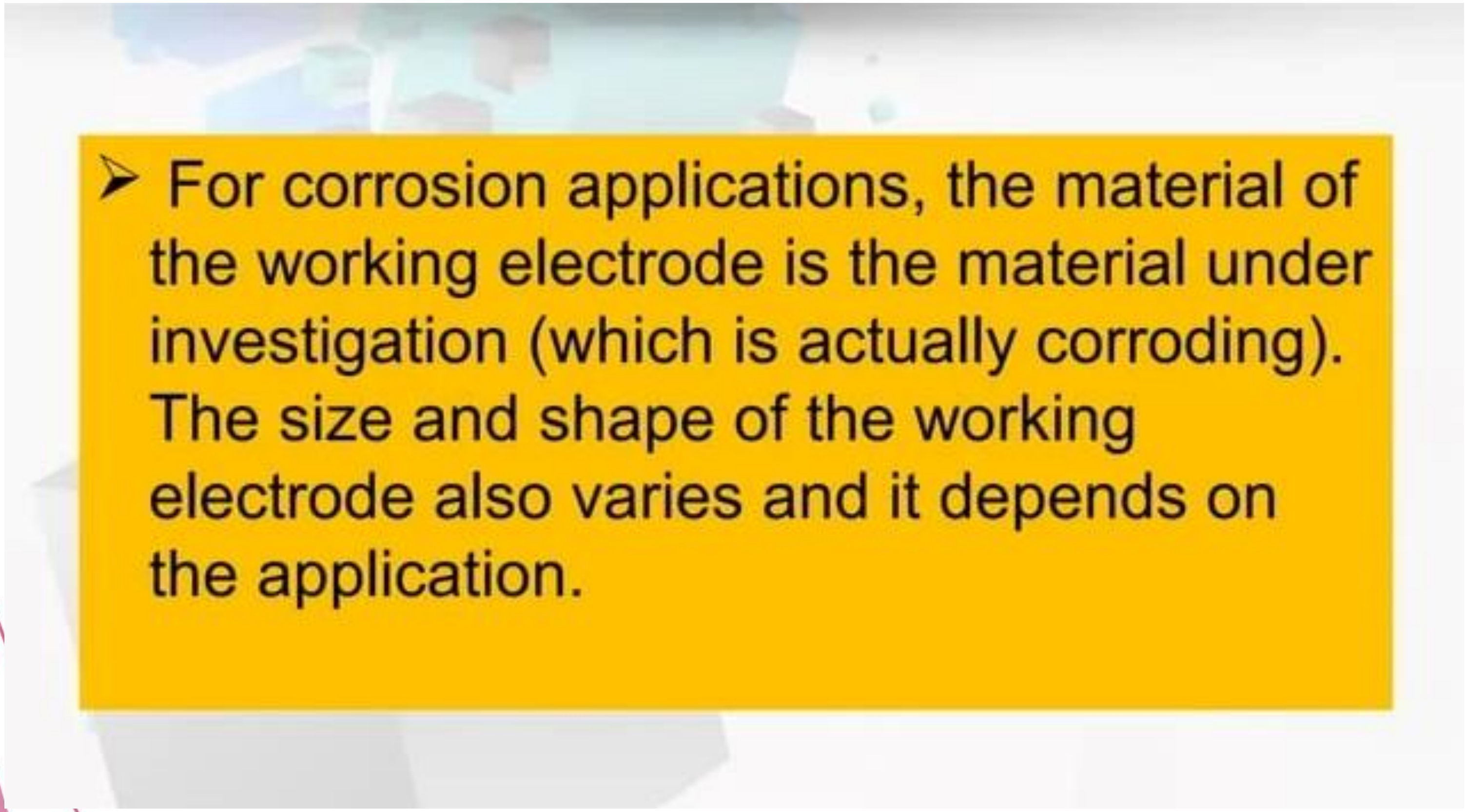
reference electrode

- an electrode which has a stable and well-known electrode potential and it is used as a point of reference in the electrochemical cell for the potential control and measurement.
- The high stability of the reference electrode potential is usually reached by employing a redox system with constant (buffered or saturated) concentrations of each participants of the redox reaction.

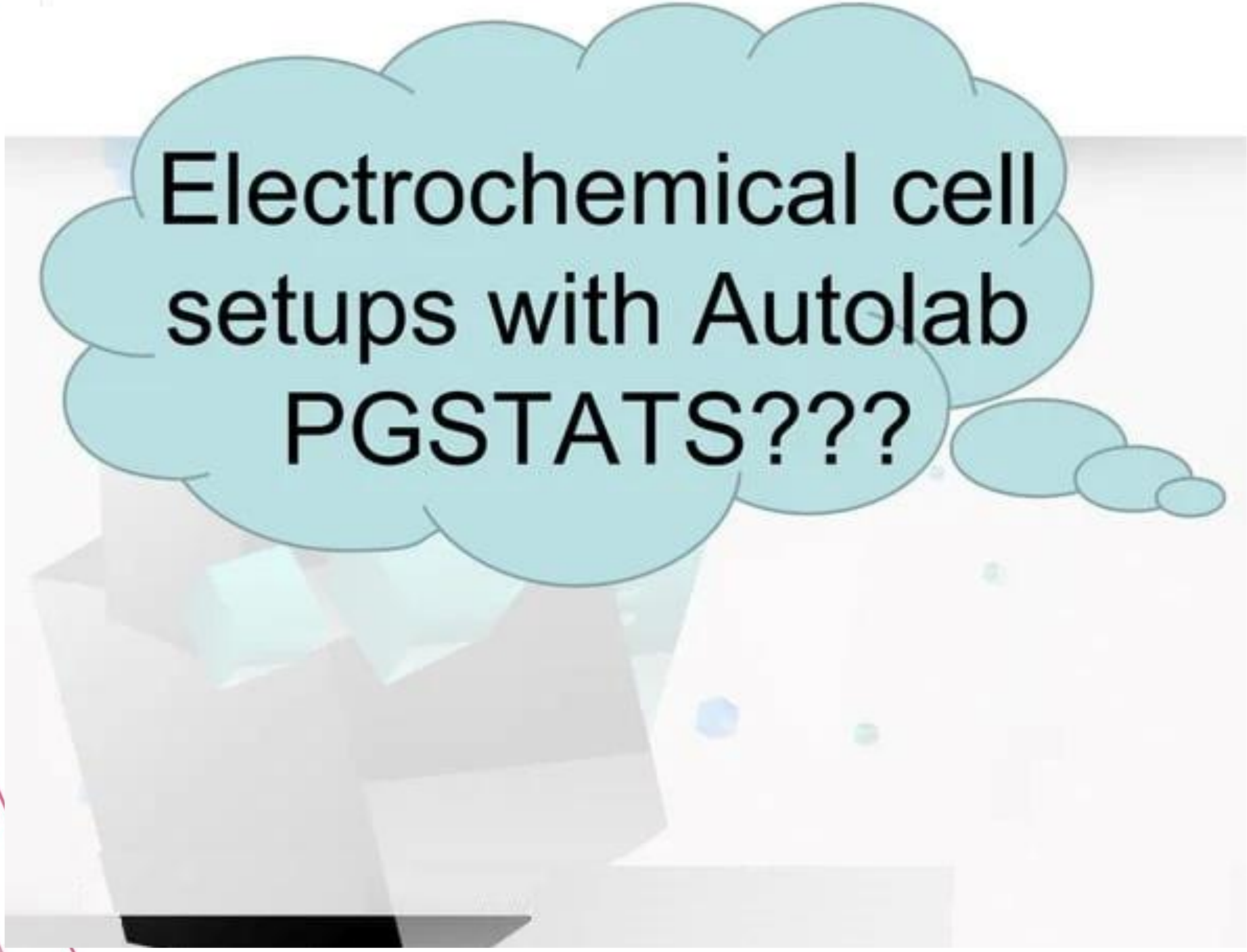


working electrode

- the electrode in an electrochemical system on which the reaction of interest is occurring. Common working electrodes can be made of inert materials such as Au, Ag, Pt, glassy carbon (GC) and Hg drop and film electrodes etc.



- For corrosion applications, the material of the working electrode is the material under investigation (which is actually corroding). The size and shape of the working electrode also varies and it depends on the application.



Electrochemical cell setups with Autolab PGSTATS???

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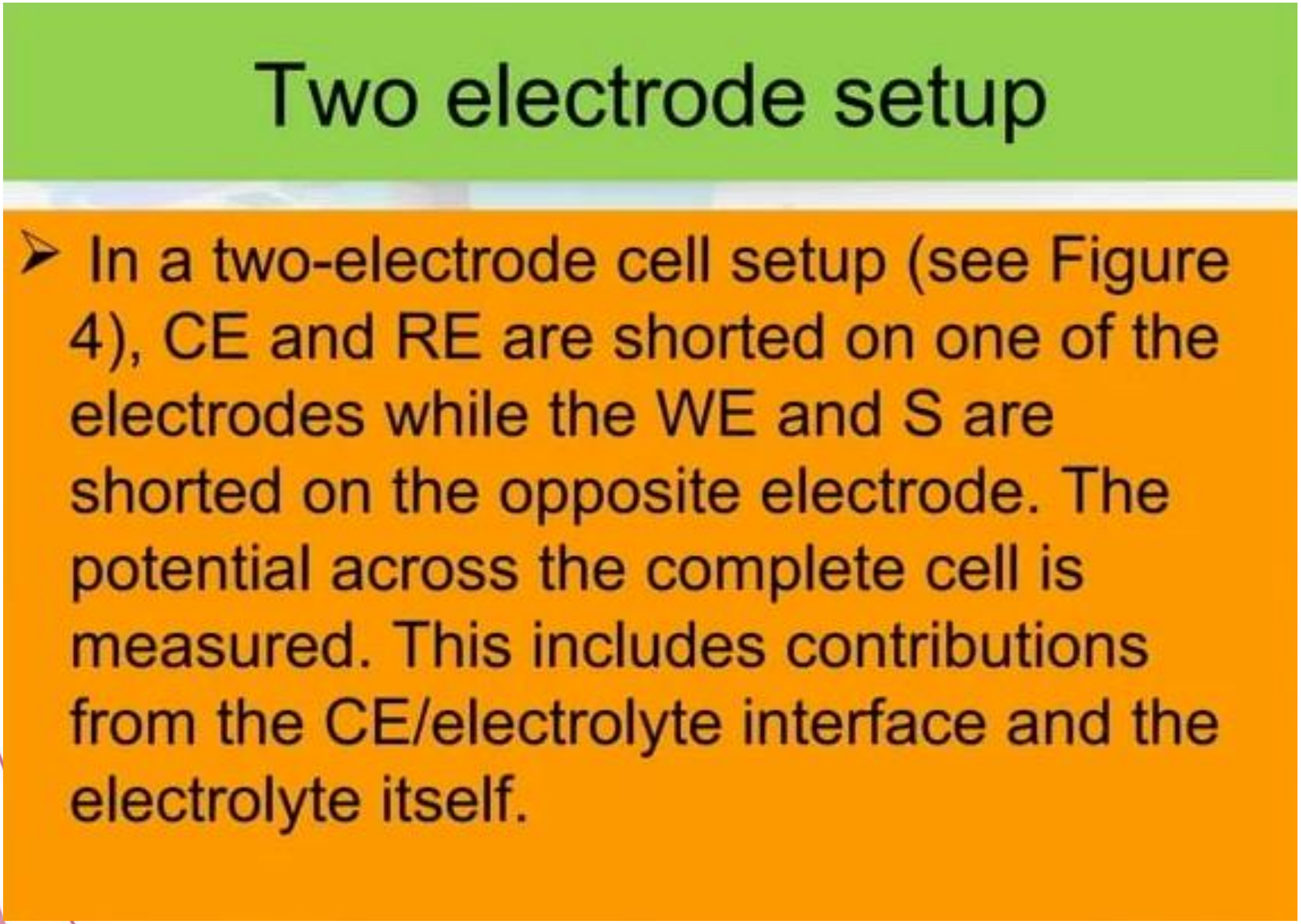


Two electrode setup

- In a two-electrode cell setup (see Figure 4), CE and RE are shorted on one of the electrodes while the WE and S are shorted on the opposite electrode. The potential across the complete cell is measured. This includes contributions from the CE/electrolyte interface and the electrolyte itself.

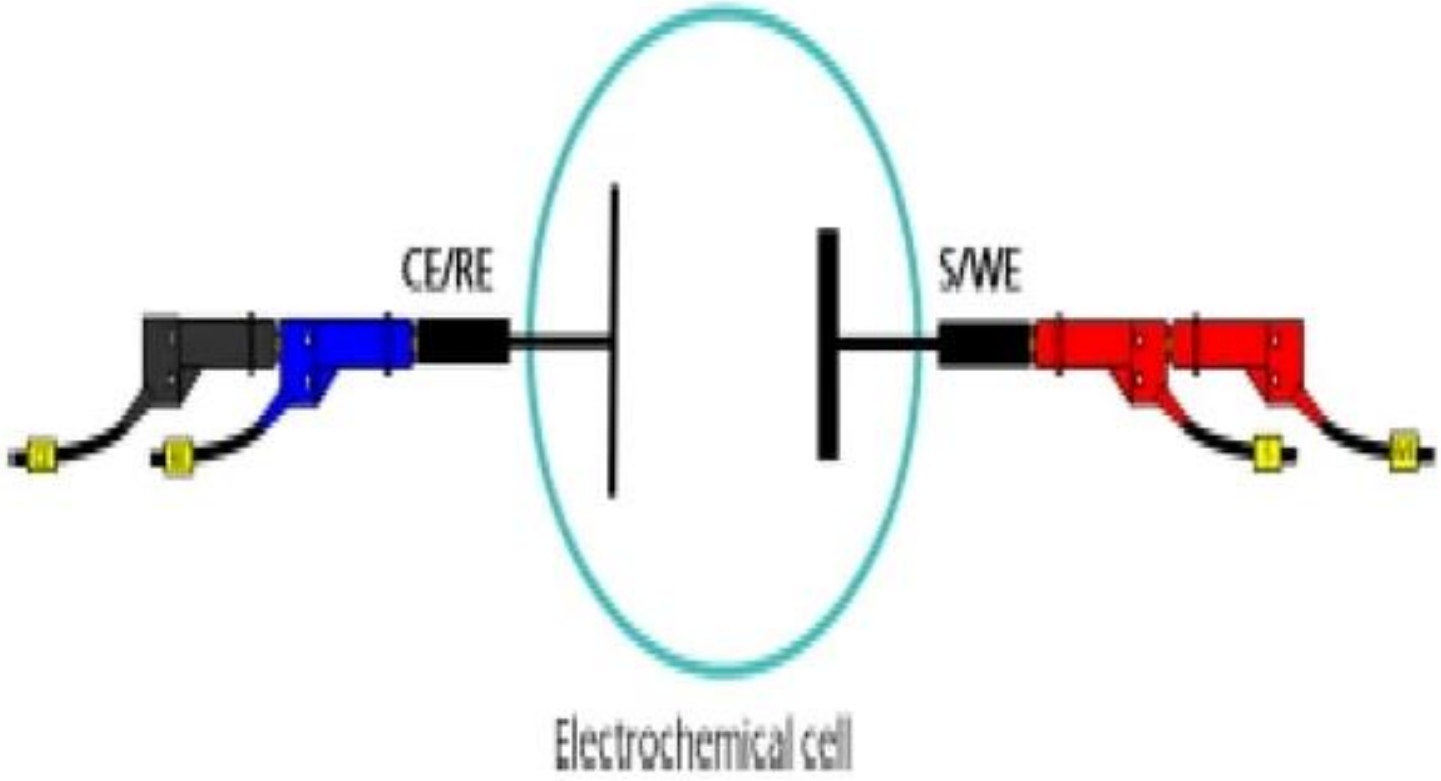
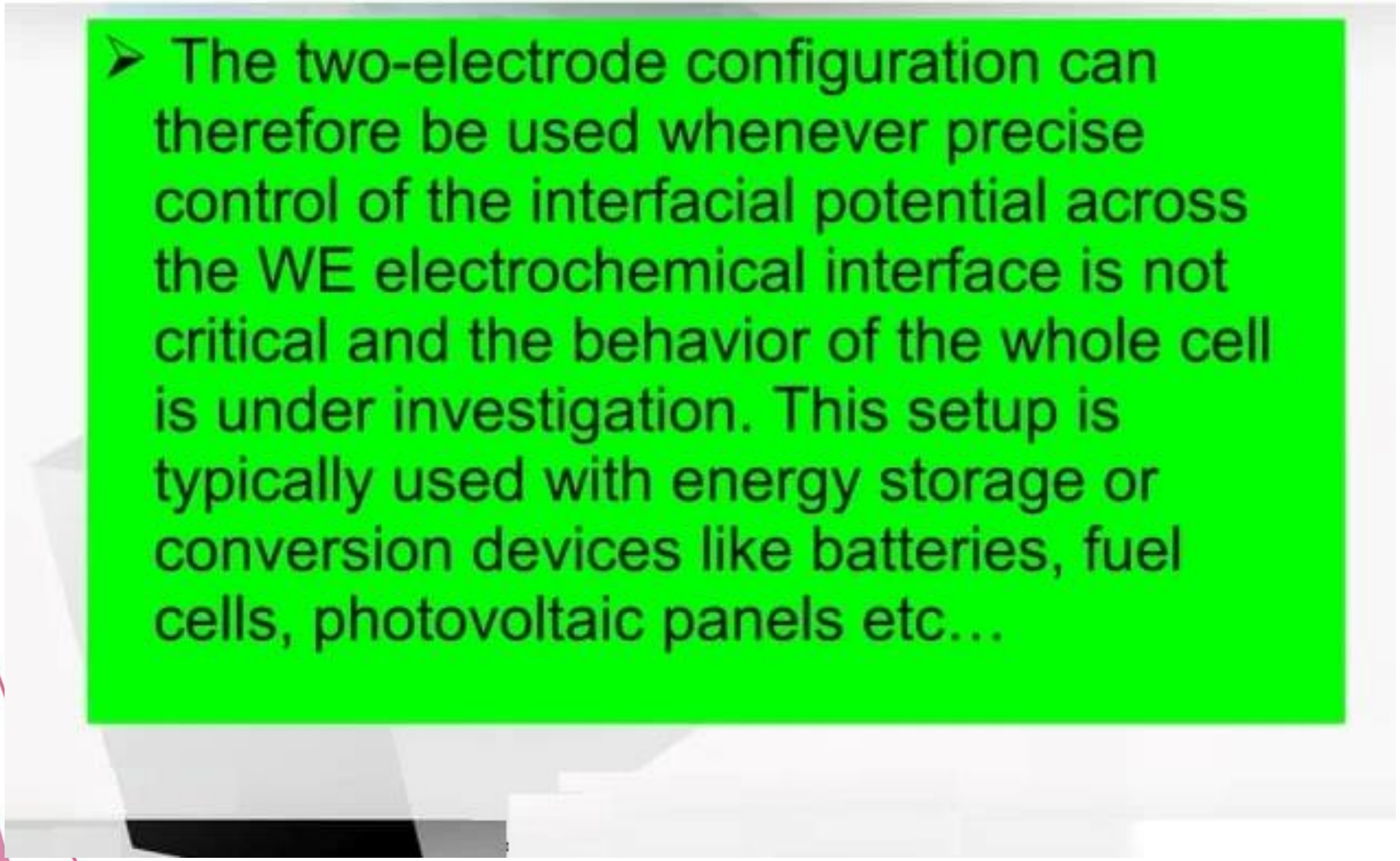
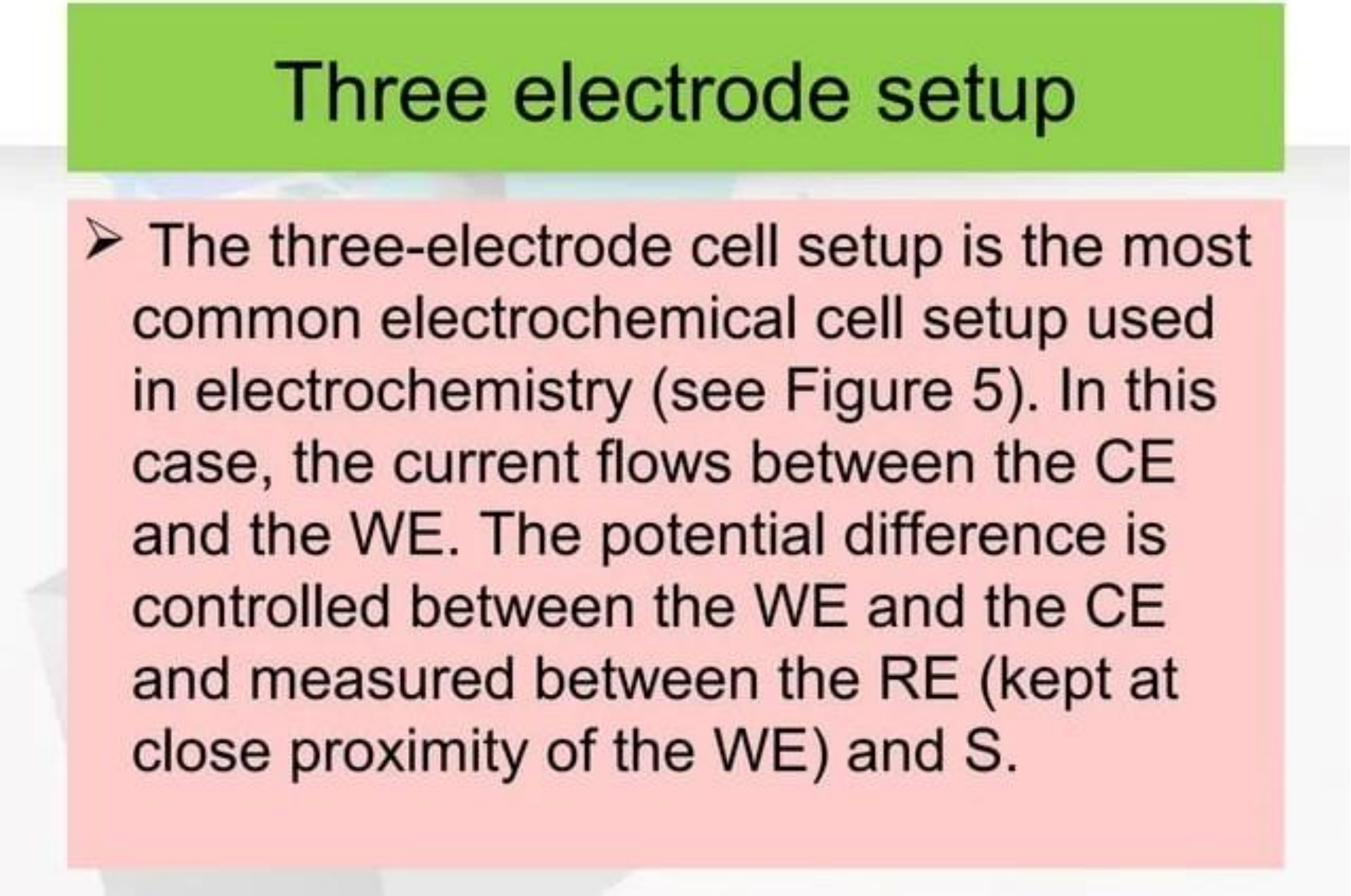


Figure 4 – Schematic view of the 2 electrode setup



- The two-electrode configuration can therefore be used whenever precise control of the interfacial potential across the WE electrochemical interface is not critical and the behavior of the whole cell is under investigation. This setup is typically used with energy storage or conversion devices like batteries, fuel cells, photovoltaic panels etc…



Three electrode setup

- The three-electrode cell setup is the most common electrochemical cell setup used in electrochemistry (see Figure 5). In this case, the current flows between the CE and the WE. The potential difference is controlled between the WE and the CE and measured between the RE (kept at close proximity of the WE) and S.

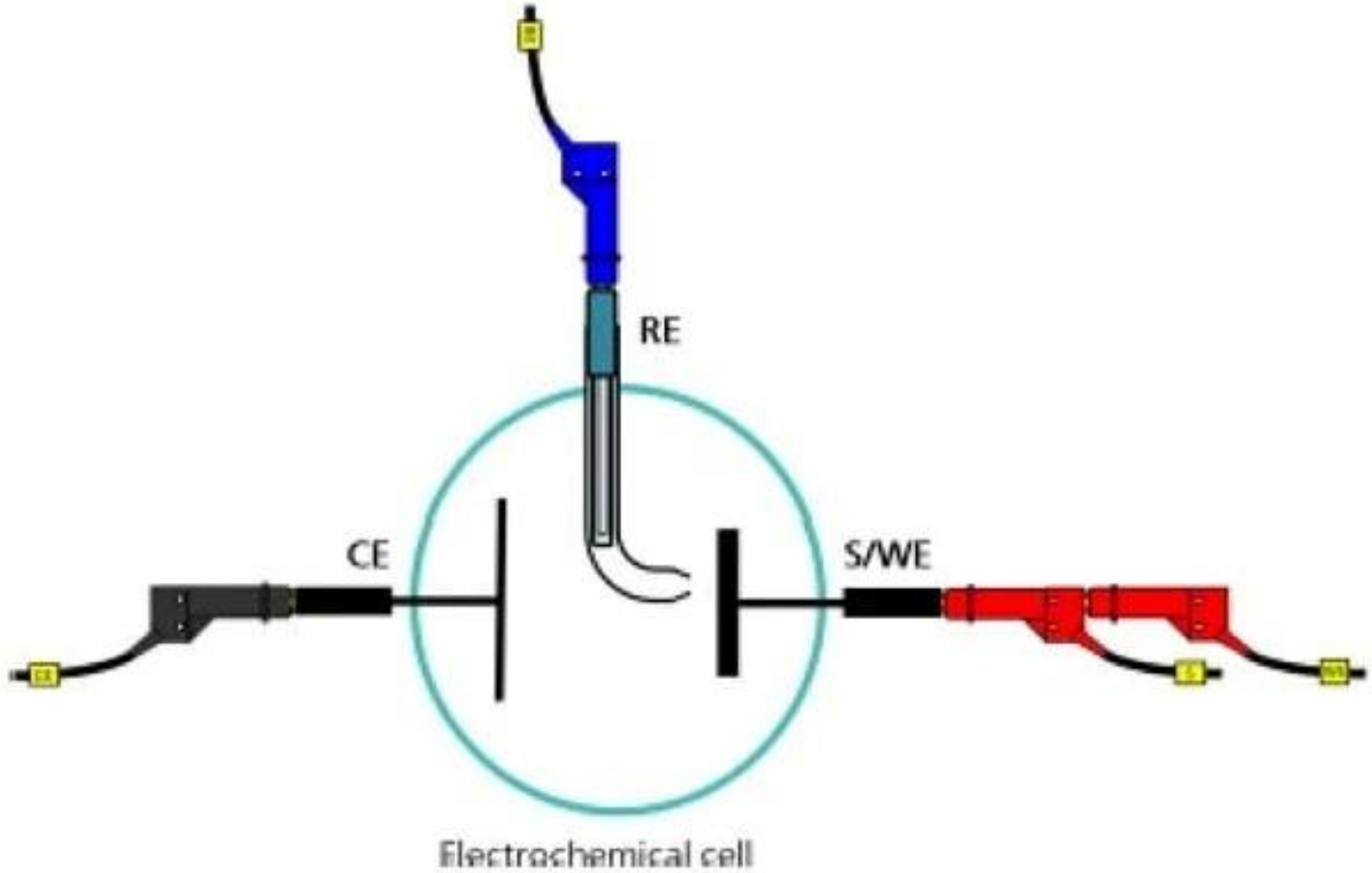
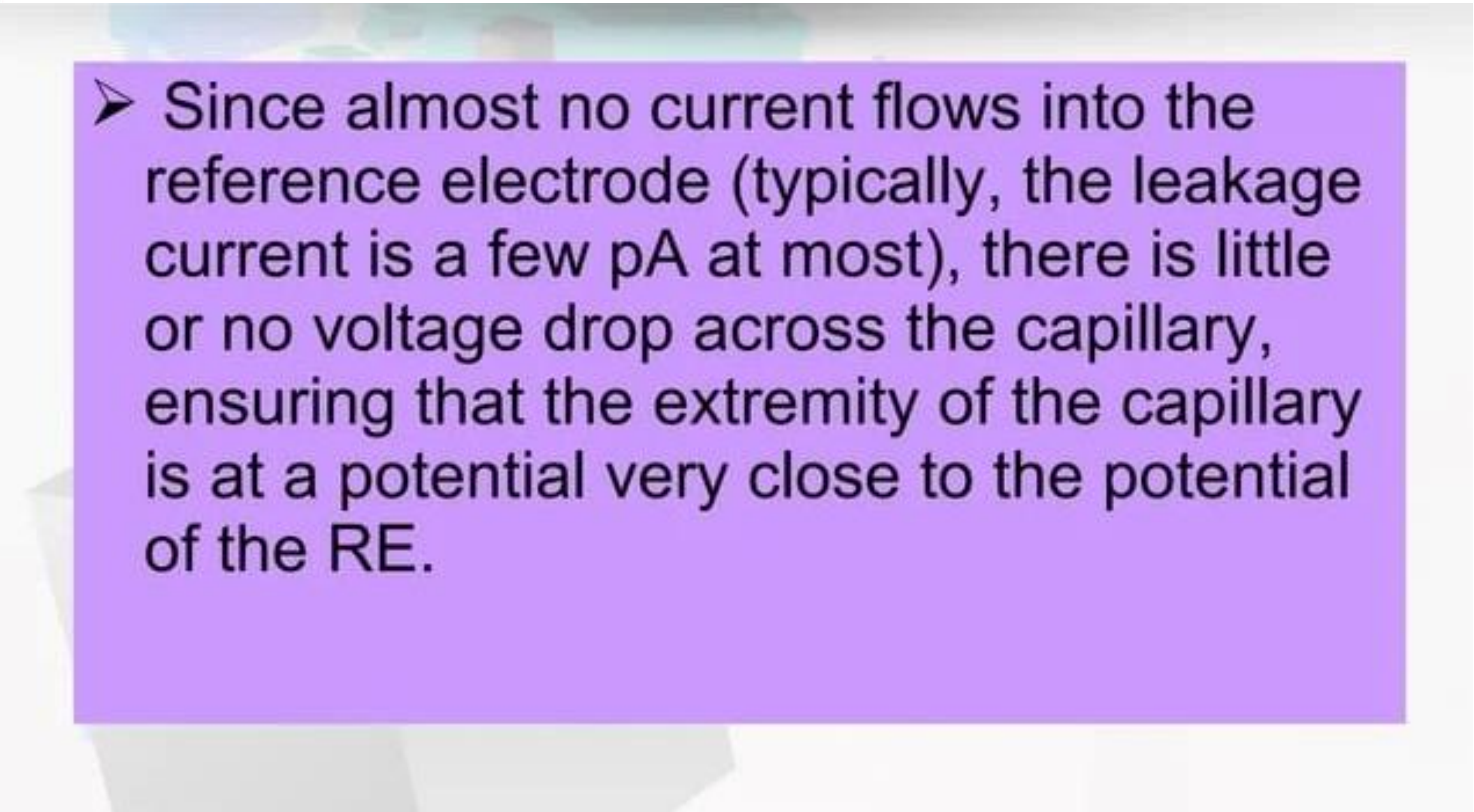
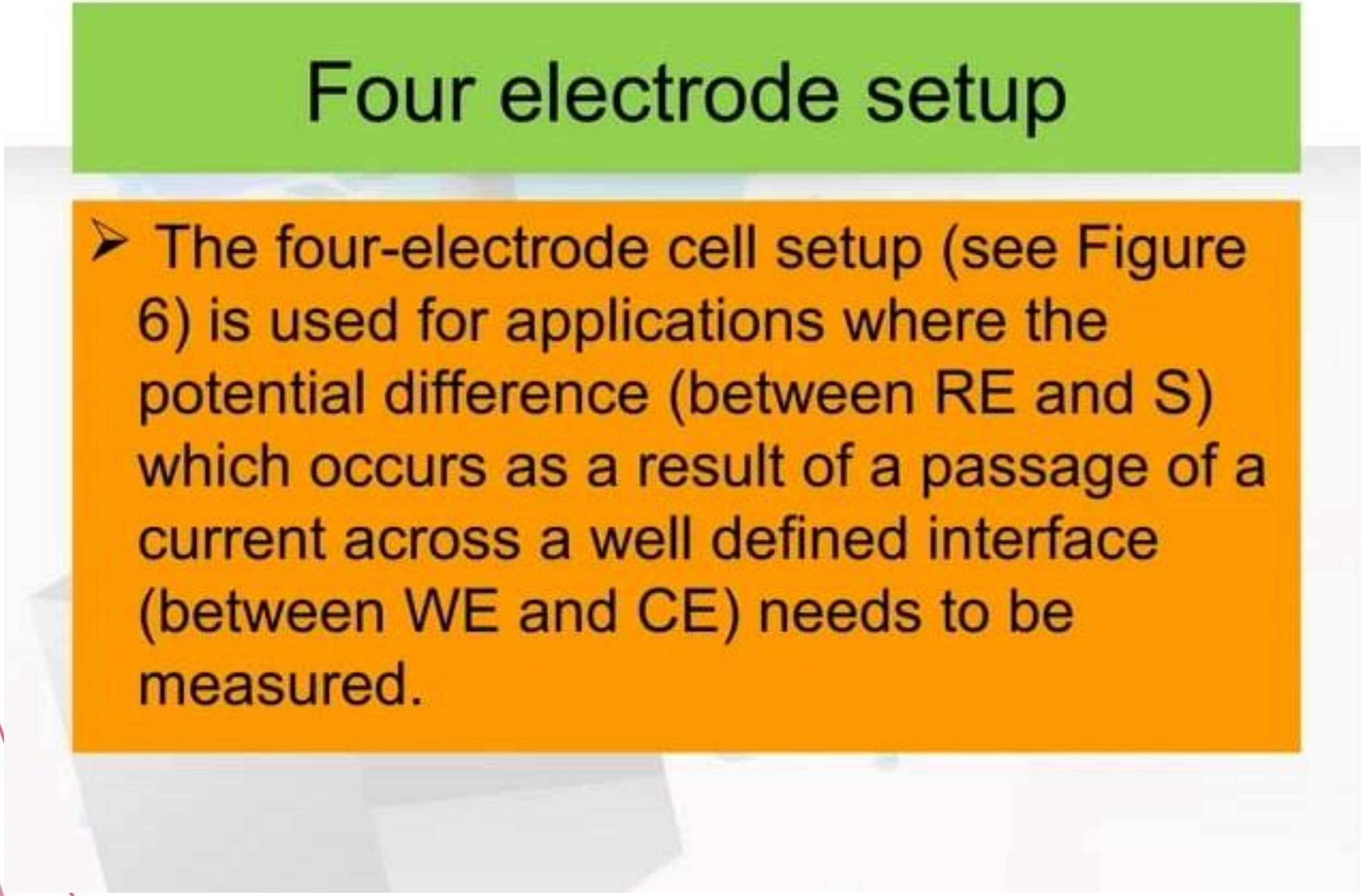


Figure 5 – Schematic view of the 3 electrode setup

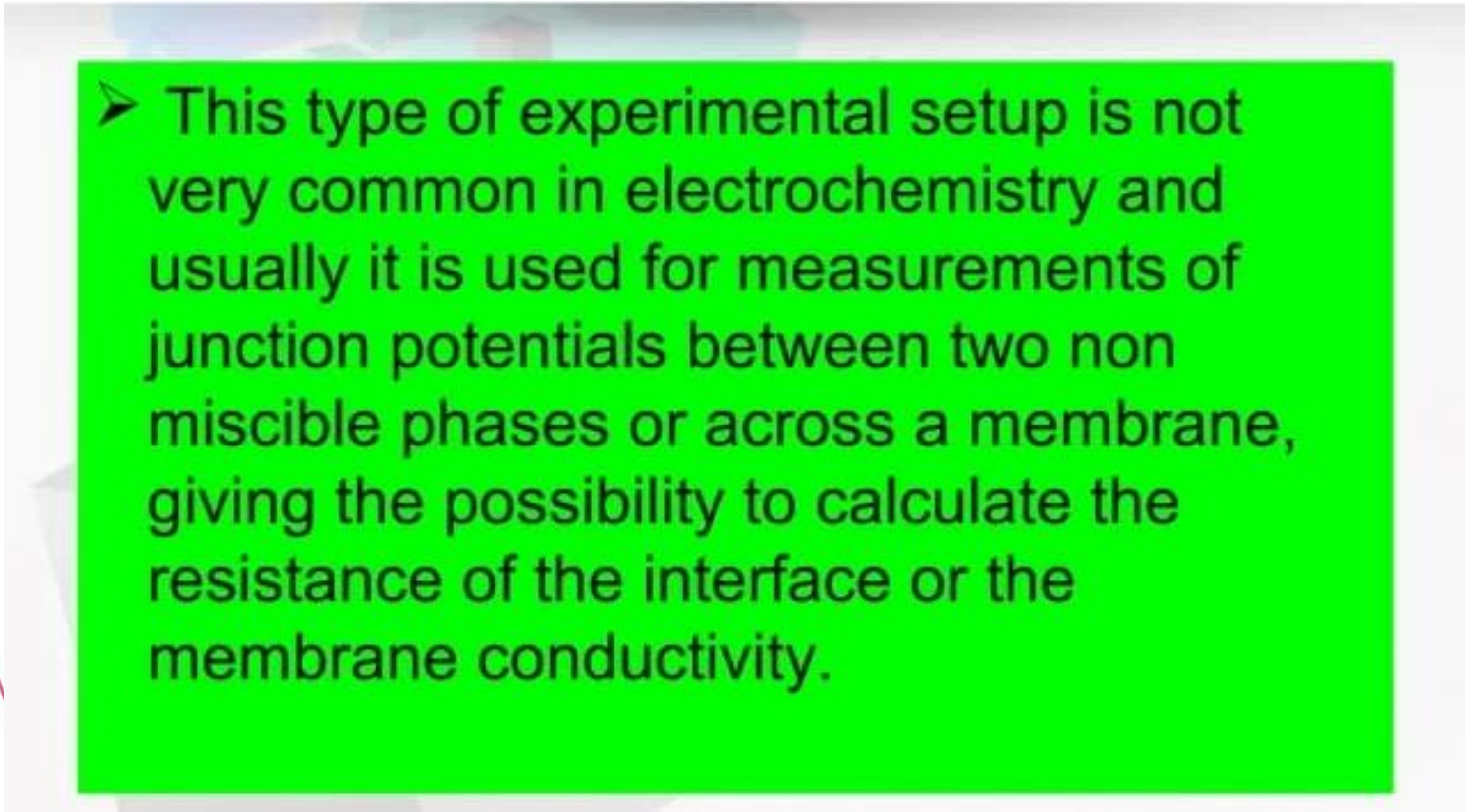


- Since almost no current flows into the reference electrode (typically, the leakage current is a few pA at most), there is little or no voltage drop across the capillary, ensuring that the extremity of the capillary is at a potential very close to the potential of the RE.



Four electrode setup

- The four-electrode cell setup (see Figure 6) is used for applications where the potential difference (between RE and S) which occurs as a result of a passage of a current across a well defined interface (between WE and CE) needs to be measured.



- This type of experimental setup is not very common in electrochemistry and usually it is used for measurements of junction potentials between two non miscible phases or across a membrane, giving the possibility to calculate the resistance of the interface or the membrane conductivity.

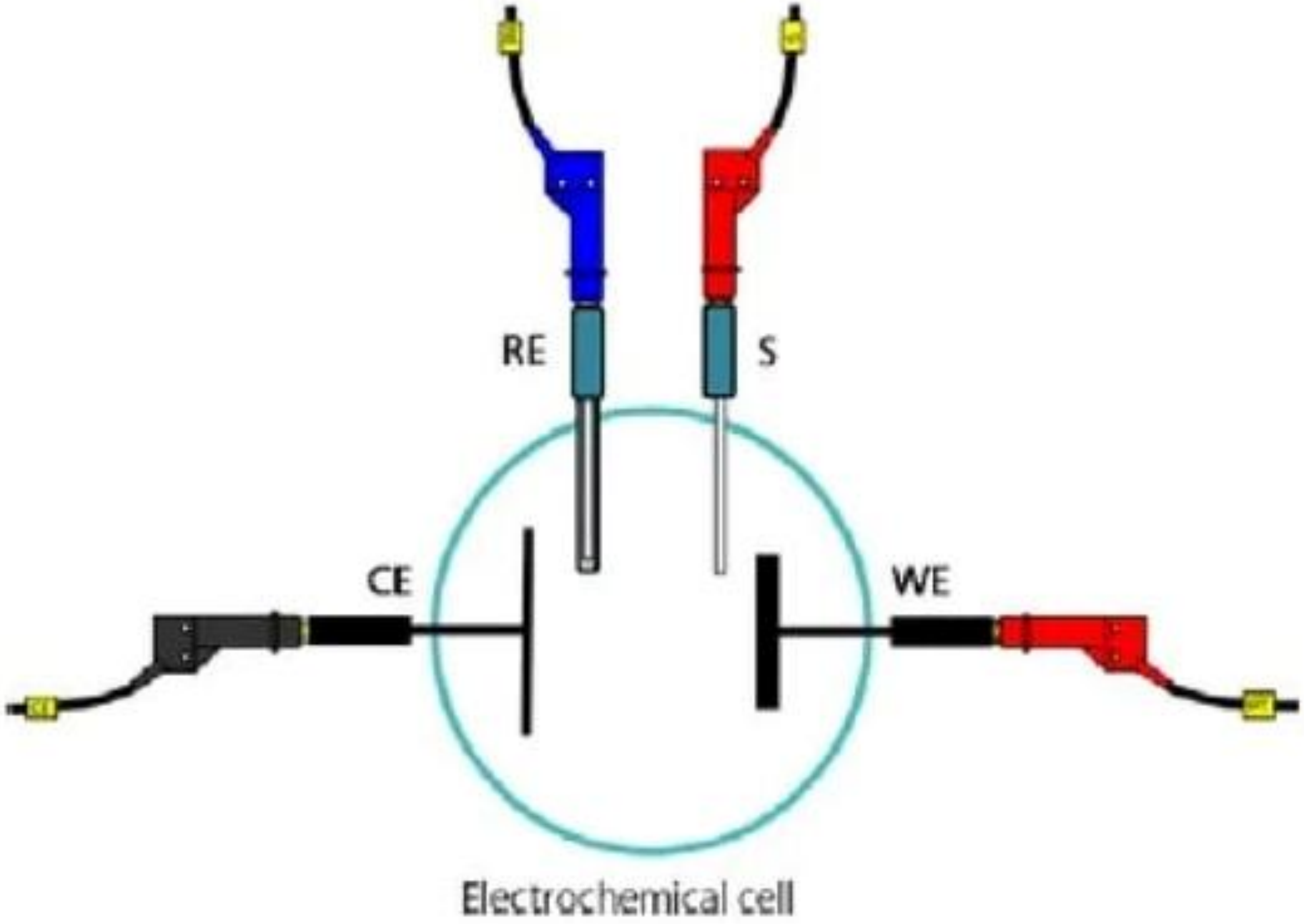


Figure 6 – Schematic view of the 4 electrode setup

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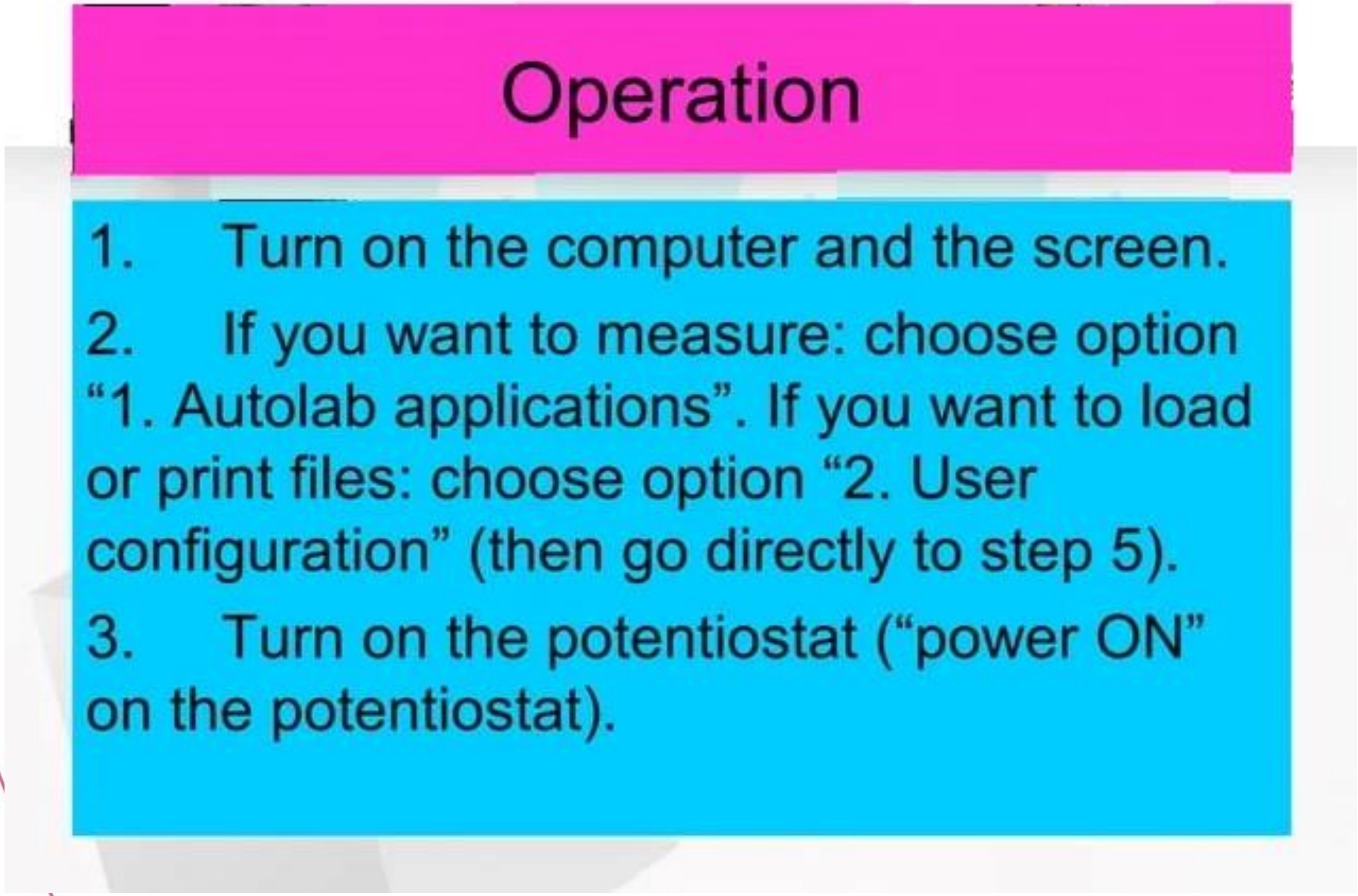
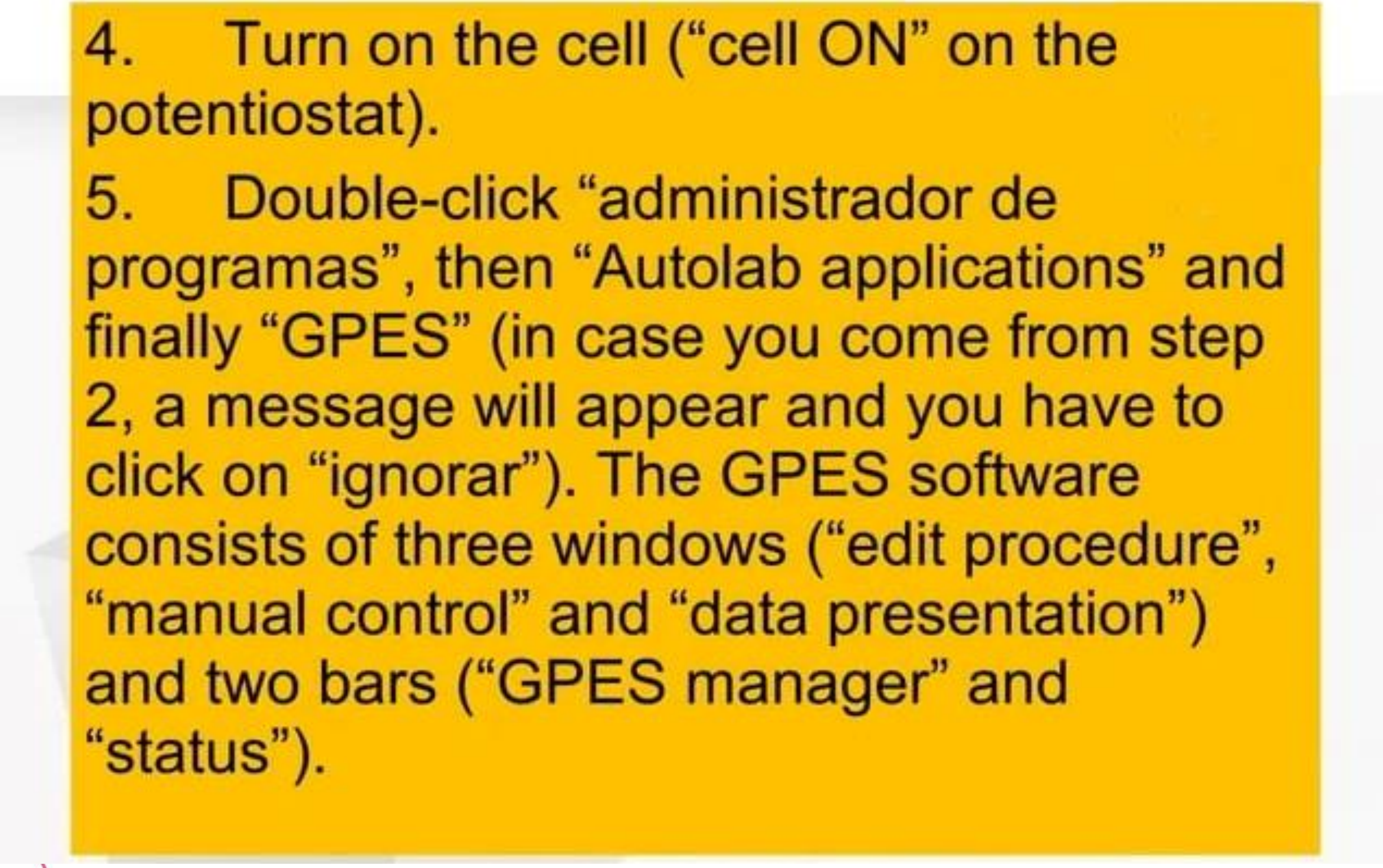


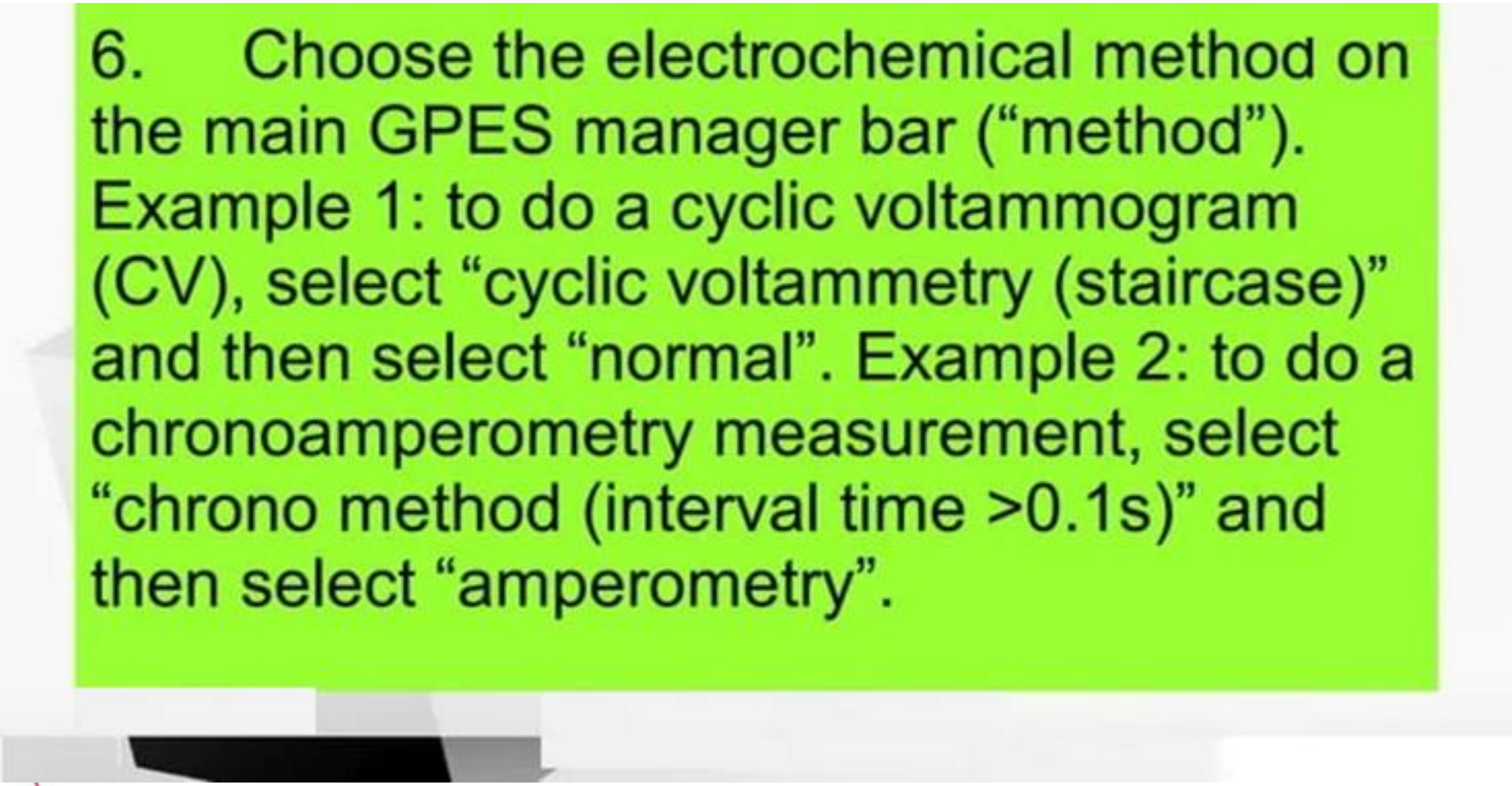
Operation

1. Turn on the computer and the screen.
2. If you want to measure: choose option "1. Autolab applications". If you want to load or print files: choose option "2. User configuration" (then go directly to step 5).
3. Turn on the potentiostat ("power ON" on the potentiostat).

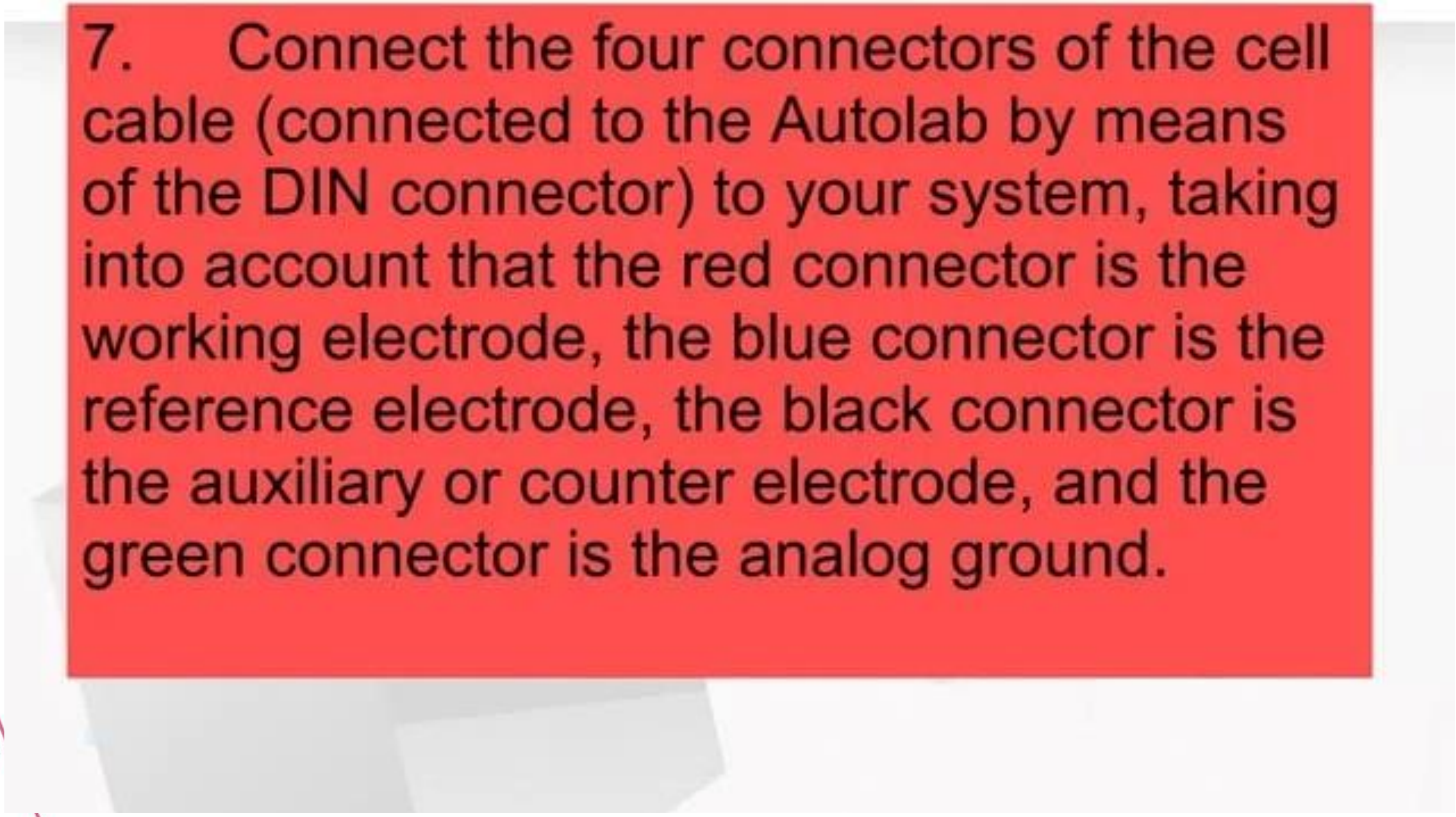


4. Turn on the cell (“cell ON” on the potentiostat).

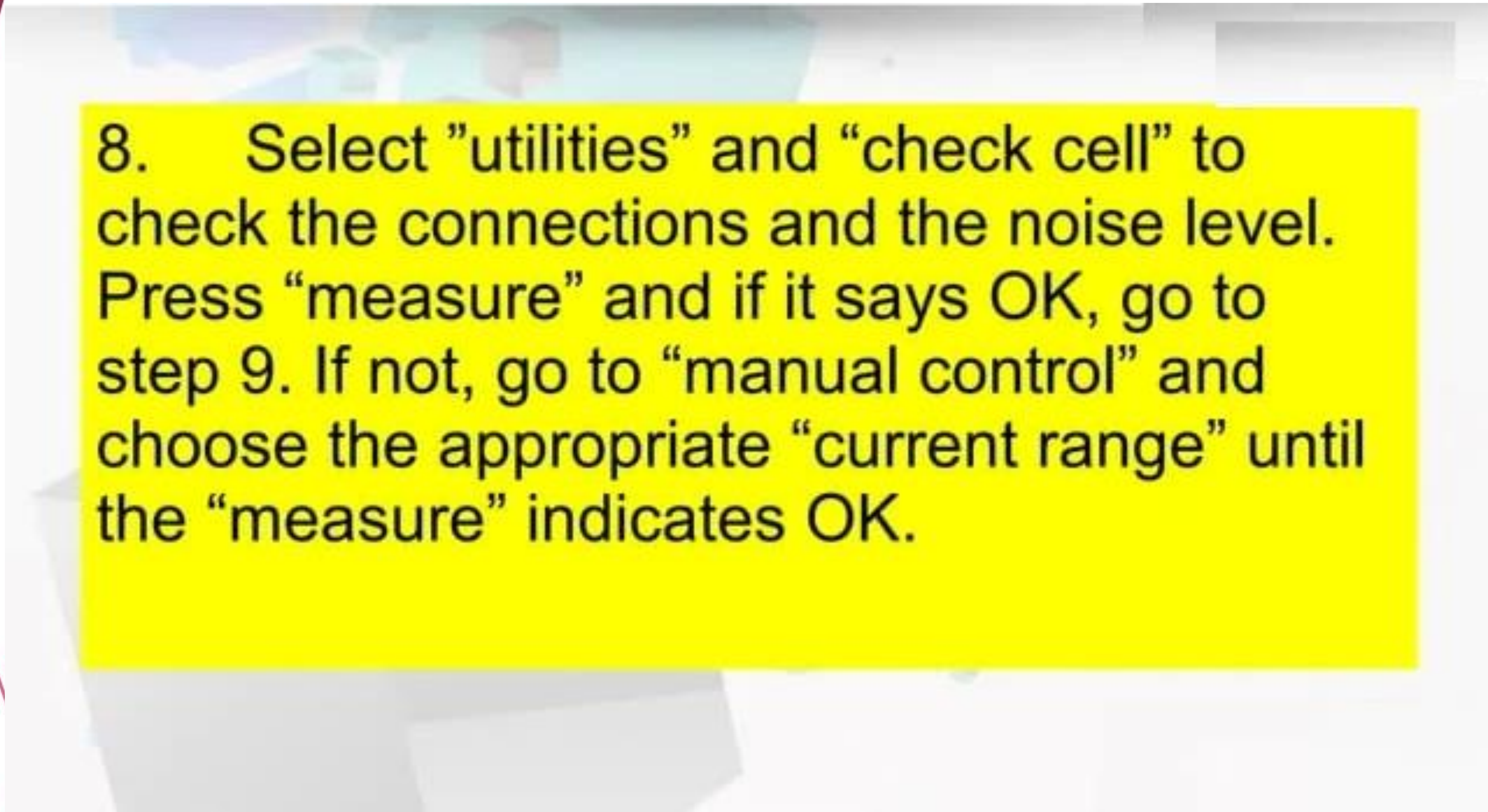
5. Double-click “administrador de programas”, then “Autolab applications” and finally “GPES” (in case you come from step 2, a message will appear and you have to click on “ignorar”). The GPES software consists of three windows (“edit procedure”, “manual control” and “data presentation”) and two bars (“GPES manager” and “status”).



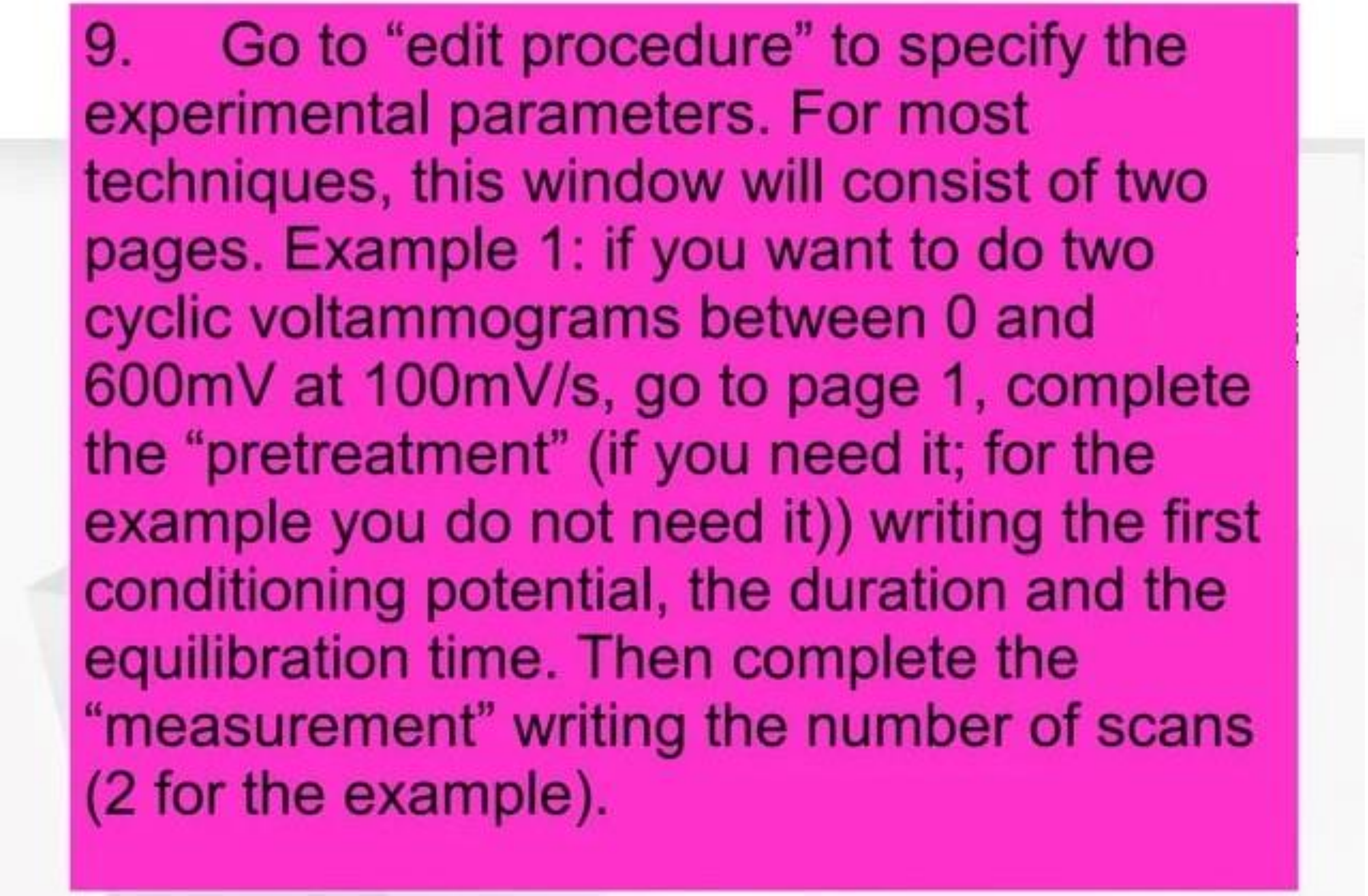
6. Choose the electrochemical method on the main GPES manager bar (“method”). Example 1: to do a cyclic voltammogram (CV), select “cyclic voltammetry (staircase)” and then select “normal”. Example 2: to do a chronoamperometry measurement, select “chrono method (interval time >0.1s)” and then select “amperometry”.



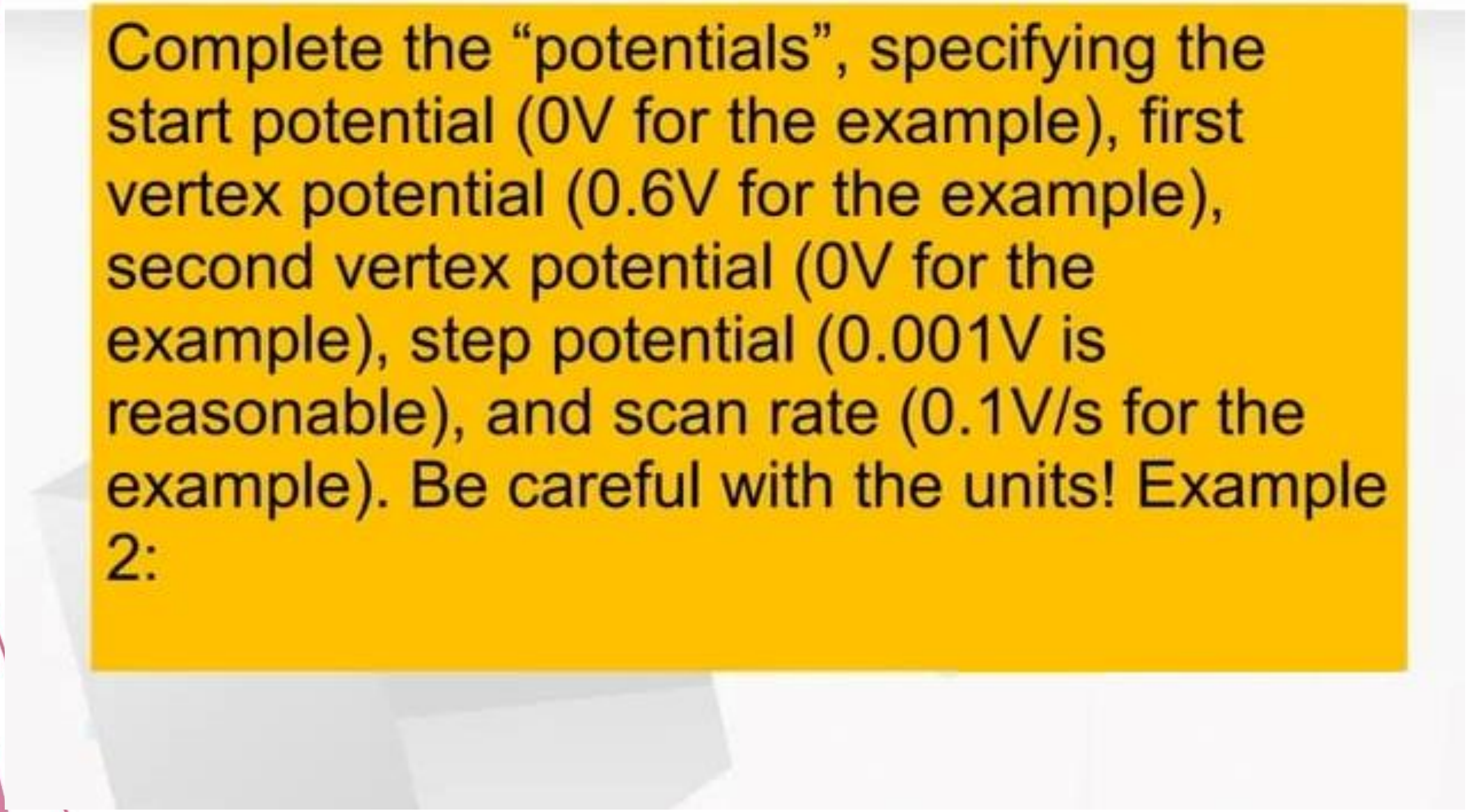
7. Connect the four connectors of the cell cable (connected to the Autolab by means of the DIN connector) to your system, taking into account that the red connector is the working electrode, the blue connector is the reference electrode, the black connector is the auxiliary or counter electrode, and the green connector is the analog ground.



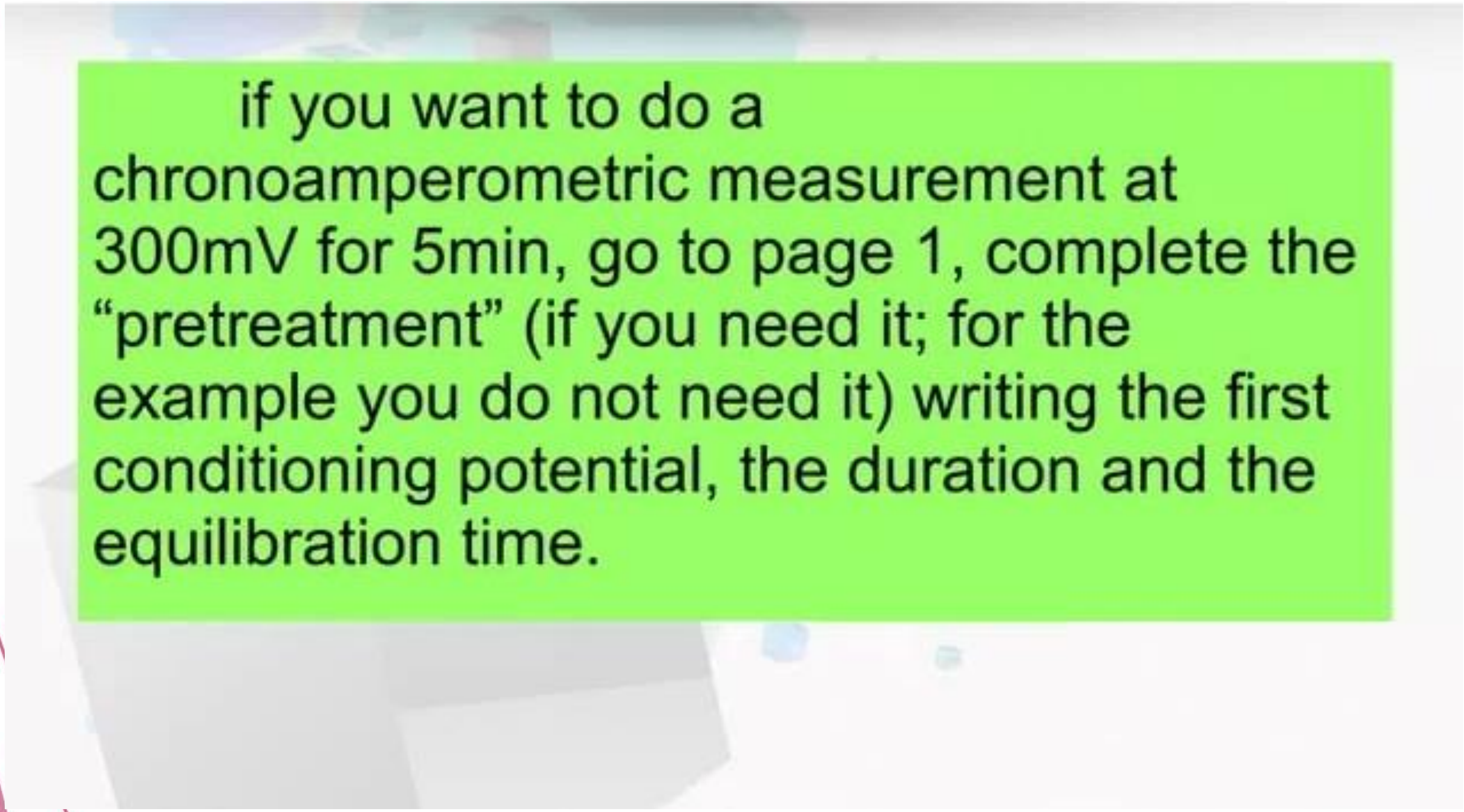
8. Select ”utilities” and “check cell” to check the connections and the noise level. Press “measure” and if it says OK, go to step 9. If not, go to “manual control” and choose the appropriate “current range” until the “measure” indicates OK.



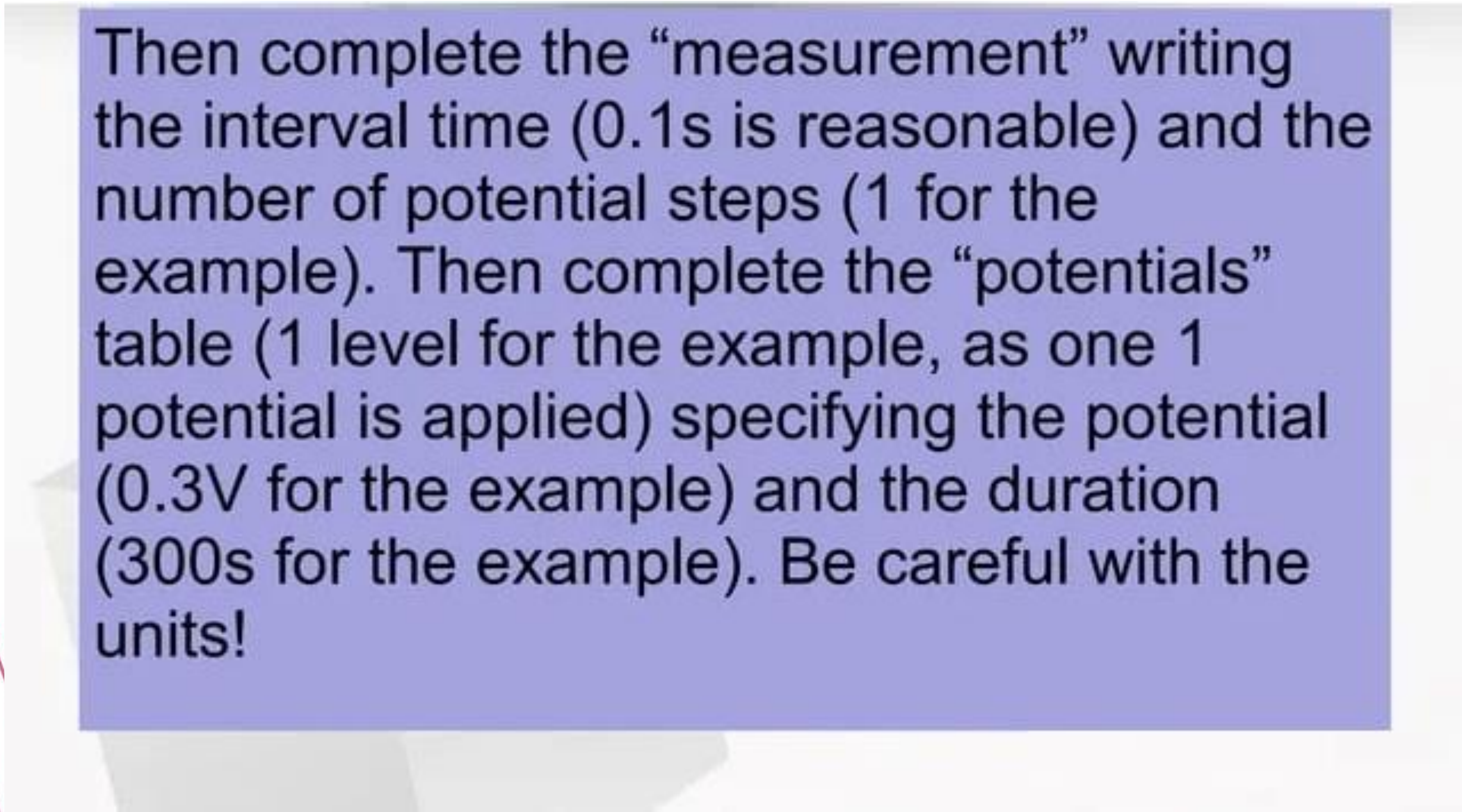
9. Go to “edit procedure” to specify the experimental parameters. For most techniques, this window will consist of two pages. Example 1: if you want to do two cyclic voltammograms between 0 and 600mV at 100mV/s, go to page 1, complete the “pretreatment” (if you need it; for the example you do not need it)) writing the first conditioning potential, the duration and the equilibration time. Then complete the “measurement” writing the number of scans (2 for the example).



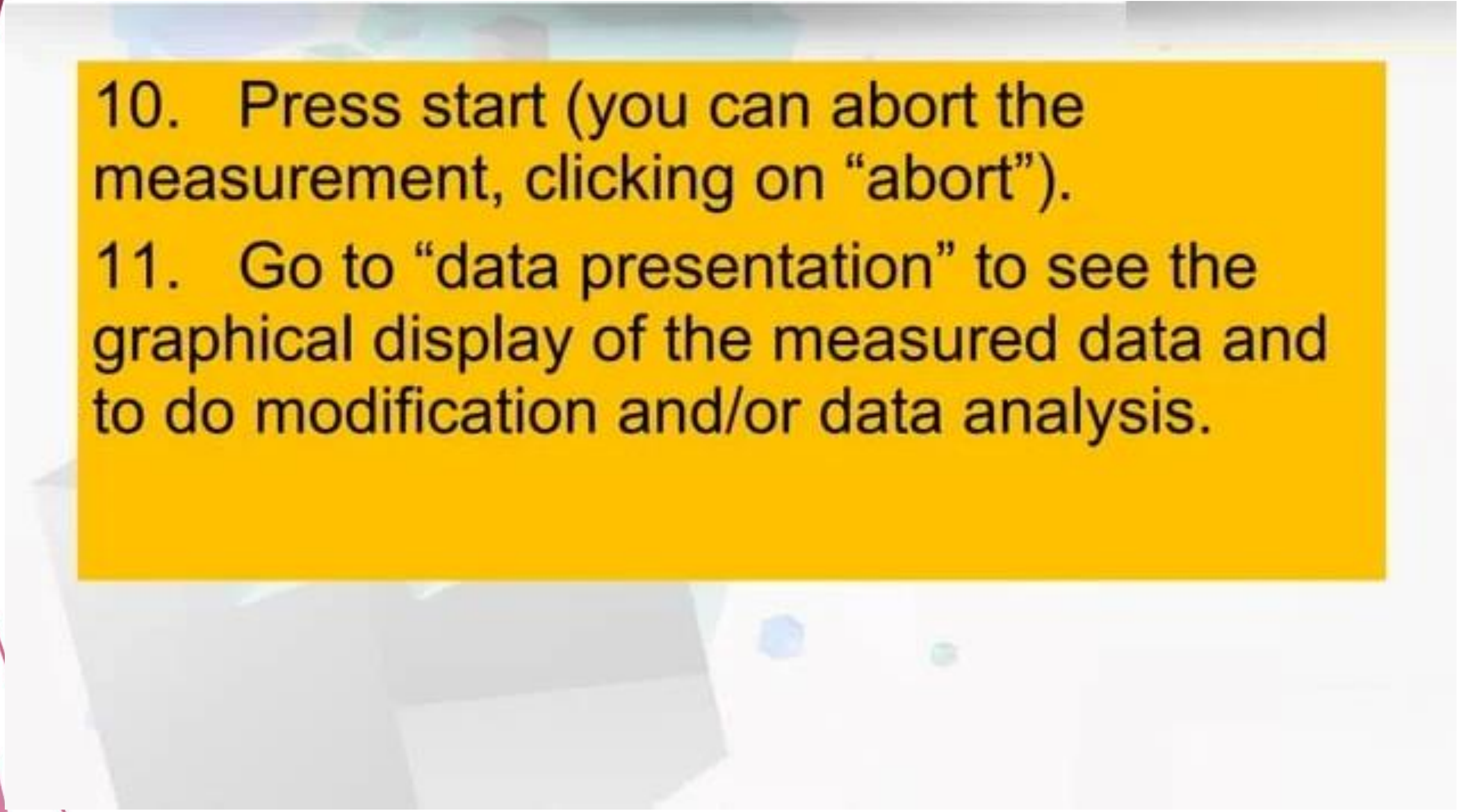
Complete the “potentials”, specifying the start potential (0V for the example), first vertex potential (0.6V for the example), second vertex potential (0V for the example), step potential (0.001V is reasonable), and scan rate (0.1V/s for the example). Be careful with the units! Example 2:



if you want to do a chronoamperometric measurement at 300mV for 5min, go to page 1, complete the “pretreatment” (if you need it; for the example you do not need it) writing the first conditioning potential, the duration and the equilibration time.

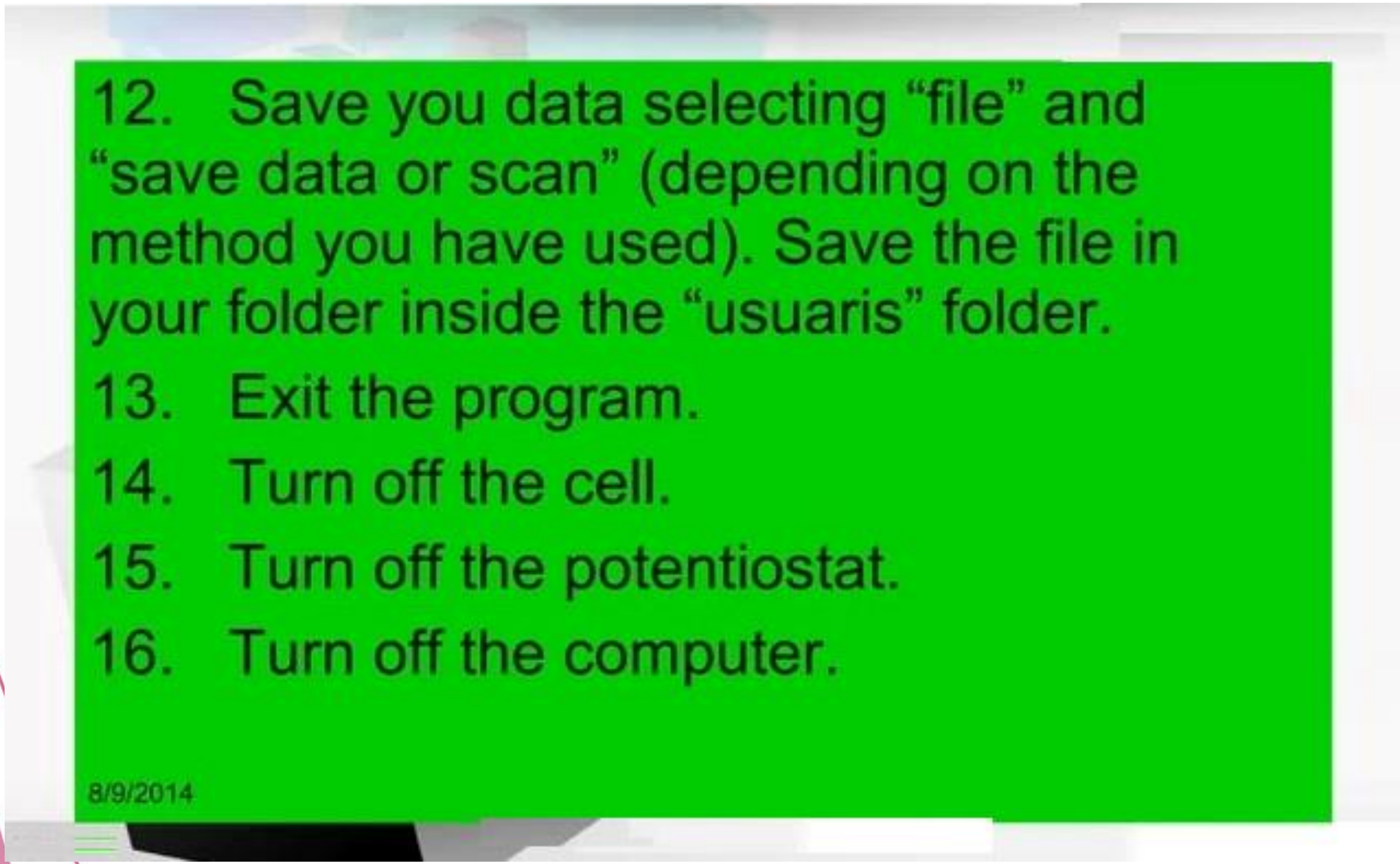


Then complete the “measurement” writing the interval time (0.1s is reasonable) and the number of potential steps (1 for the example). Then complete the “potentials” table (1 level for the example, as one 1 potential is applied) specifying the potential (0.3V for the example) and the duration (300s for the example). Be careful with the units!



10. Press start (you can abort the measurement, clicking on “abort”).

11. Go to “data presentation” to see the graphical display of the measured data and to do modification and/or data analysis.



12. Save you data selecting “file” and “save data or scan” (depending on the method you have used). Save the file in your folder inside the “usuaris” folder.
13. Exit the program.
14. Turn off the cell.
15. Turn off the potentiostat.
16. Turn off the computer.

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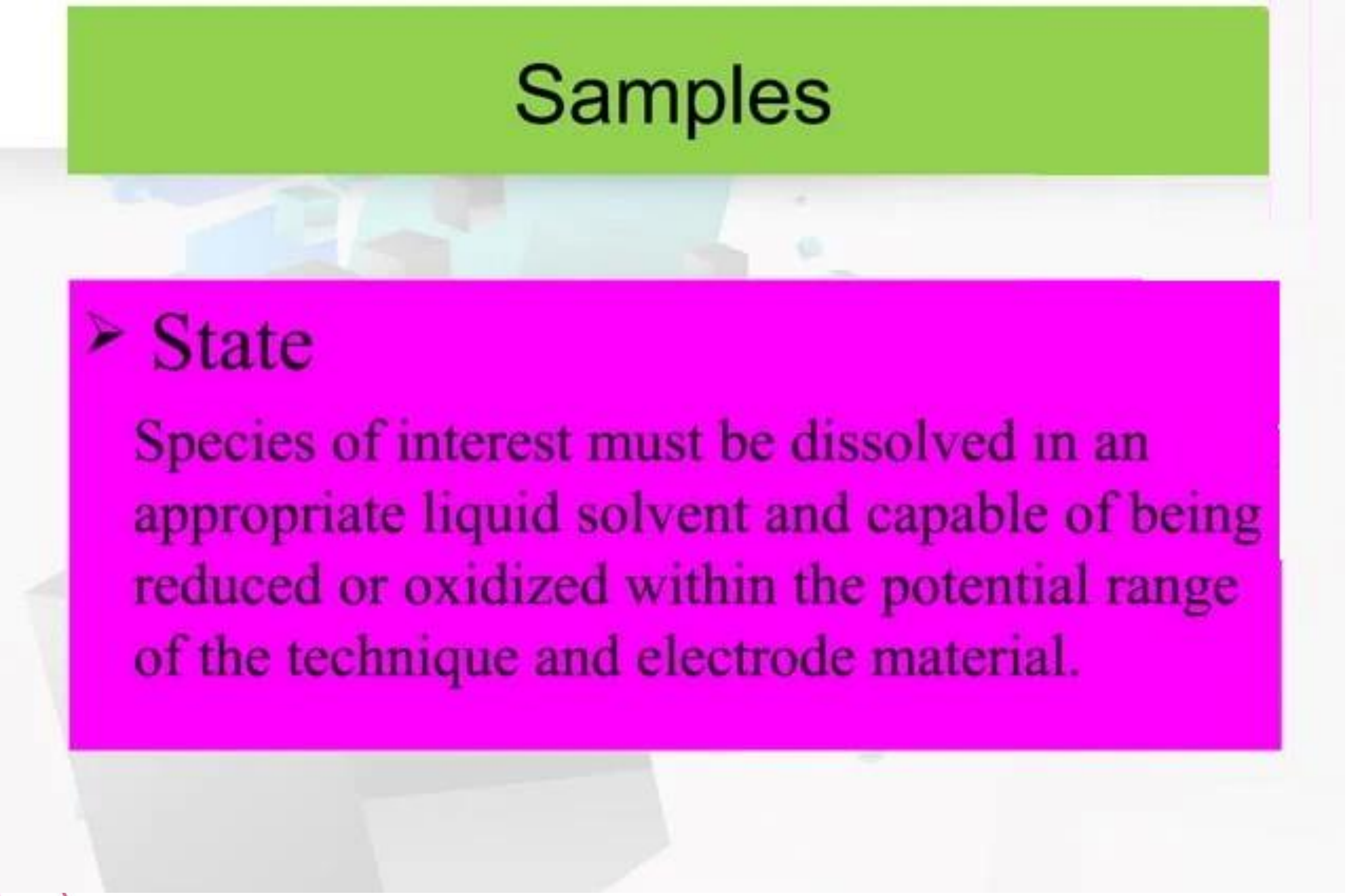
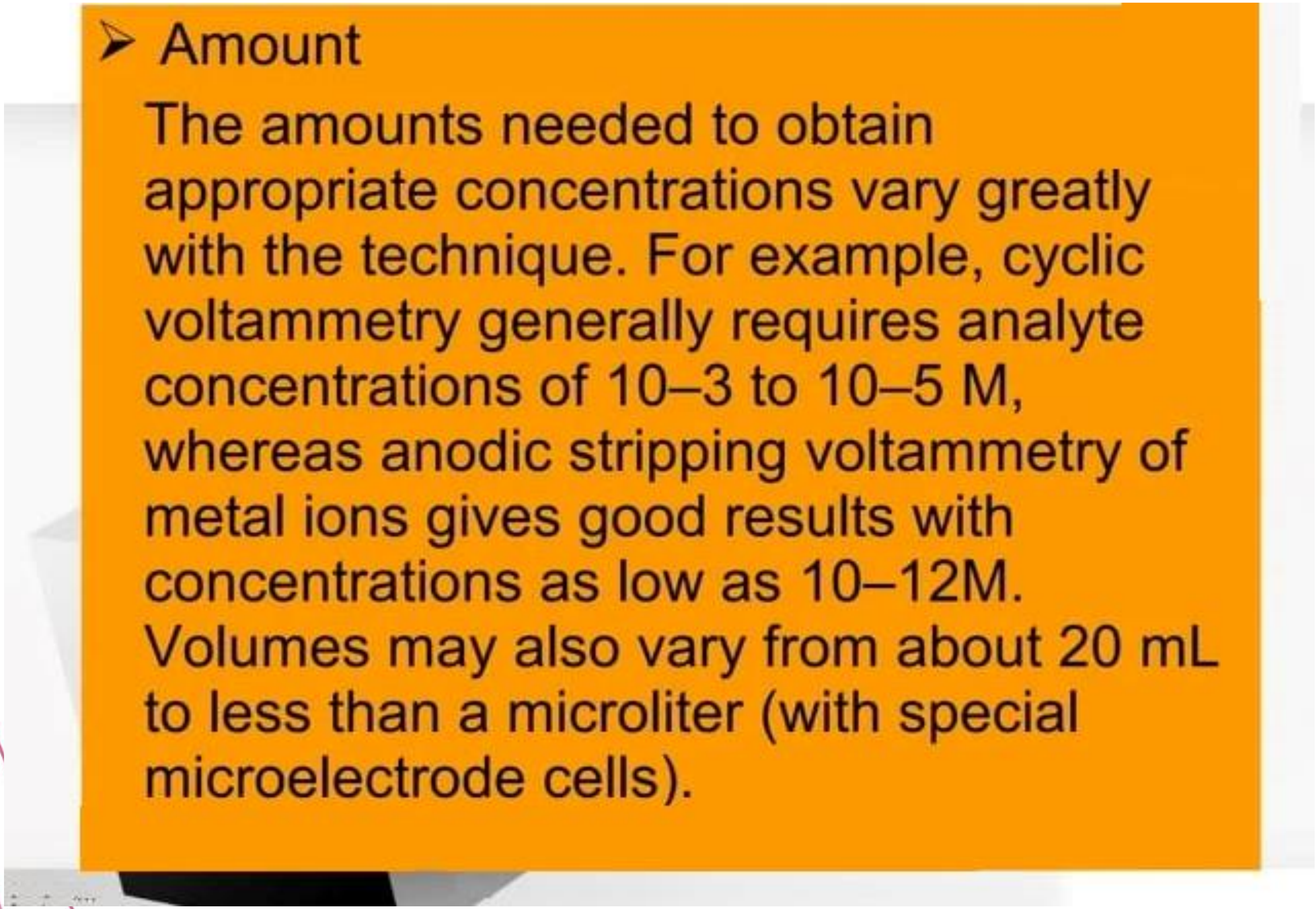


Samples

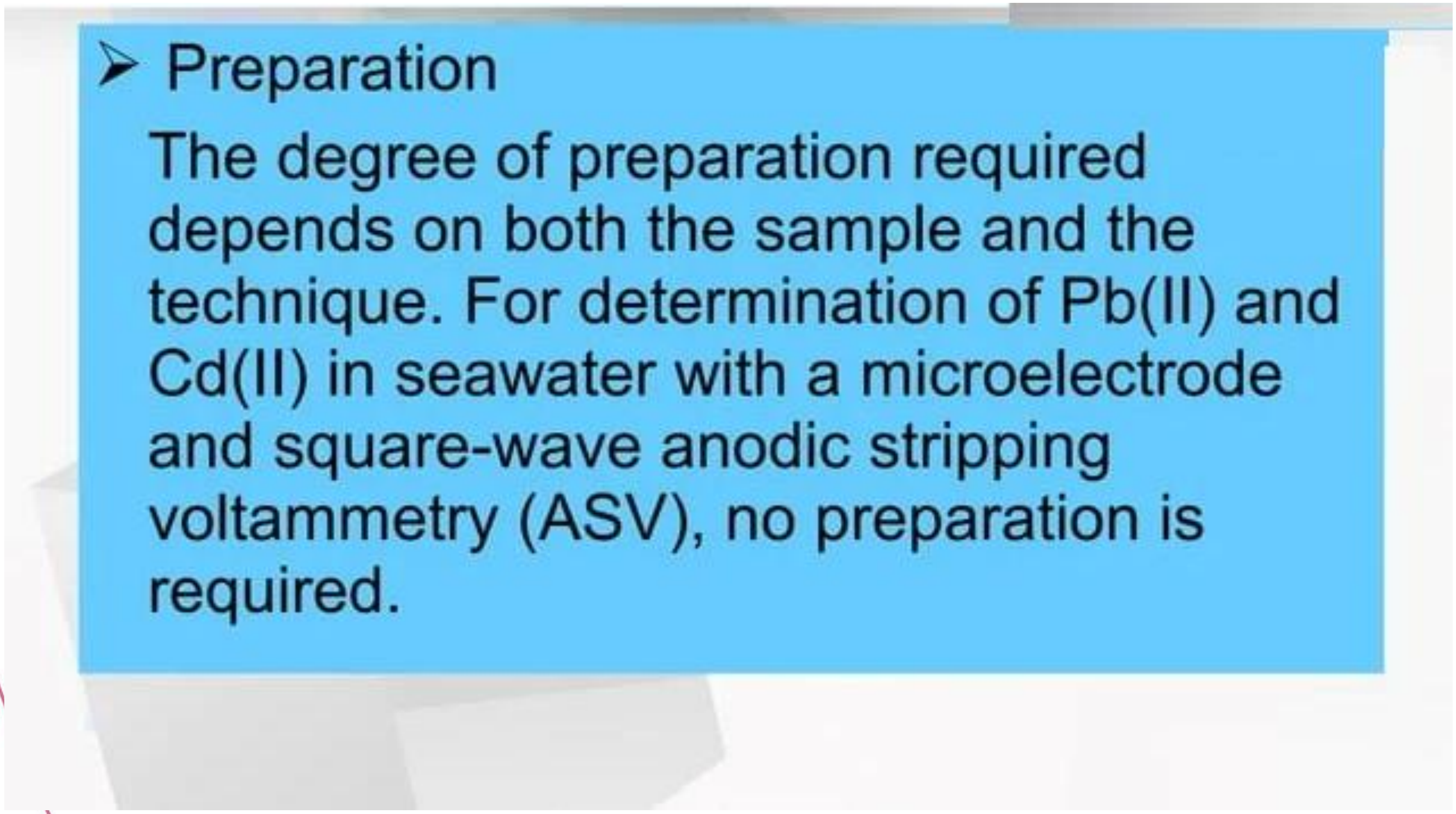
- State

  Species of interest must be dissolved in an appropriate liquid solvent and capable of being reduced or oxidized within the potential range of the technique and electrode material.



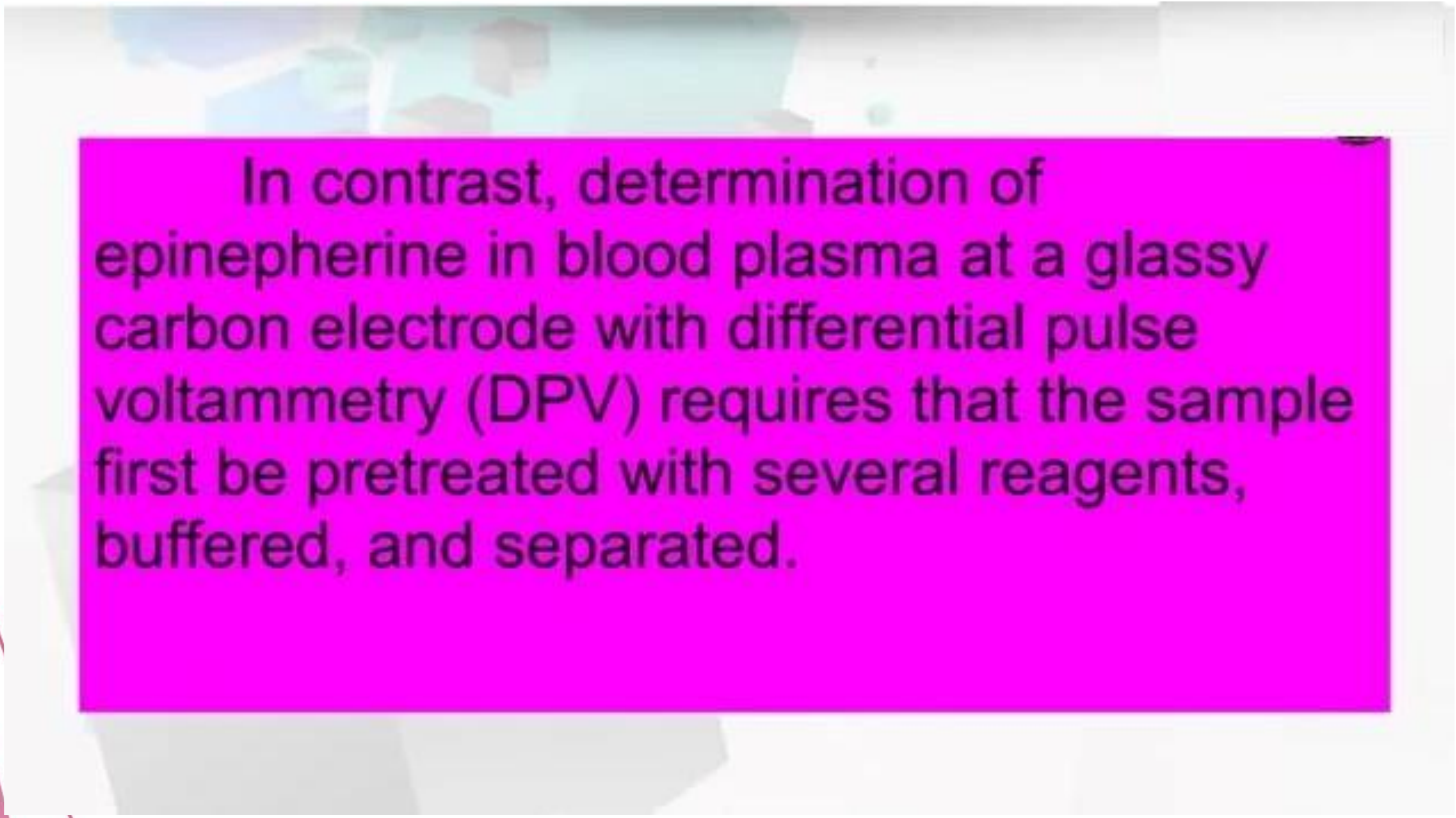
- Amount

The amounts needed to obtain appropriate concentrations vary greatly with the technique. For example, cyclic voltammetry generally requires analyte concentrations of 10–3 to 10–5 M, whereas anodic stripping voltammetry of metal ions gives good results with concentrations as low as 10–12M. Volumes may also vary from about 20 mL to less than a microliter (with special microelectrode cells).

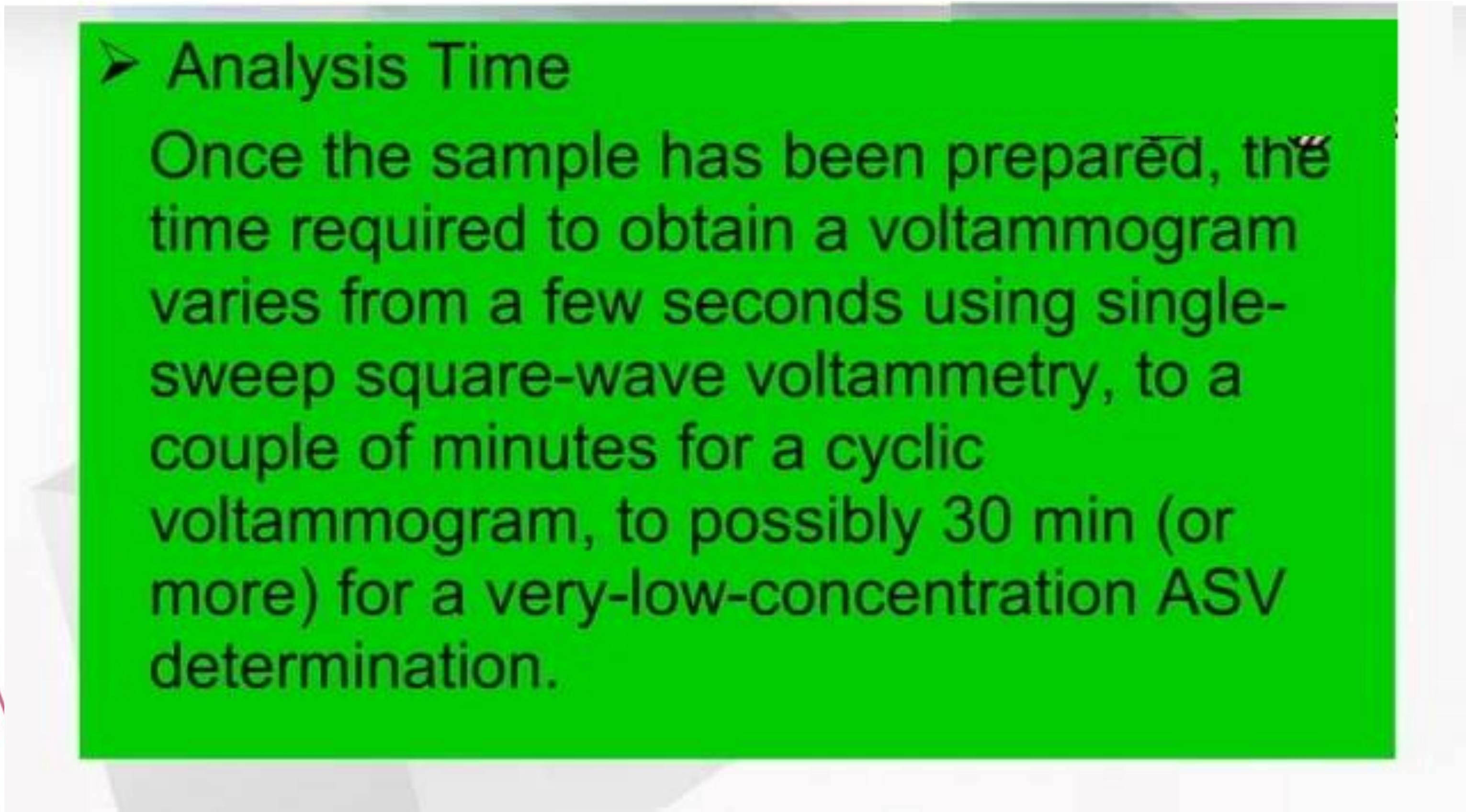


- Preparation

  The degree of preparation required depends on both the sample and the technique. For determination of Pb(II) and Cd(II) in seawater with a microelectrode and square-wave anodic stripping voltammetry (ASV), no preparation is required.

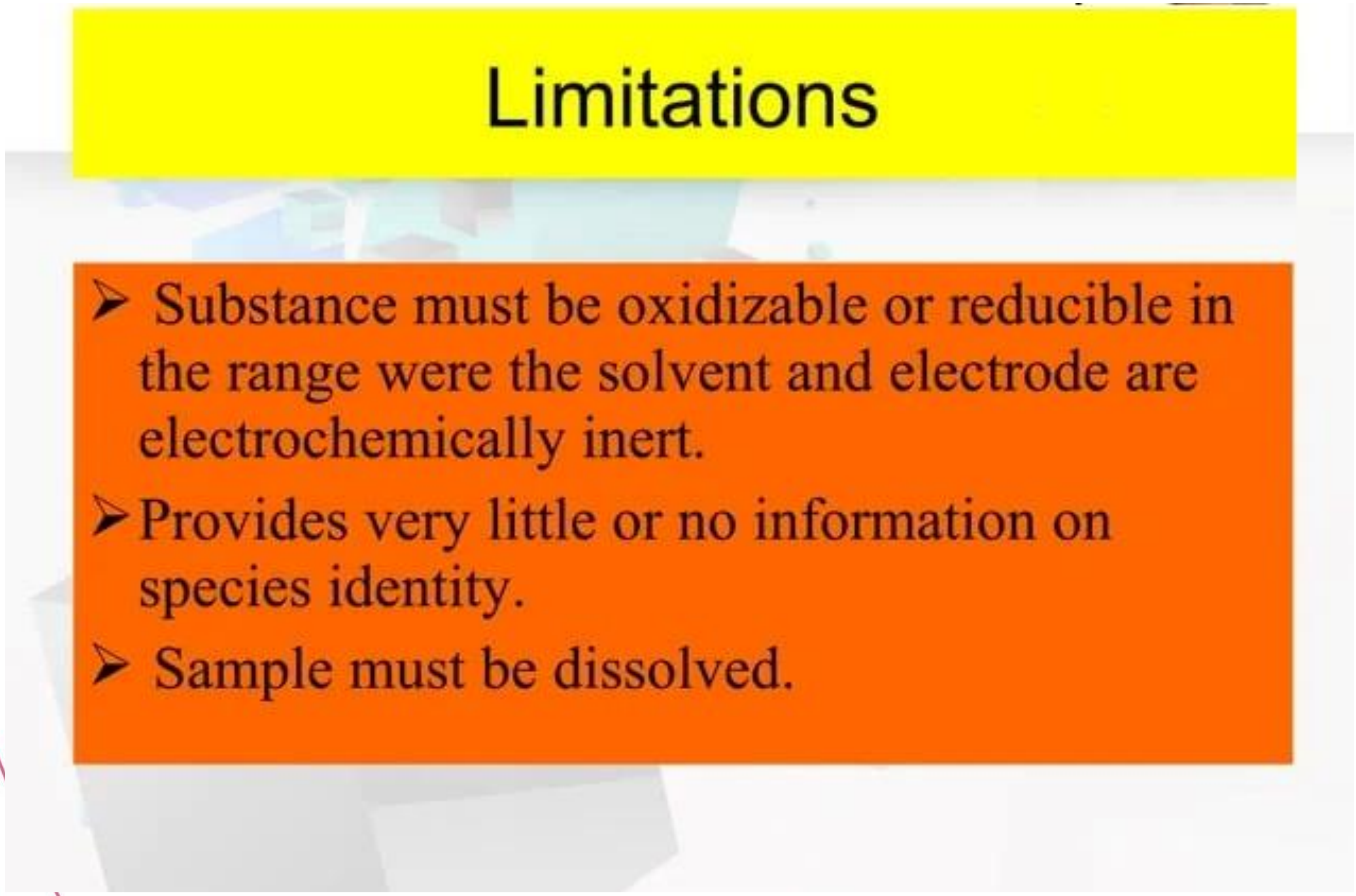


In contrast, determination of epinepherine in blood plasma at a glassy carbon electrode with differential pulse voltammetry (DPV) requires that the sample first be pretreated with several reagents, buffered, and separated.



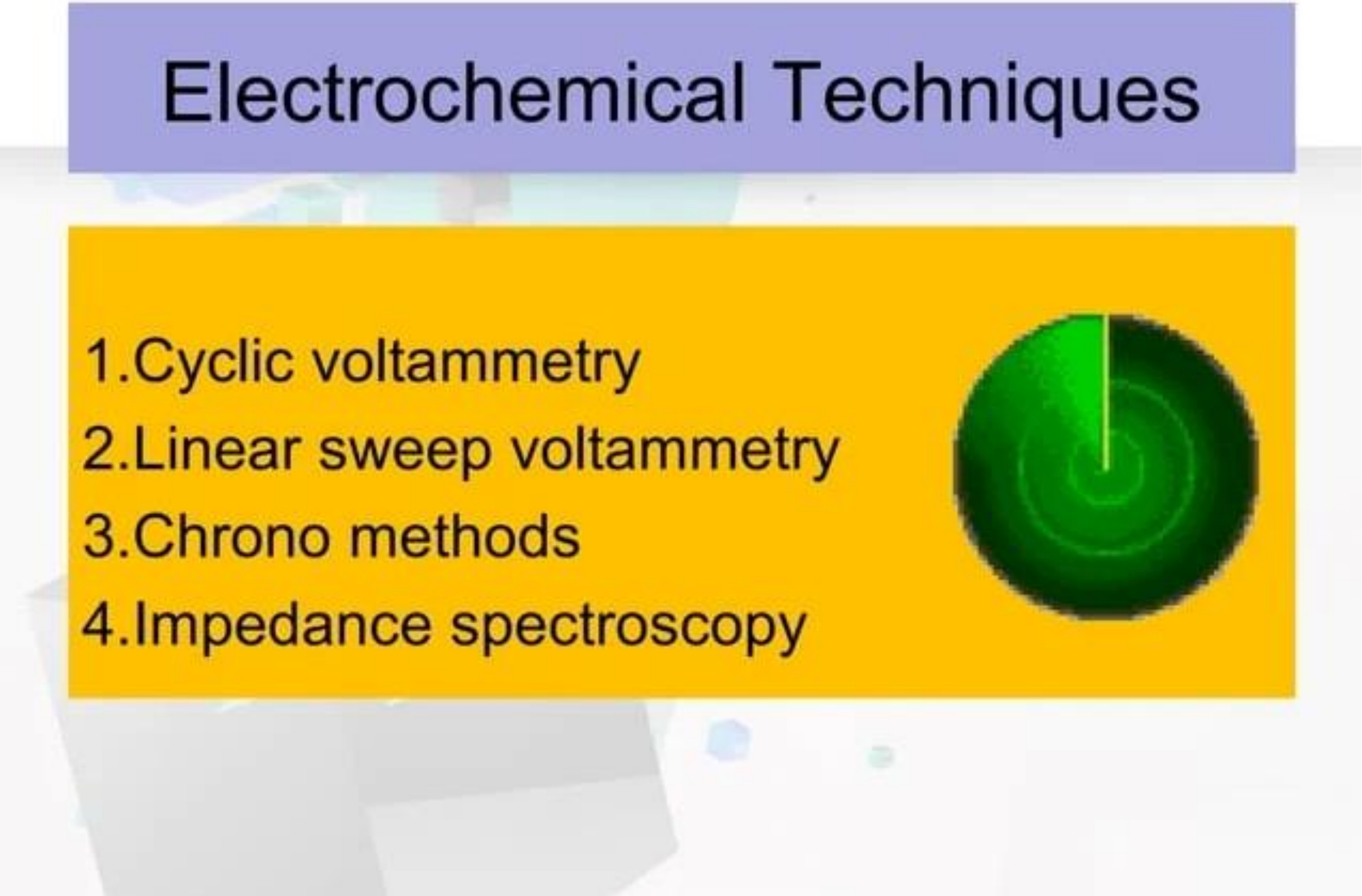
- Analysis Time

Once the sample has been prepared, the time required to obtain a voltammogram varies from a few seconds using single-sweep square-wave voltammetry, to a couple of minutes for a cyclic voltammogram, to possibly 30 min (or more) for a very-low-concentration ASV determination.



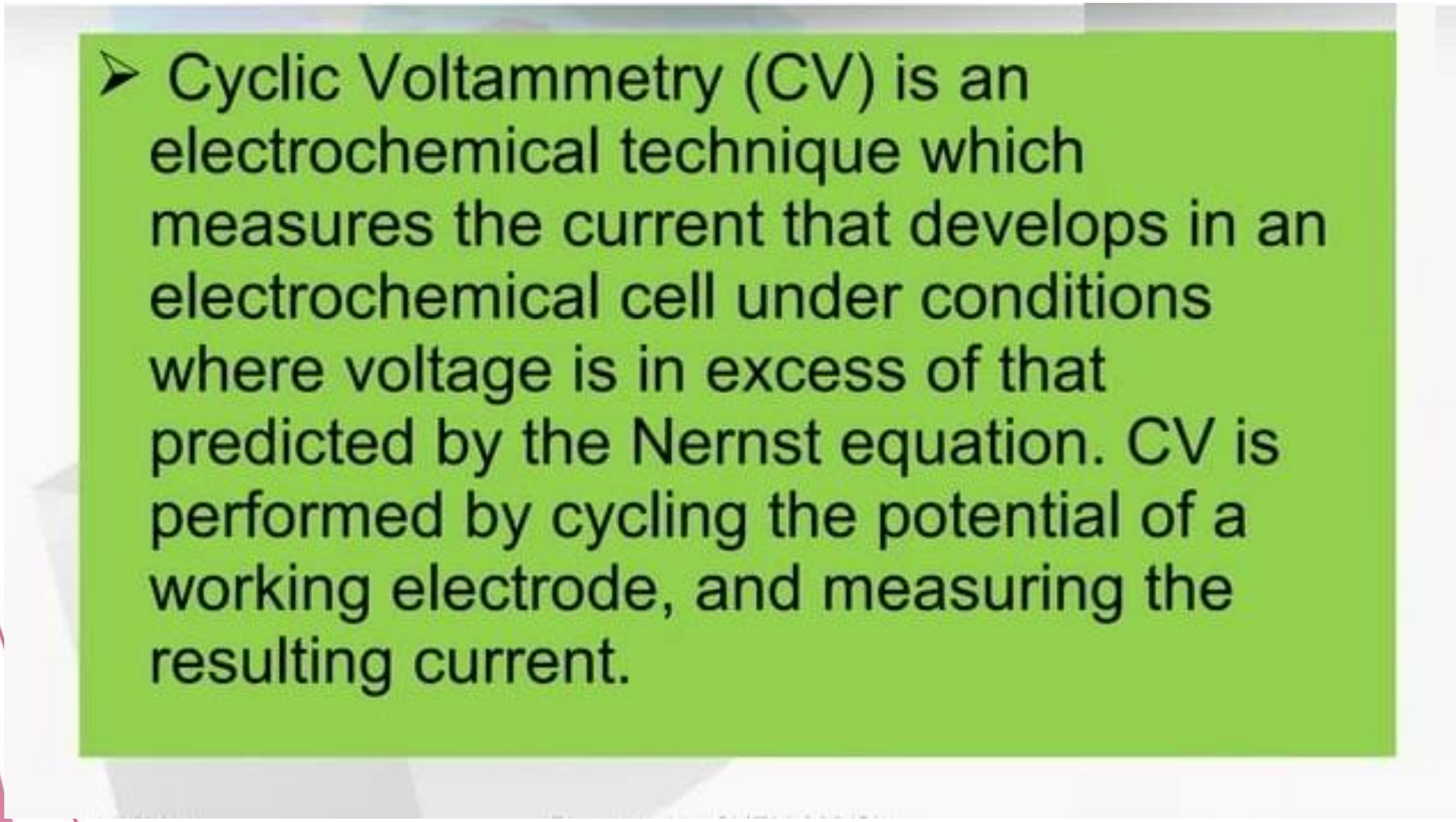
Limitations

- Substance must be oxidizable or reducible in the range were the solvent and electrode are electrochemically inert.
- Provides very little or no information on species identity.
- Sample must be dissolved.

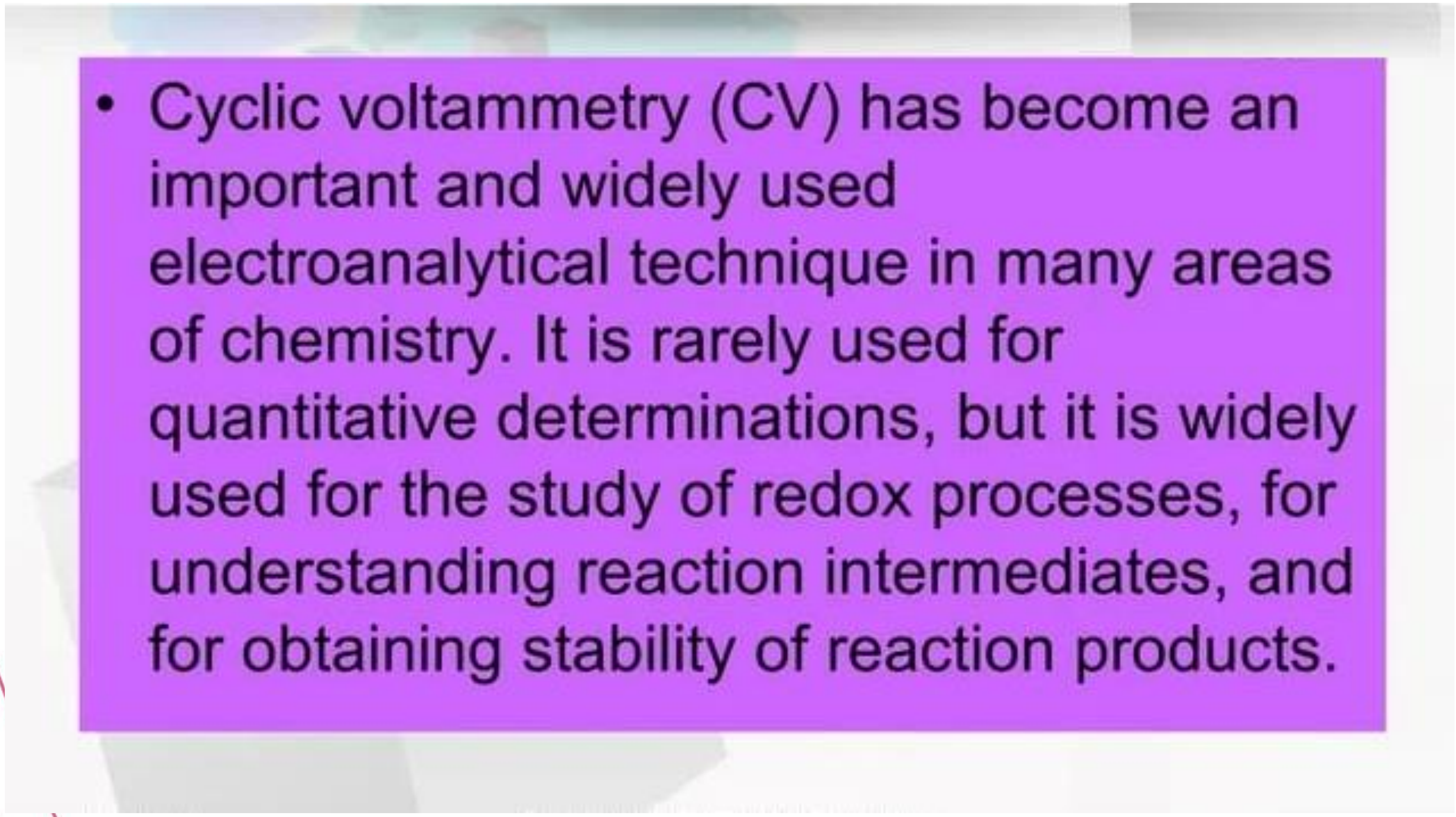


Electrochemical Techniques

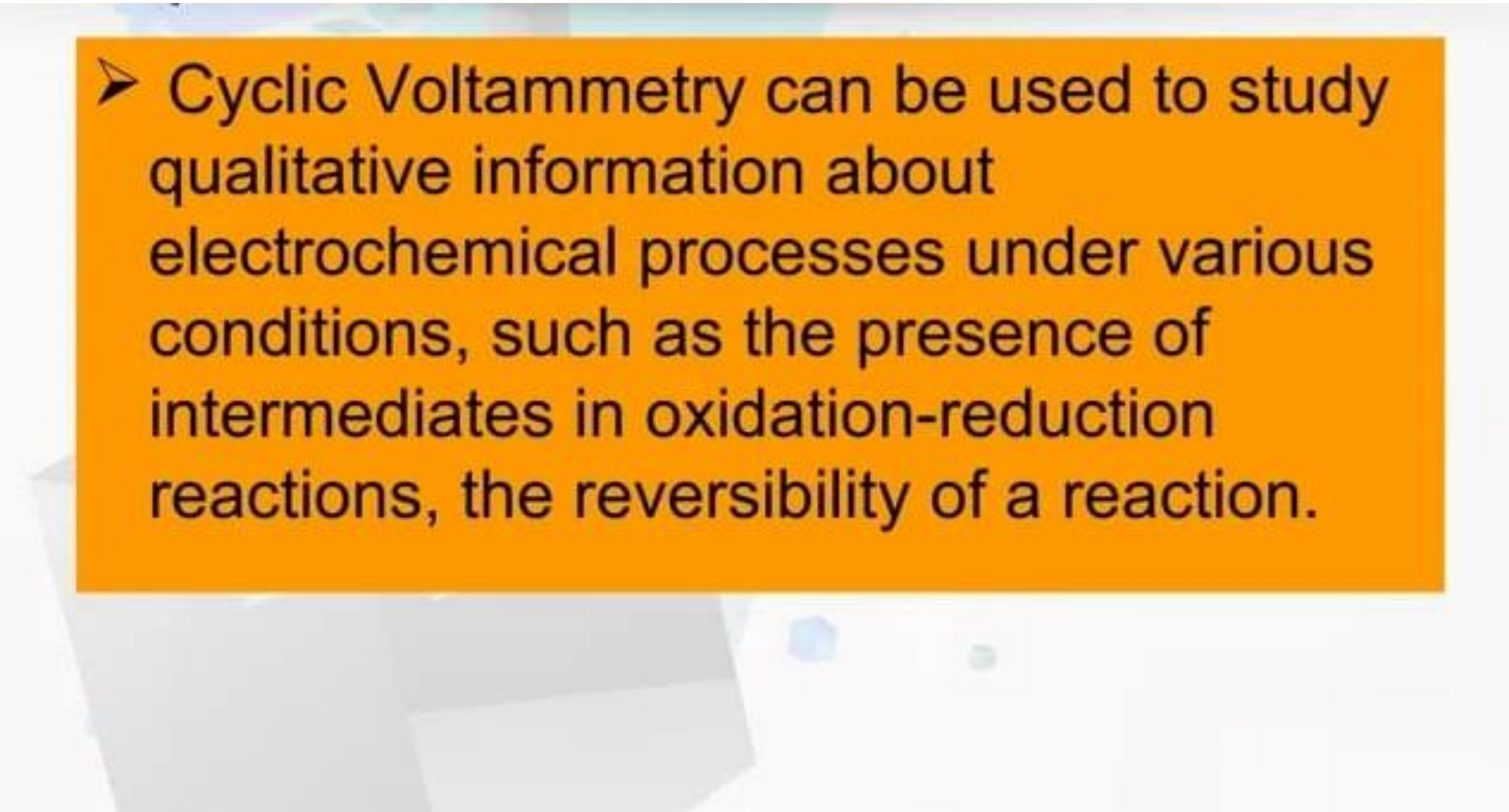
1. Cyclic voltammetry
2. Linear sweep voltammetry
3. Chrono methods
4. Impedance spectroscopy



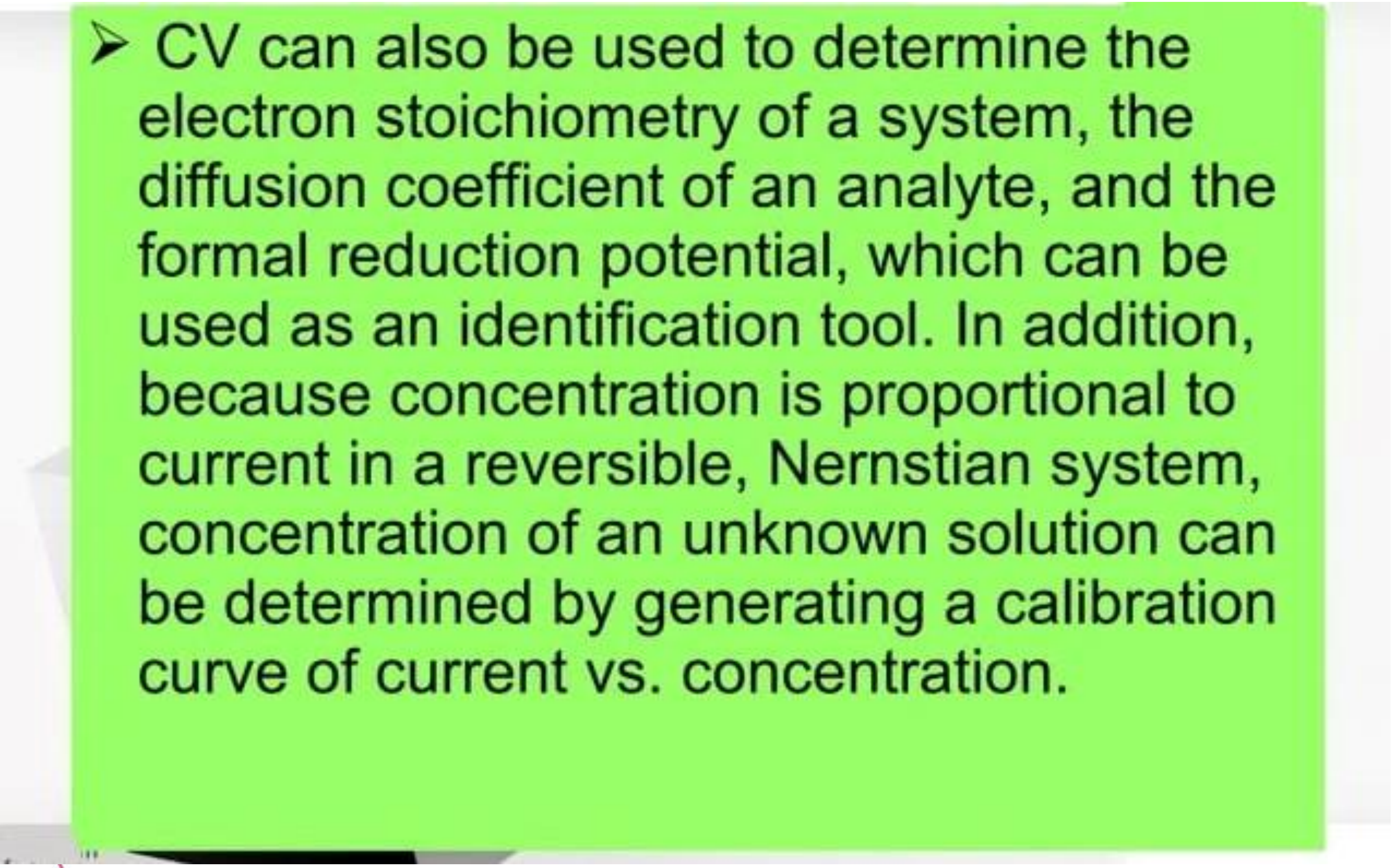
- Cyclic Voltammetry (CV) is an electrochemical technique which measures the current that develops in an electrochemical cell under conditions where voltage is in excess of that predicted by the Nernst equation. CV is performed by cycling the potential of a working electrode, and measuring the resulting current.



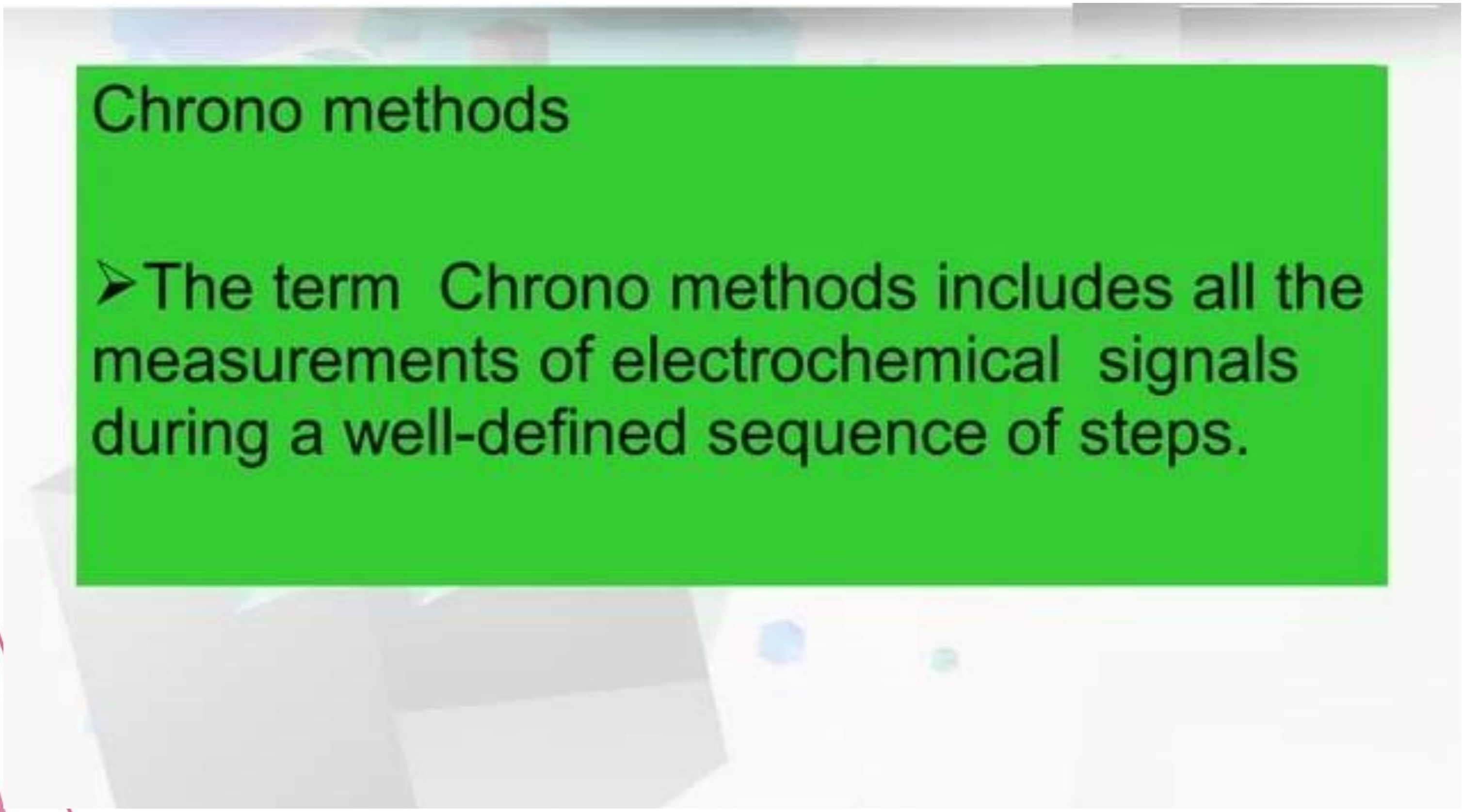
- Cyclic voltammetry (CV) has become an important and widely used electroanalytical technique in many areas of chemistry. It is rarely used for quantitative determinations, but it is widely used for the study of redox processes, for understanding reaction intermediates, and for obtaining stability of reaction products.



- Cyclic Voltammetry can be used to study qualitative information about electrochemical processes under various conditions, such as the presence of intermediates in oxidation-reduction reactions, the reversibility of a reaction.

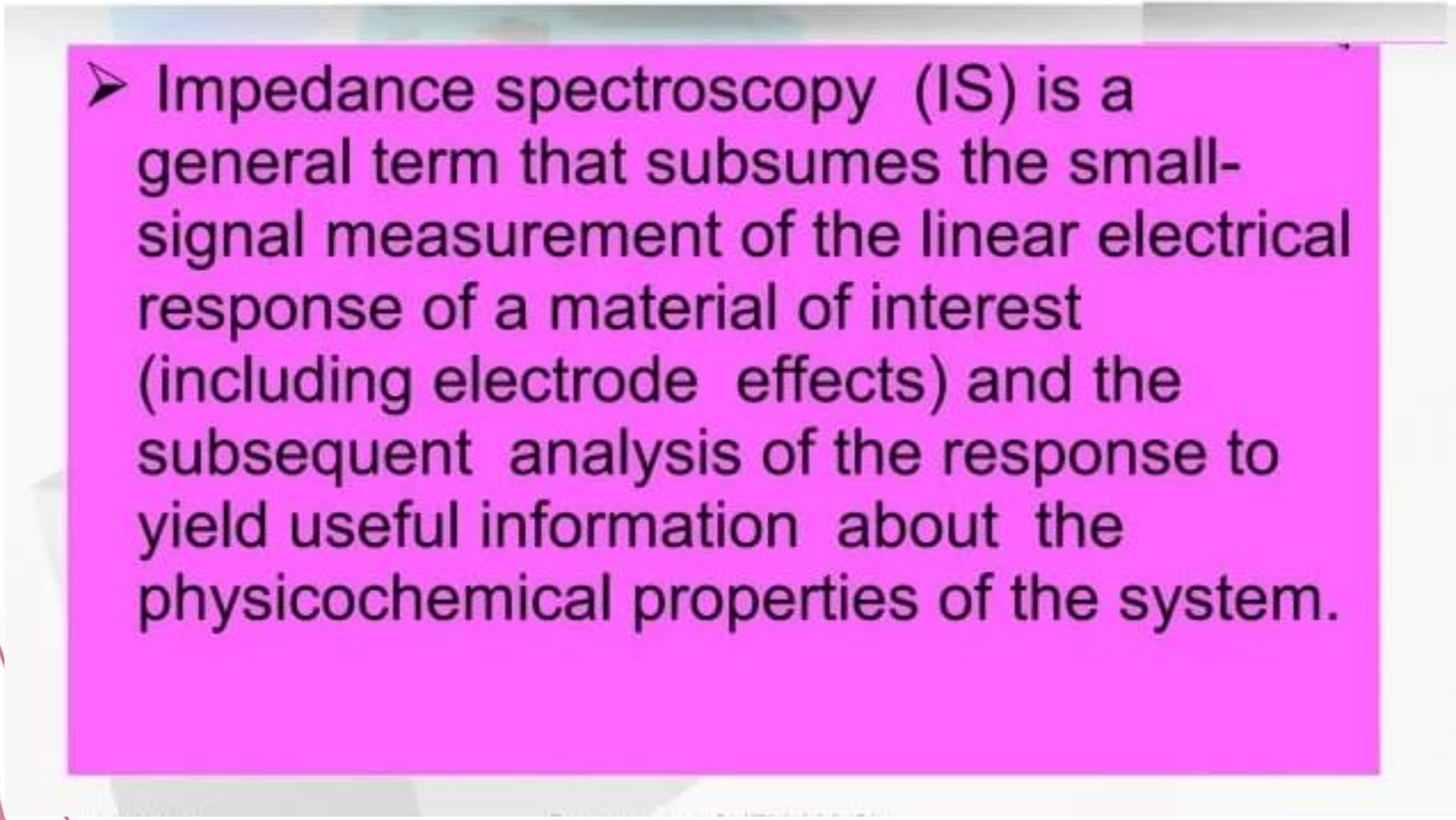


- CV can also be used to determine the electron stoichiometry of a system, the diffusion coefficient of an analyte, and the formal reduction potential, which can be used as an identification tool. In addition, because concentration is proportional to current in a reversible, Nernstian system, concentration of an unknown solution can be determined by generating a calibration curve of current vs. concentration.

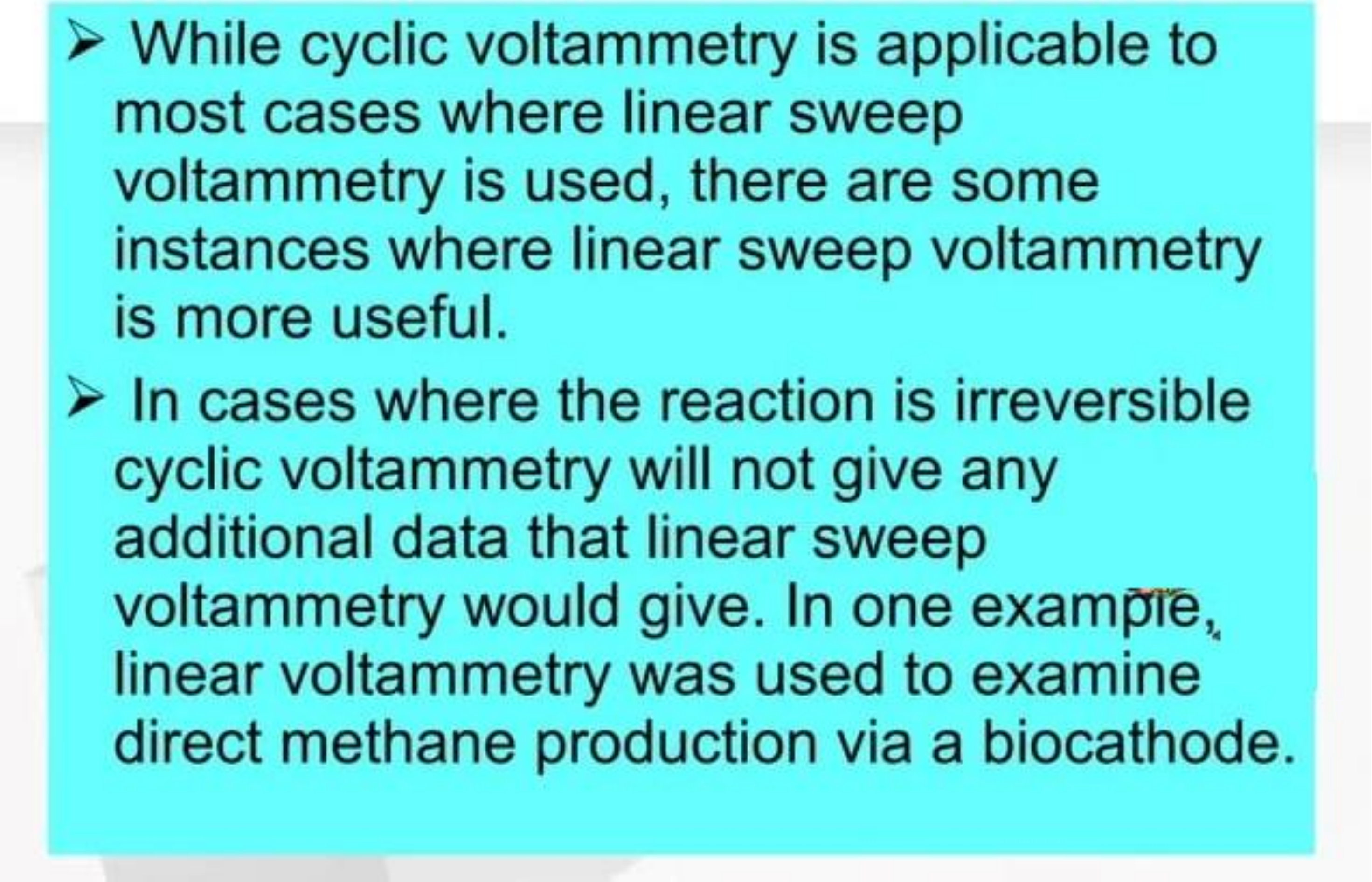


Chrono methods

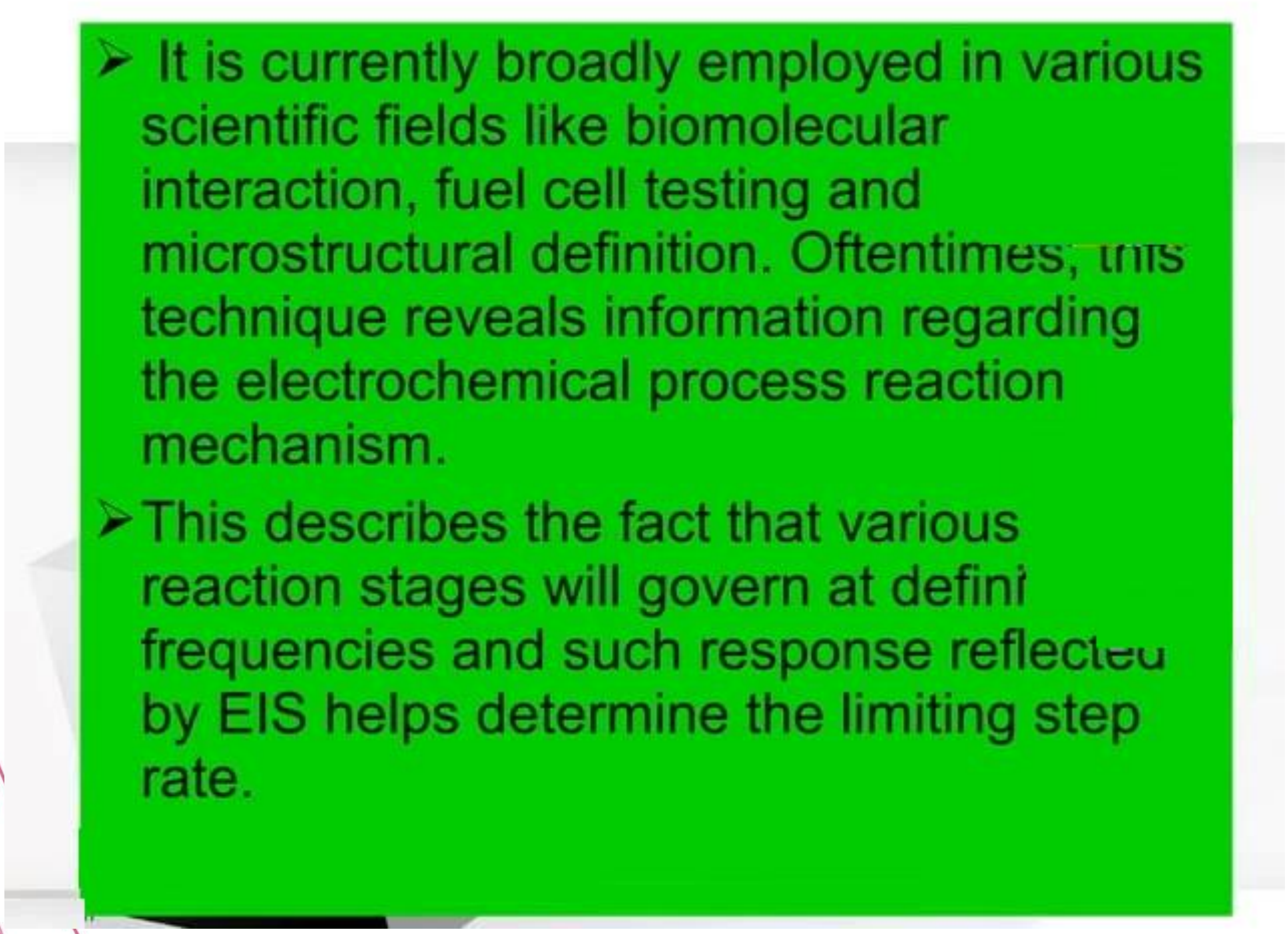
- The term Chrono methods includes all the measurements of electrochemical signals during a well-defined sequence of steps.



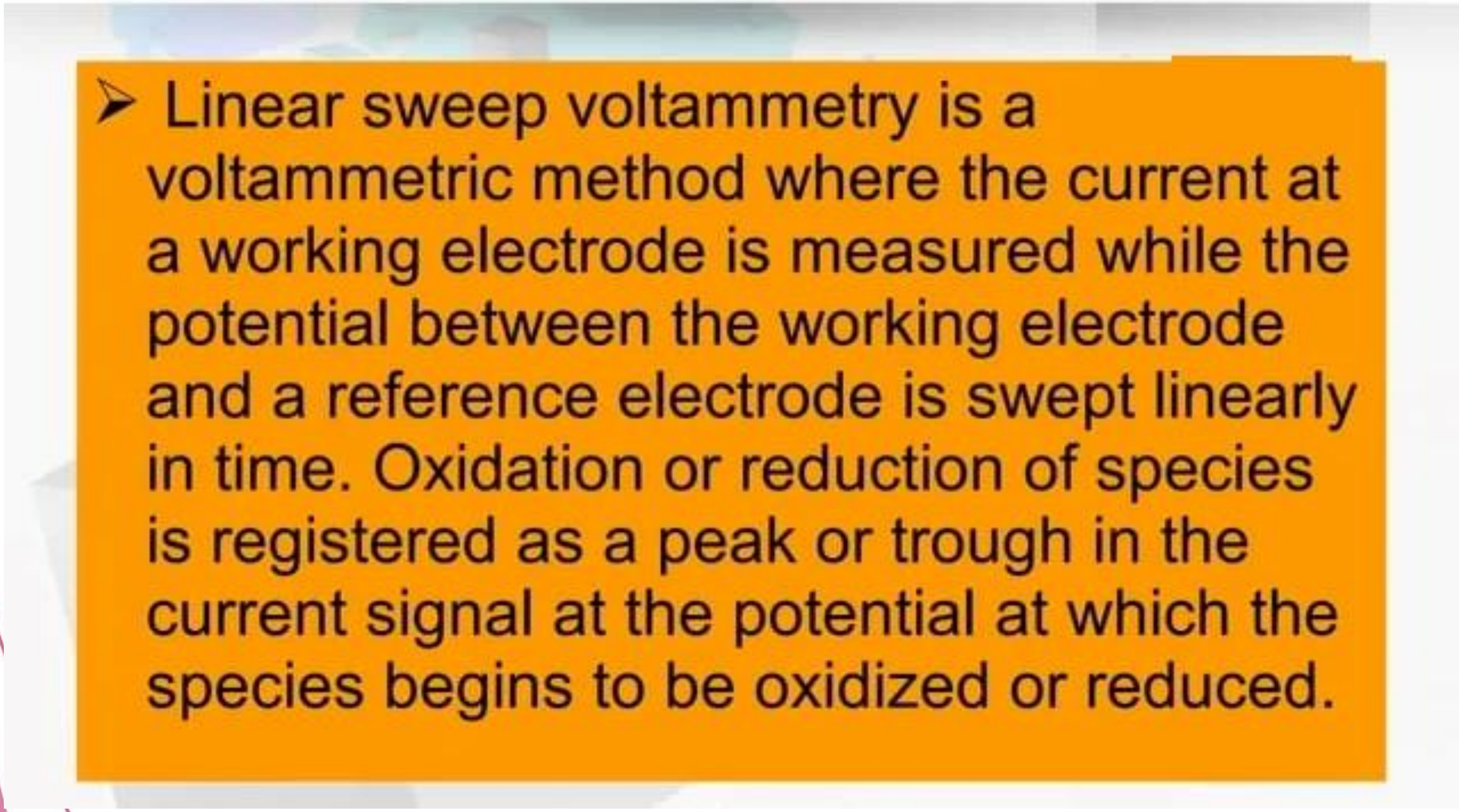
- Impedance spectroscopy (IS) is a general term that subsumes the small-signal measurement of the linear electrical response of a material of interest (including electrode effects) and the subsequent analysis of the response to yield useful information about the physicochemical properties of the system.



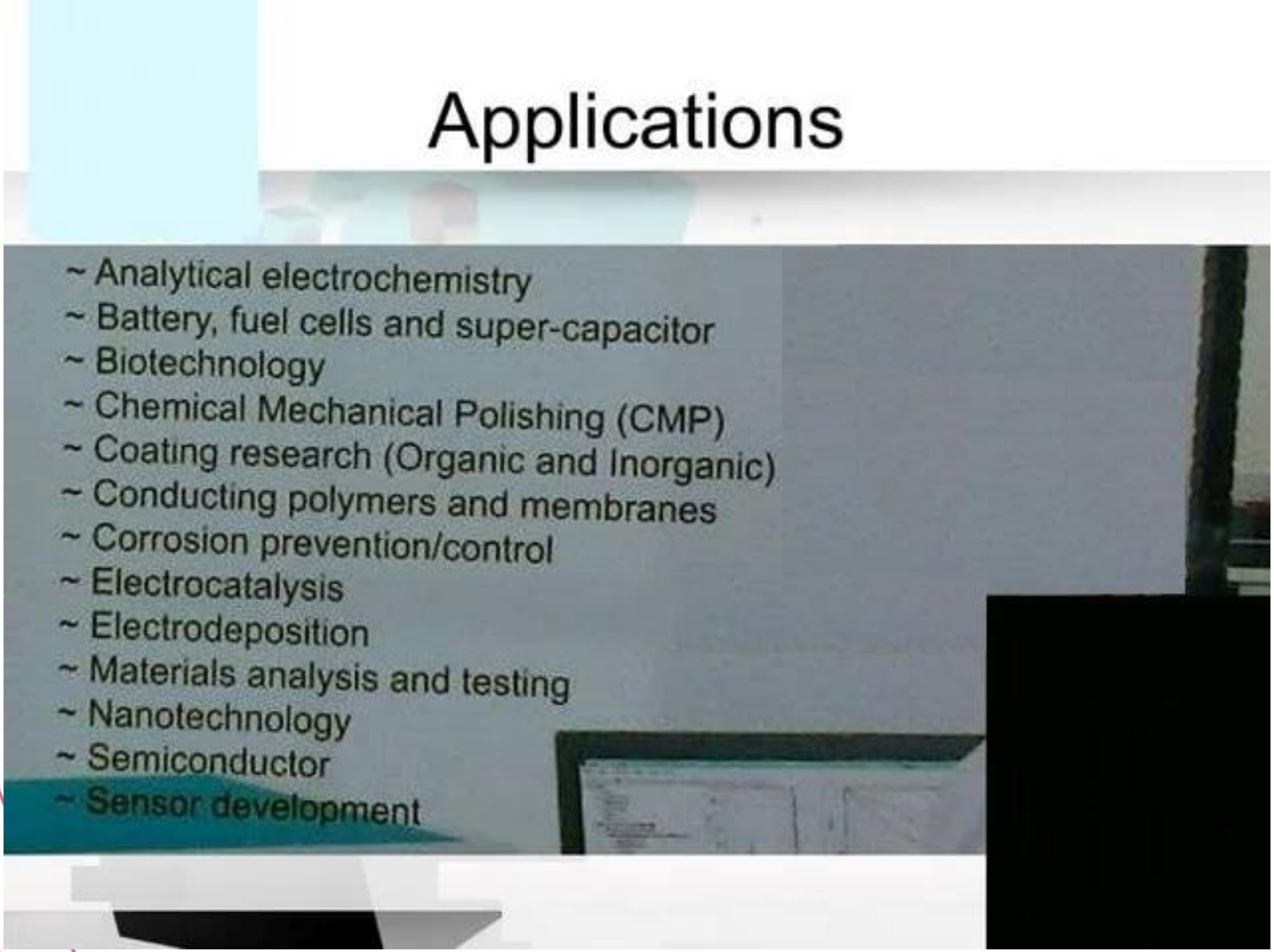
- While cyclic voltammetry is applicable to most cases where linear sweep voltammetry is used, there are some instances where linear sweep voltammetry is more useful.
- In cases where the reaction is irreversible cyclic voltammetry will not give any additional data that linear sweep voltammetry would give. In one example, linear voltammetry was used to examine direct methane production via a biocathode.



- It is currently broadly employed in various scientific fields like biomolecular interaction, fuel cell testing and microstructural definition. Oftentimes, this technique reveals information regarding the electrochemical process reaction mechanism.
- This describes the fact that various reaction stages will govern at defini frequencies and such response reflected by EIS helps determine the limiting step rate.



- Linear sweep voltammetry is a voltammetric method where the current at a working electrode is measured while the potential between the working electrode and a reference electrode is swept linearly in time. Oxidation or reduction of species is registered as a peak or trough in the current signal at the potential at which the species begins to be oxidized or reduced.



Applications

- ~ Analytical electrochemistry
- ~ Battery, fuel cells and super-capacitor
- ~ Biotechnology
- ~ Chemical Mechanical Polishing (CMP)
- ~ Coating research (Organic and Inorganic)
- ~ Conducting polymers and membranes
- ~ Corrosion prevention/control
- ~ Electrocatalysis
- ~ Electrodeposition
- ~ Materials analysis and testing
- ~ Nanotechnology
- ~ Semiconductor
- ~ Sensor development

THANK YOU

FOR

YOUR ATTENTION

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