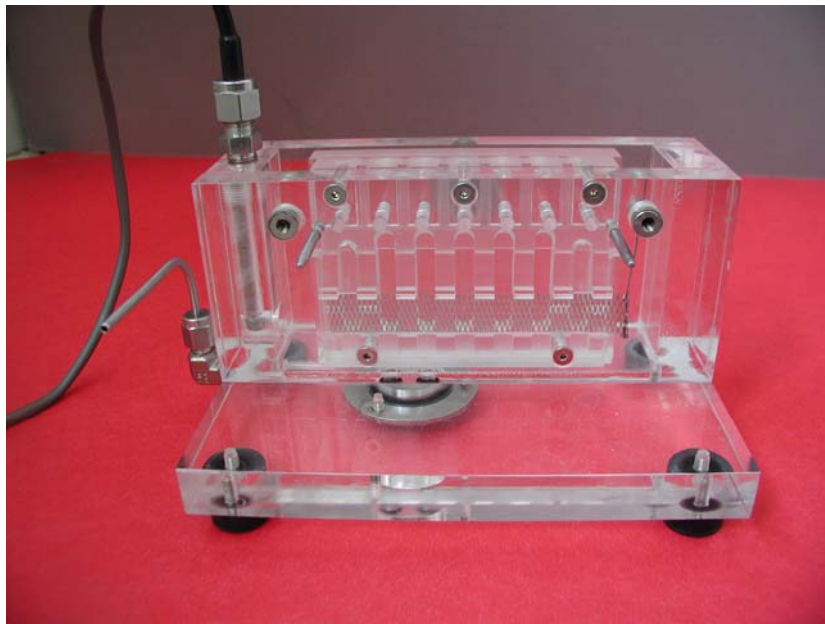


# *L-Cell*®

## *User's Manual*



**Automated Plating Process Diagnostics**

**Patent Pending**



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Patent applications for the L-Cell are pending.

## L-Cell applications -

- **Computer-based quantitative process diagnostics**
  - o Acceptable plating range
  - o Quantitative electrolyte aging/contamination indication
  - o Small-volume testing (50 ml sample) of electrolyte for bath maintenance and composition adjustment
  
- **Process parameters:**
  - o Polarization curves
  - o Electrode Kinetics parameters
  - o Electrolyte conductivity
  - o Throwing power as a function of the current density
  - o Current efficiency as a function of the current density (after separate measurements of the deposit thickness)
  
- **Deposit samples across a current density range**
  - o Template with pads are plated at different, pre-determined current densities, for visual inspection and compositional analysis
  
- **Fully automated rapid experiment (Less than 3 min.)**
  - o Automatic and comprehensive collection and storage of all test data
  - o On-screen, graphical comparison with previous tests

## Advantages of the L-Cell –

- Quantitative analysis replacing guesswork and estimates
- Completely automated – designed for rapid, effortless analysis by non-experts
- Relatively inexpensive
- Provides simultaneously samples plated at different, precisely measured, current densities *and* quantitative electrolyte performance analysis

*The L-Cell provides the 21<sup>st</sup> century replacement for the Hull-Cell*

## The complete, ready-to use package, contains –

1. L-Cell →



2. Electronics control box including power supply and data acquisition card

3. Temperature probe



4. All cables

- Multi-pin signal connector (connecting the electronics control box to the L-Cell)
- Serial cable (for data communication between the control box and the computer)
- AC power cable (110V for US units, 220V for European Service)
- Air supply tube (clear tube, connecting the air pump [within the control box] and the cell, providing air agitation, as needed)

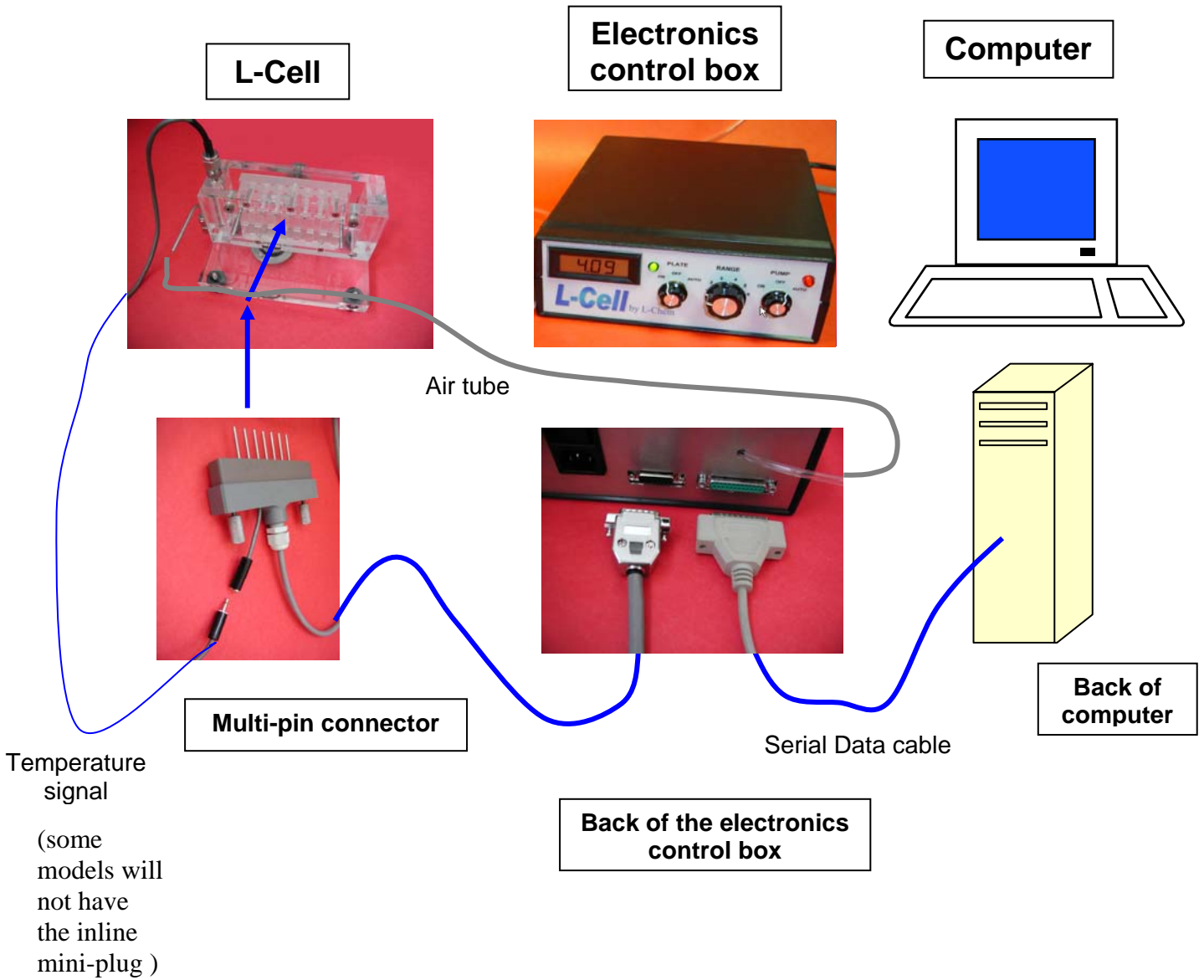
5. Software for data acquisition and analysis (on the accompanying CD)

6. 30 test cartridges (good for 120 tests -- additional cartridges can be purchased from L-Chem at a nominal cost)

7. User's manual

# Wiring Diagram –

Connect the system per the diagram:



## Detailed initial set-up instructions:

1. **Cell** – Remove all packaging material and place cell on a level table-top.
2. **Electronics control box** – Set next to cell
3. **AC Power line** - Insert power cable (provided) to back of cell and plug to 110 V AC electric outlet (220V in Europe).
4. **Multi-pin signal connector** - Insert the multi-pin connector (wide, gray PVC connector with 7 S.S. pins) into the cell (it will fit only in one correct position – do not force!). ***Do not tighten at this time*** – and connect the second end to the matching socket on the back of the electronics control box.



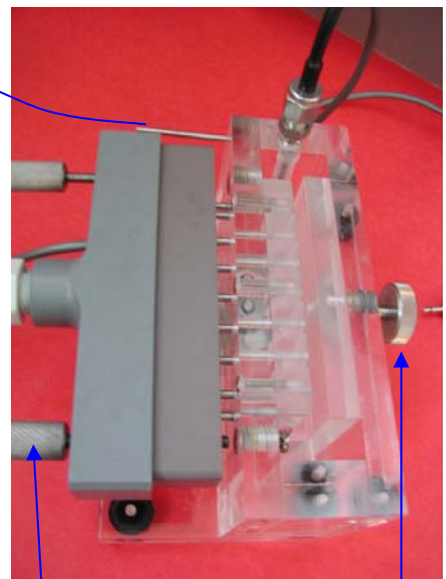
5. **Data cable** - Connect the data cable to the serial port at the back of the electronics control box and the other end to the serial (modem) port of your computer.



Multi-pin  
connector  
to L-Cell

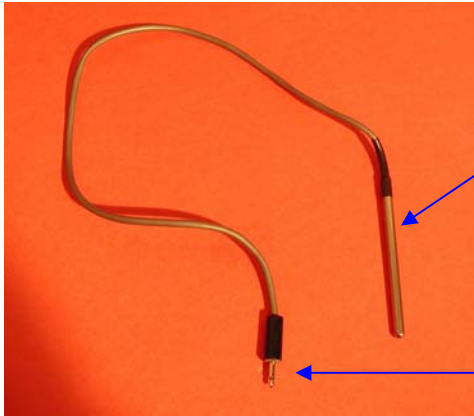
Serial  
cable to  
computer

Air tube  
to L-  
Cell

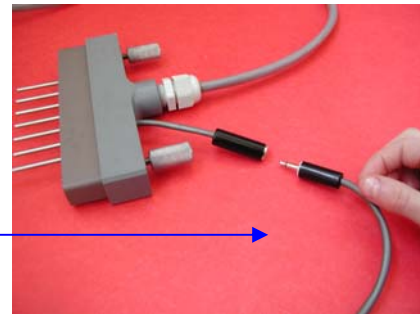


Do not tighten before  
template is inserted

6. **Temperature sensor** - Insert the temperature sensor into the provided cavity in the I-cell, (down to the black insulator), hand tighten the swagelock fitting and connect the mini-plug to the matching mini-jack attached to the multi-pin connector (some models do not have the mini plug and are directed connected to the multi-pin connector) .



Insert this end into cavity on top-front of the I-Cell, hand tighten the swage-lock.



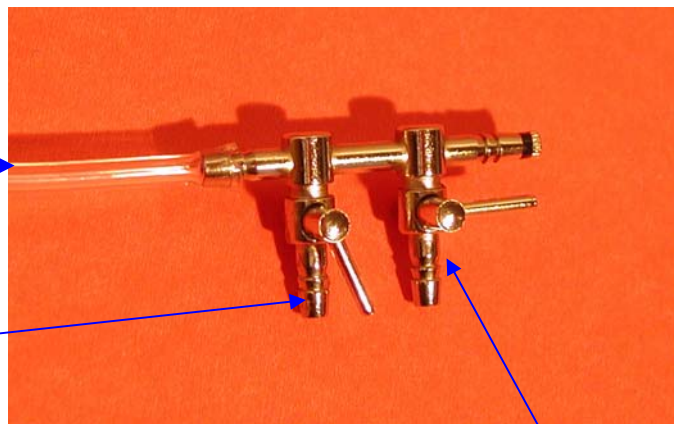
Plug into the mini-jack on the back of the multi-pin connector.

**Note: some models do not incorporate the mini-plug and are 'hard-wired' to the multi-pin connector**

7. **Air supply** - Connect the air tubing that is attached to the back of the electronics control box to the matching SS tube on the side of the cell. Make sure that the supply valve is open and the bleed valve is closed.

Tube from the air pump in the back of the electronics control box

Connect the supplied clear tube from here to the stainless-steel bent tube in front of the L-Cell. Make sure that the valve is partially open




Bleed valve should be closed

**8. Software** – continue with the instructions on the following page.

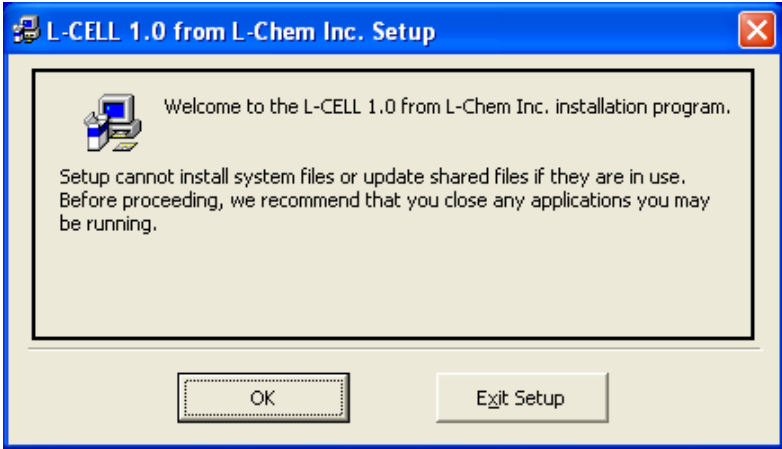
- a. Insert the installation CD with L-Cell software into the CD or DVD drive.
- b. Locate the SETUP.EXE file on the CD and double click on it.
- c. Follow the prompts on the screen.

Below are the prompts and the messages that should appear on the screen during a normal installation:

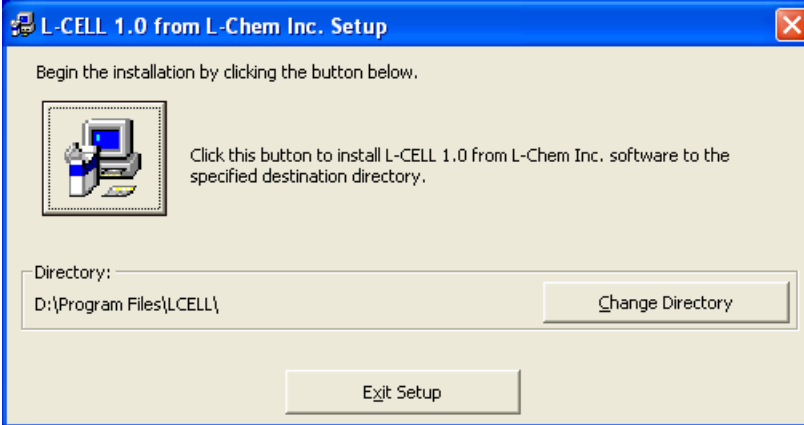
**Step 1.**



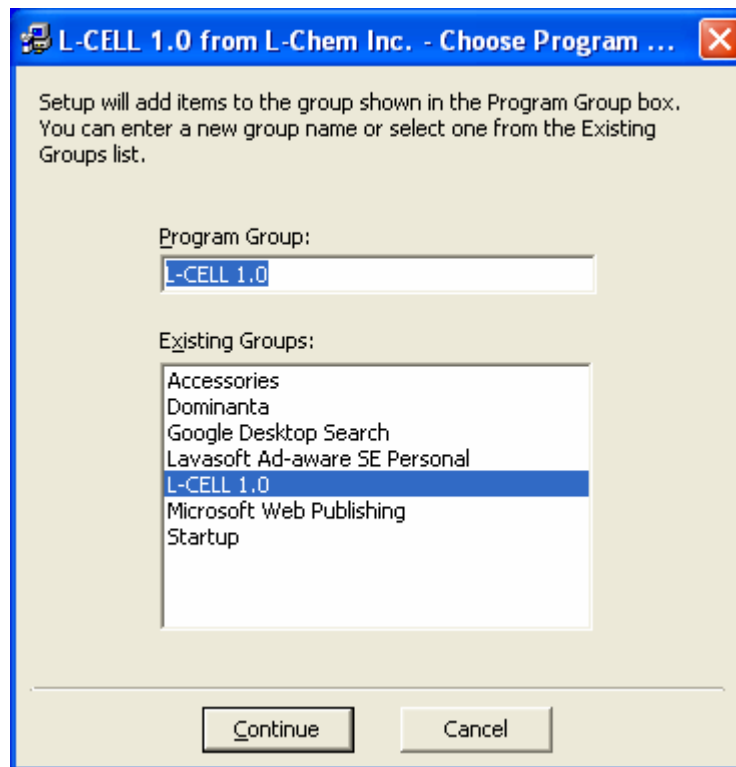
**Step 2.**



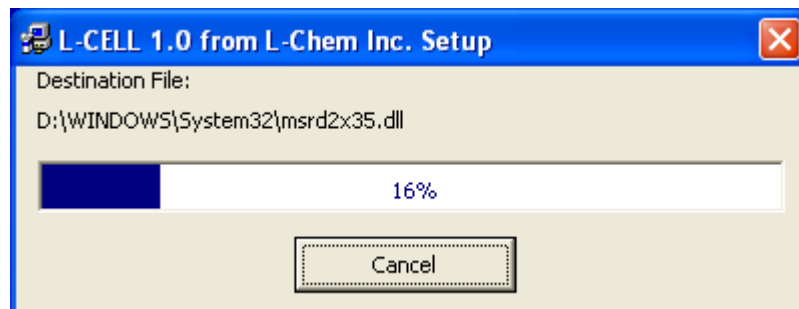
**Step 3.**



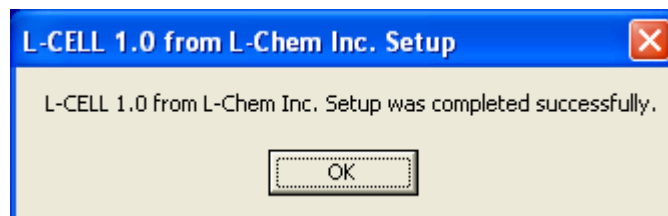
Step 4.



Step 5.



Step 6.



4. Reboot the computer.

**You are now ready to run the tests.**

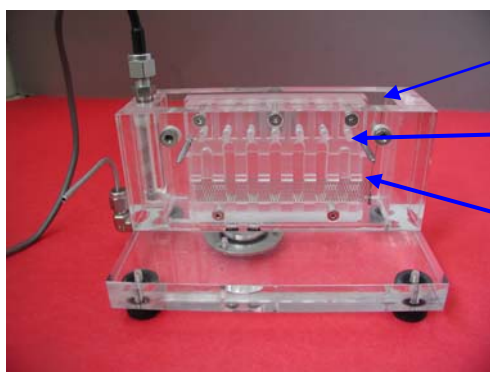
# PREPARING FOR A TEST

## 1. Electrolyte Fill –

- a. Fill the cell with a **50 ml electrolyte** – *note: cell will only be partially (about 1/4) filled.* Fill only to the mark, just above the platinum anode level. If the level is incorrect, the volume can be adjusted using a dropper or a syringe to add or remove electrolyte.

Fill the cell **slowly** from the end opposite to the temperature sensor.

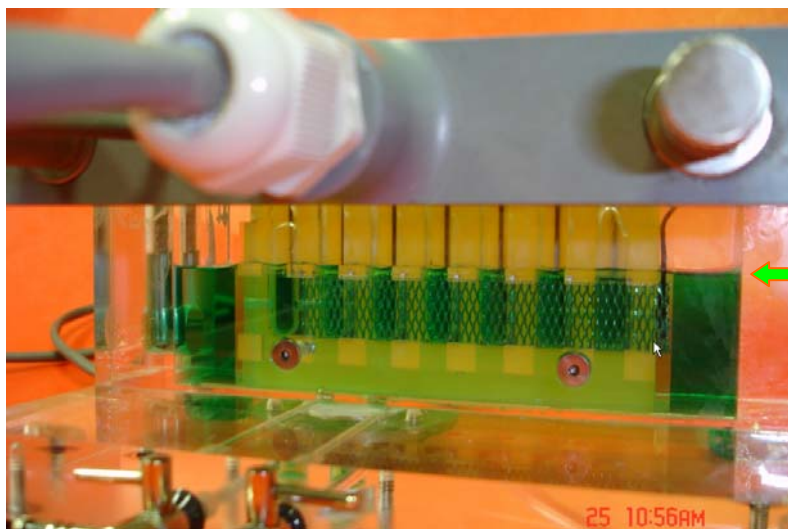
Fill the cell carefully so that you **do not to wet the contact pins holes or the plating mask.** Wetting may cause shunt currents leading to erroneous results. If wetting above the electrolyte level occurs, open the cell and dry wetted parts above electrolyte level using an adsorbent paper towel, or by blowing air.



Fill cell **slowly** here

Do not wet channels

Electrolyte level -  
**Make sure not to fill above this level.**



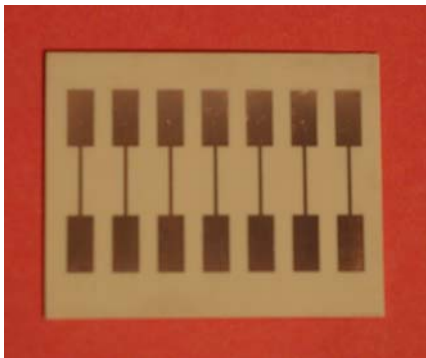
The L-Cell filled with nickel plating solution.

**Fill to this level (just cover completely the anode mesh)**

2. **Cartridge preparation** – The cartridges are shipped covered with a transparent protective layer to prevent the metal substrate from excessive oxidation. Prior to the test, use a **wet, grade 600 or grade 0 silicon carbide paper** to **lightly** polish the surface. Do not apply excessive force to minimize scratching of the surface.

The cartridge is double-sided and symmetric and may be used four (4), when inserted the second time top-down, and the third and fourth time when it is inverted to the other side. .

*Never use a cartridge more than once on each side (4 times total for a double-sided cartridge). Multiple uses (even after re-polishing) may lead to erroneous results.*

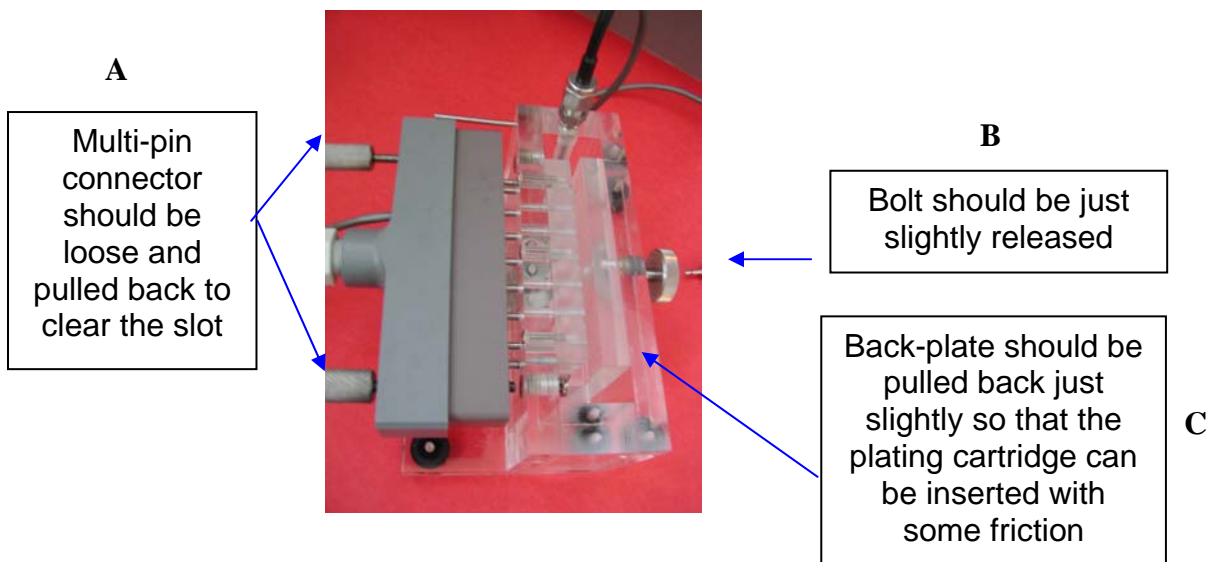


The plating cartridge, should be polished lightly (e.g., by silicon carbide paper) prior to use.

The cartridge is symmetric and double sided: when turned upside down it can be used a second time. When it is turned back-to-front it can be used twice more.

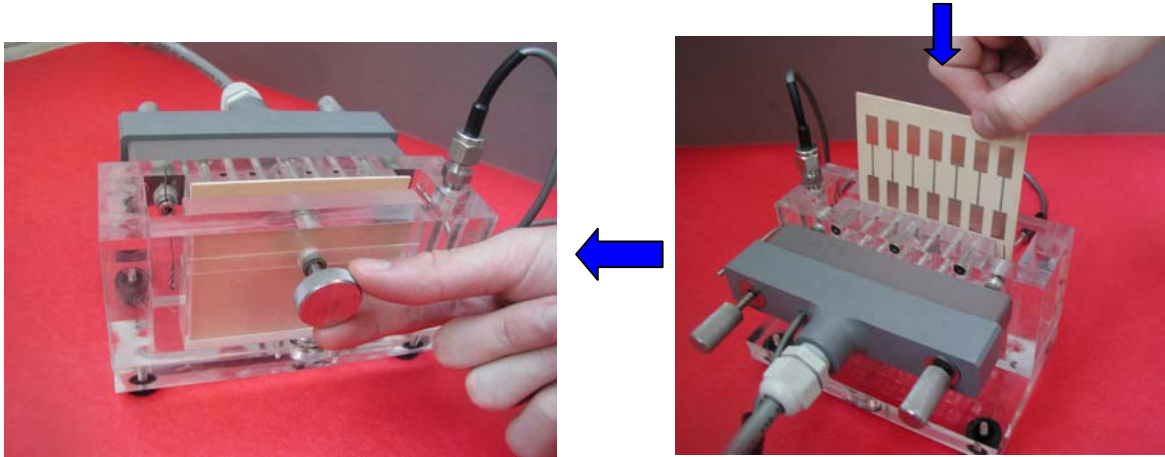
**3. Inserting the cartridge into the cell – (Important - please follow these instructions carefully):**

- a. Make sure that the multi-pin grey connector is loose and pulled out so that the contact pins do not block the insertion of the cartridge. Alternatively, you may initially remove the connector completely out of the cell.
- b. Open the bolt tightening the back plate just slightly so that the back plate can be pulled slightly away from the mask clearing a narrow slot just sufficient for inserting the cartridge (without any slack) using a slight pushing force to overcome friction. This snug fit during insertion is important for preventing wetting of the cartridge above the immersion depth.



- c. Insert the cartridge just a little bit (~1/2 inch). Then lightly tighten the backplate bolt so that the cartridge is held in place by slight friction.
- d. Slide slowly the cartridge down towards the bottom of the cell, until it is seated all the way in.

- e. Hand-tighten slowly the backplate bolt so that the backplate pushes and holds the cartridge firmly against the mask. *Do not over-tighten – this may damage the cell.*



- f. Push in the (grey) multi-pin connector and tighten its two side-bolts. Make sure that the connector is seated all the way against the cell.

**4. Electronic control box settings –**

- a. **Power** – Switch main power switch at the back of the electronics control box to ‘on’. The power indicator red light on the right side of the front panel will light up.



- b. **Pump** – Turn the pump switch to ‘auto’.
- c. **Range** – Turn the range knob to select a range (displayed on the digital voltmeter) that is appropriate to the tested electrolyte – see table below. It is recommended that the voltage will be selected sufficiently high as to provide the largest plating range, (preferably corresponding to the range of plating interest) yet not exceed on the segment

with the highest current density the limiting current for the L-Cell (see below). This limiting current in the L-Cell corresponds an equivalent, Nernst-type, boundary layer of  $1.3 \times 10^{-2}$  cm.

Typical range settings:

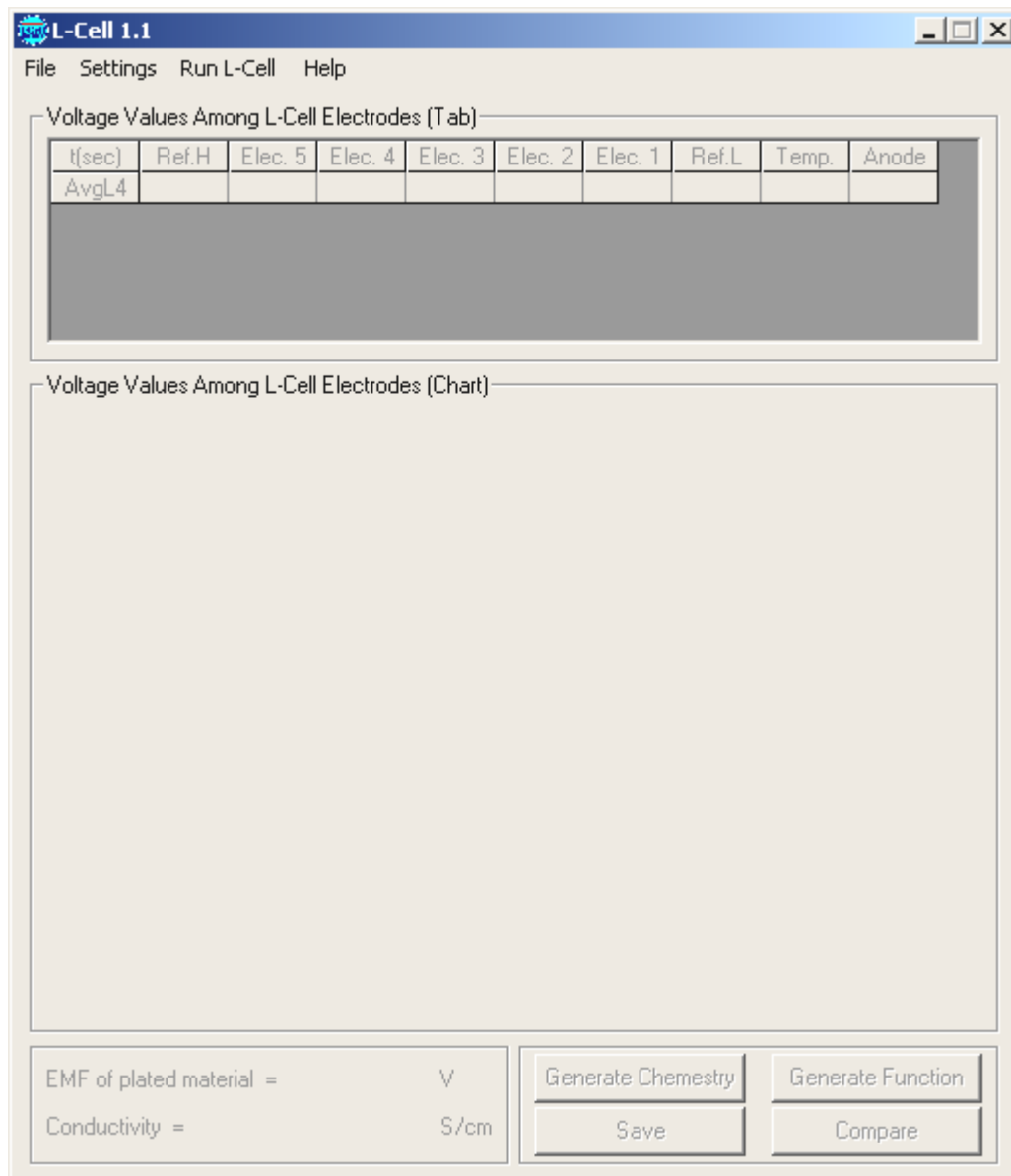
Plating solution	Metal conc. [M]	pH range	Suggested Voltage Setting [V]	Highest Current density [mA/cm <sup>2</sup> ]	Lowest current density [mA/cm <sup>2</sup> ]	Maximal voltage settings [V]	Limiting current density [mA/cm <sup>2</sup> ]
Copper sulfate (Acidified)	0.5	0-1	3	25	5	3.5	40-45
Same as above	0.1	0-1	~ 1.5	~ 8	~2	~1.7	8-10
Copper sulfate (Non Acidified)	0.5	2 - 4	3.5 - 4	30	6	4.5	60-65
Same as above	0.1	2 - 4					12-15
Watts Nickel	1.1	2-4	5 - 6	75	10-15	7	> 100

d. **Plate** – set to ‘AUTO’

# BATH PERFORMANCE ANALYSIS – Polarization and Kinetics

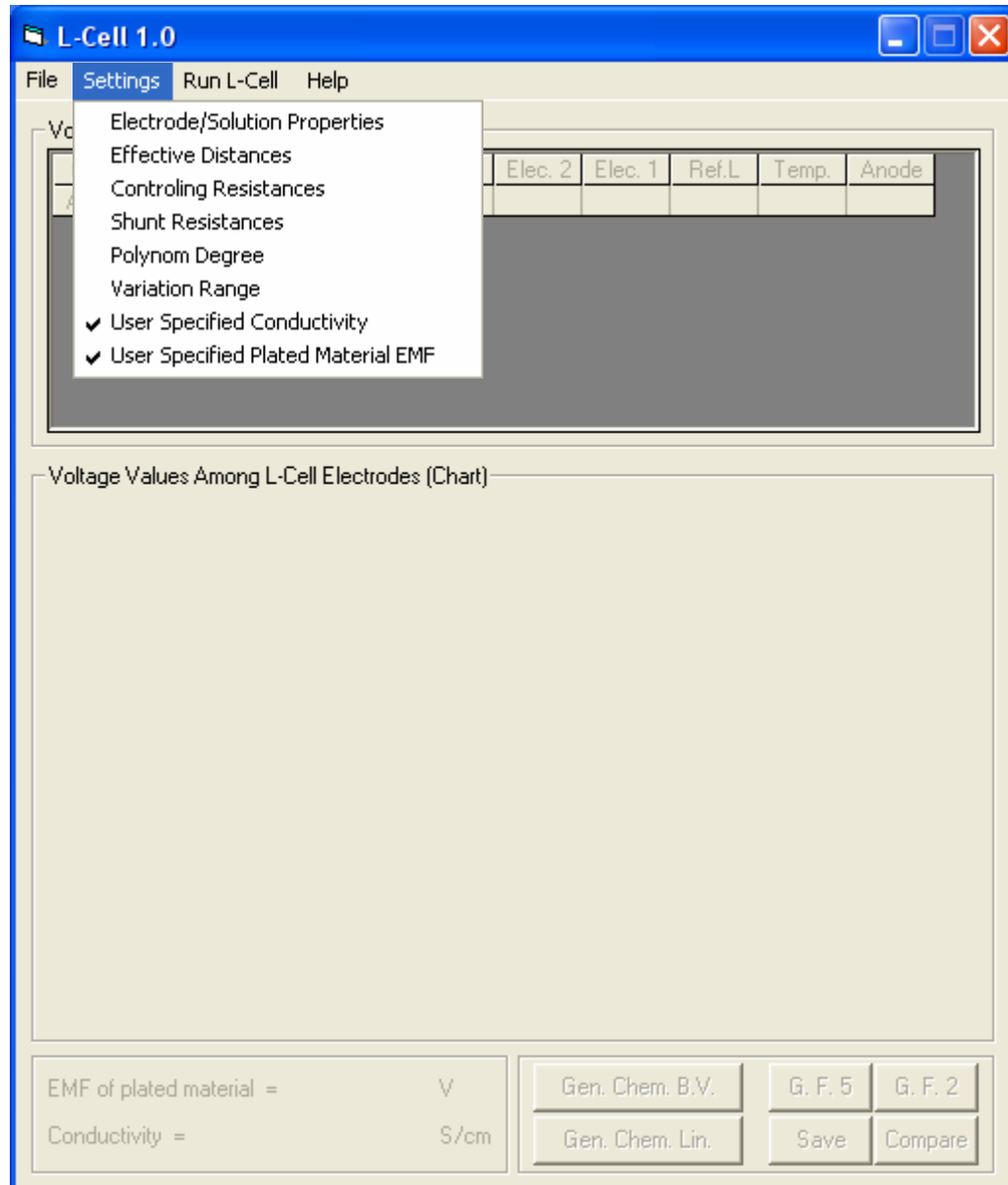
Fill up the L-Cell, insert the plating cartridge, and set the control module per instructions in the previous section.

1. **Run the control program** – on the computer, click on ‘Start’ > ‘Programs’ > ‘L-Cell’ or click on the ‘L-Cell’ icon on your desk-top.
2. The following **start-up screen** will show-up.



### 3. Adjust the Settings (if necessary)

Click on 'Settings' in the main menu. Select (click on) 'Electrode/solution Properties'.

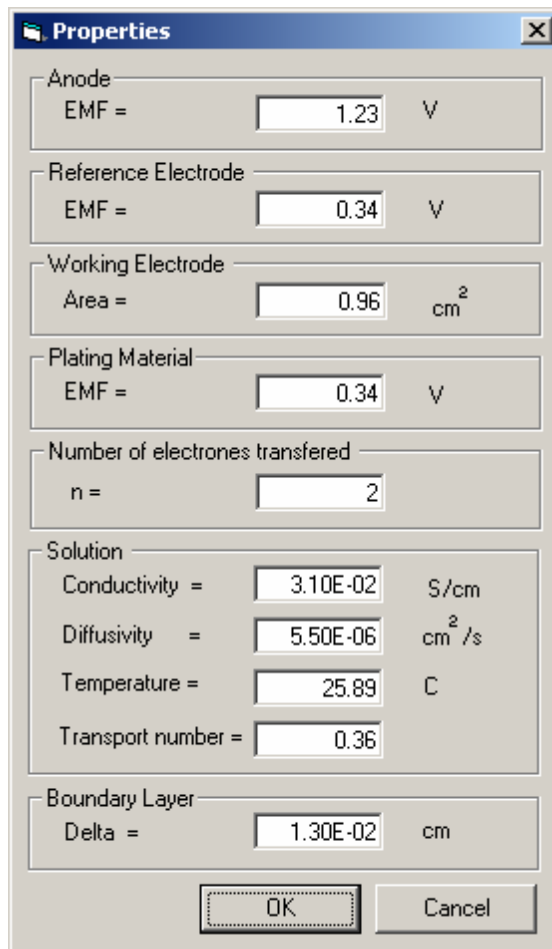


The following sub menu will appear:

Make sure that the following parameters are correctly set for the plated metal:

- Reference electrode EMF (standard potential) – should be set to the same value as that of the plated metal when using (as recommended) a cartridge that is pre-plated with the same metal as the tested metal (see below)
- Plated material EMF – type in the appropriate value for the tested metal (see below)
- Numbers of electrons transferred – type in the appropriate value for the tested metal (see below)
- Conductivity – enter value if accurately known. Otherwise, leave the default value and the L-Cell program will substitute it with a measured value (see discussion later-on)
- Transport number – see table below

All other parameters – leave in default values unless otherwise indicated



#### Select List of Suggested Parameters

Metal	EMF [V]	Number of Electrons Transferred	Transport Number
Copper, acidified (pH ~ 0)	0.34	2	0
Copper, low acidity (pH ~ 2-4)	0.34	2	0.36
Watts Nickel	-0.24	2	0.1

**All other defaults values have been set and unless you make any principal configuration changes you are not expected to change those.**

4. To *run the test*: from the main menu select 'Run L-Cell'. The following window will appear.

L-Cell Control panel

Specify following properties :

Metal : Unknown

Type : Unknown

Make : Unknown

Metal concentration : mol/L

Recommended plating time

Time : 60 Sec.

Make sure :

1. L - Cell box is connected
2. Pour ~50 ml solution into cell
3. Insert template and tighten clamp
4. Connect power manifold to cell
5. Connect air agitation (optional)
6. Turn power (control box) 'ON'

CLOSE NEXT STEP

Fill in the required fields. The Metal, Type and the Make are optional identifiers that will later appear as a combined name for the optionally saved file with the summary of the kinetics analysis. For example, the data for low acidity 0.5 M copper solution are shown. If you want to change the default time unclick the 'Recommended time' check box and specify a new time.

L-Cell Control panel

Specify following properties :

Metal : Cu

Type : Pure

Make : L-Chem Inc.

Metal concentration : 0.5 mol/L

Recommended time

Time : 60 Sec.

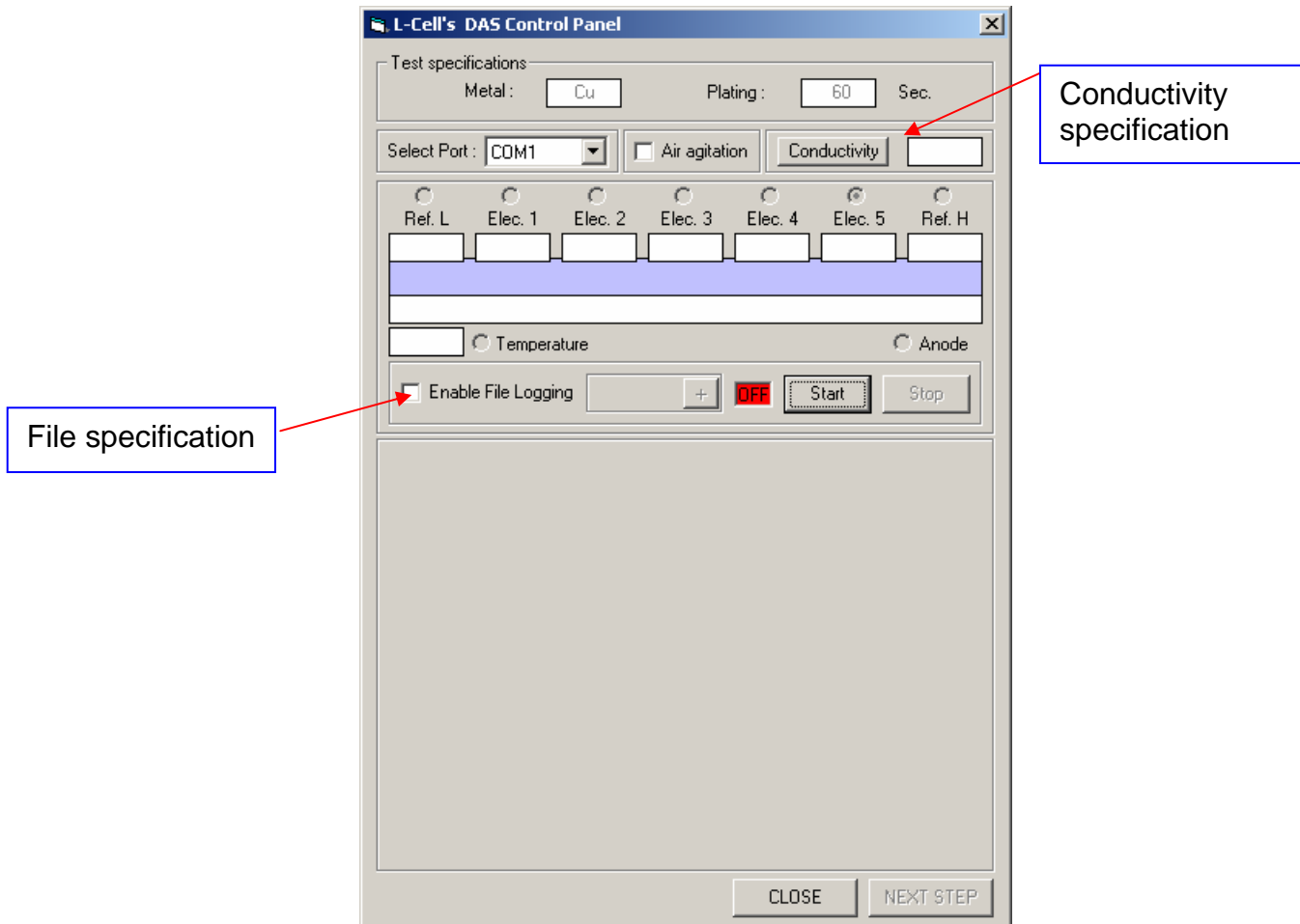
Make sure :

1. L - Cell box is connected
2. Pour ~50 ml solution into cell
3. Insert template and tighten clamp
4. Connect power manifold to cell
5. Connect air agitation (optional)
6. Turn power (control box) 'ON'

CLOSE NEXT STEP

**For kinetics measurements the default value of 60 seconds is recommended. For long term deposition with the objective of creating thicker deposit samples set the Time value to the desired deposition time.**

After clicking Next Step, the following window will show:



Conductivity:

To include the conductivity measurements, click on the 'Conductivity' button. A flashing red reading will appear in the conductivity pane. Wait until flashing stops and the conductivity value turns into gray. This reading will be incorporated in the analysis.

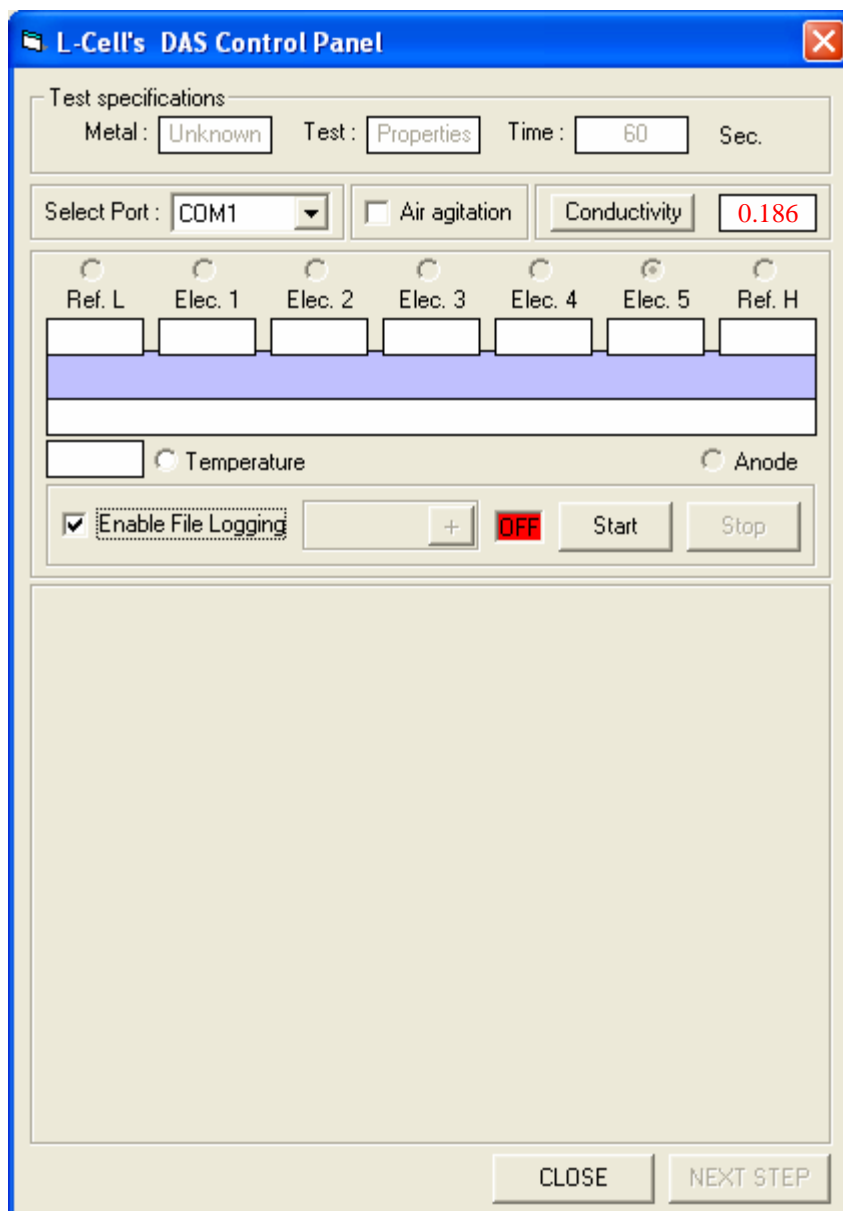
Please note that the built-in conductivity meter is most reliable within the range of 0.001 - 0.2 S/cm. If you have a more accurate external reading you should skip the conductivity measurements part and enter it in the 'Settings' menu. To access it select from the main menu: 'Settings' > 'Electrode/Solution properties'. A window will appear in which you can enter the externally provided conductivity value.

Data storage:

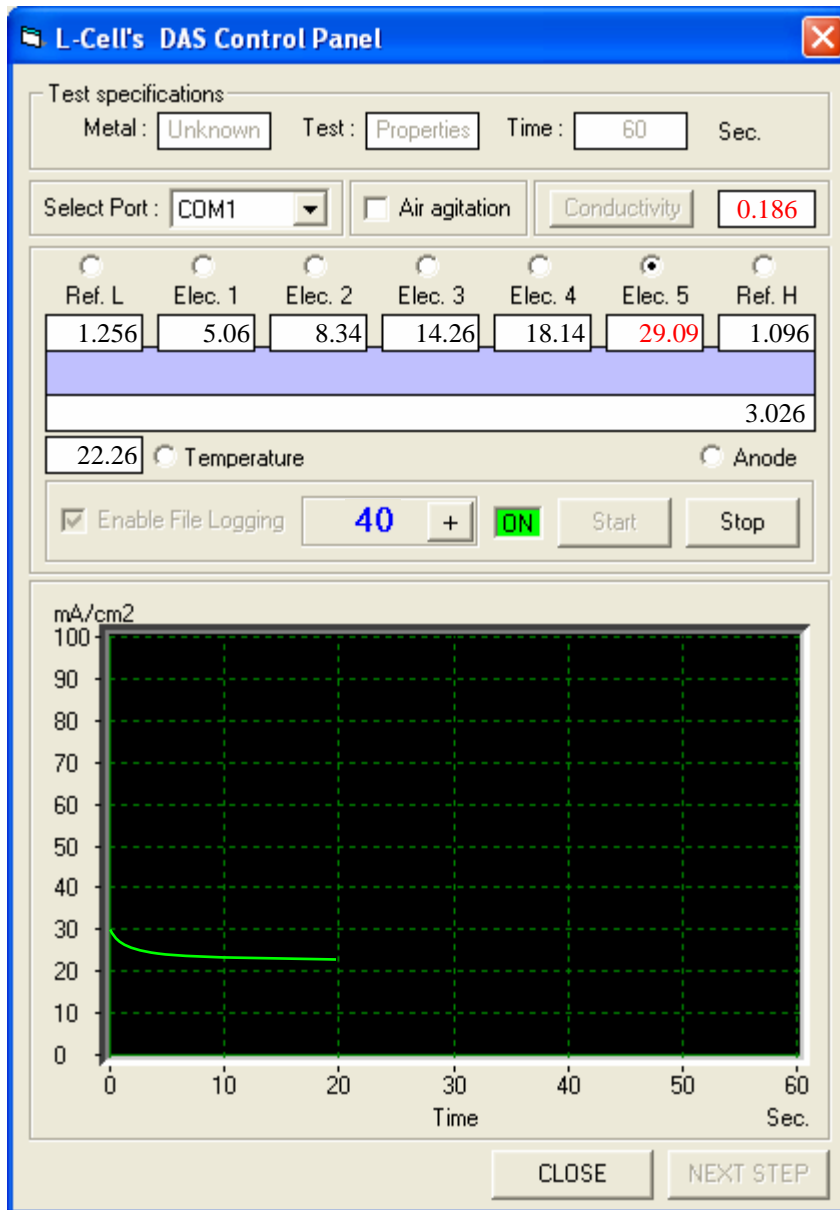
Check the Enable File Logging check box and provide a name for the file in which you want to store the experimental results.

**For kinetics measurements do NOT apply the air agitation by checking the Air agitation check box.**

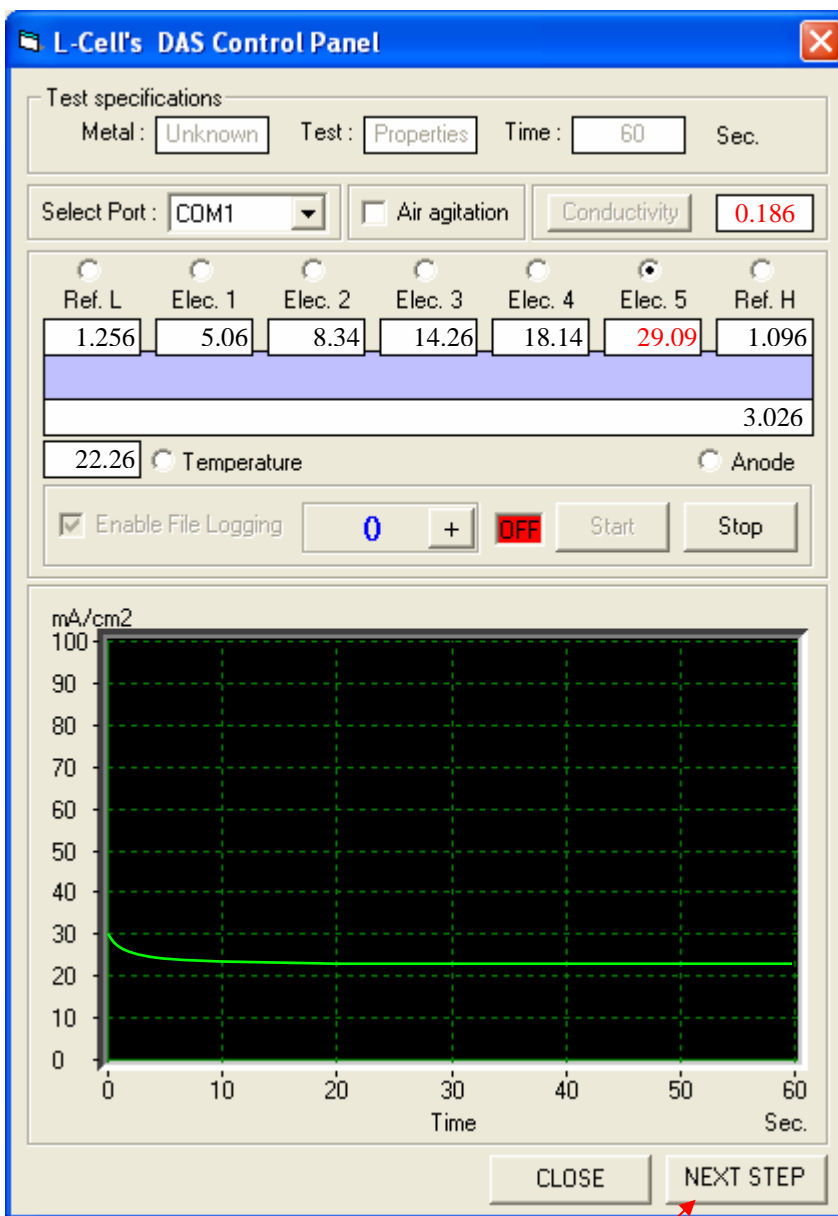
The following window shows the measured conductivity value and the file logging specification.



To run the test click on the Start button. The following window will show up. The numbers in the small frames indicates the local currents on the corresponding pads. The values on the two extremes are the reference electrode readings. The oscilloscope type graph represents the real-time reading of the current density of the electrode which has been checked.

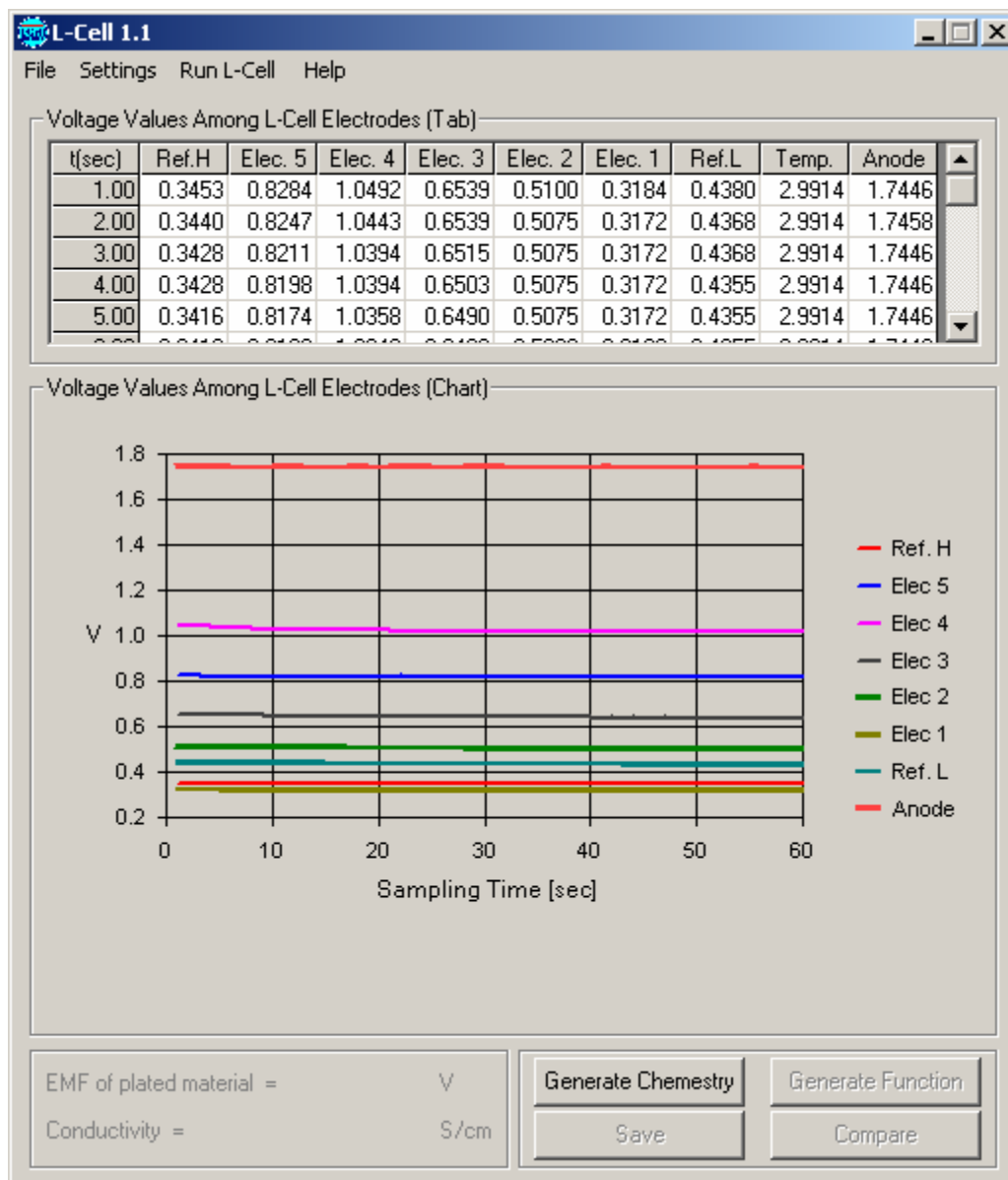


Once the test has been complete the ON indicator changes to red OFF. Click on the Next Step button to continue the analysis.



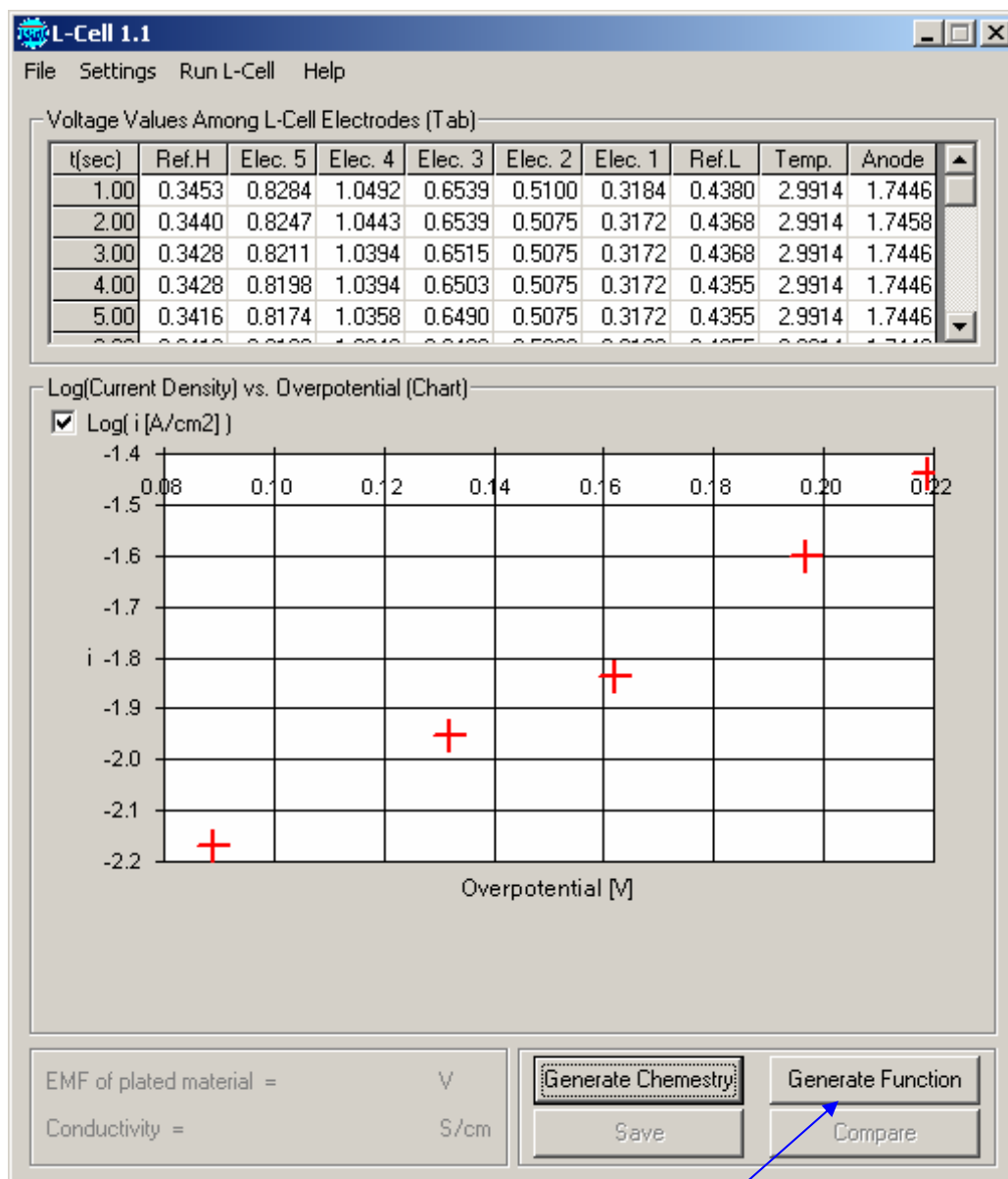
Click here to proceed

The following screen will appear.



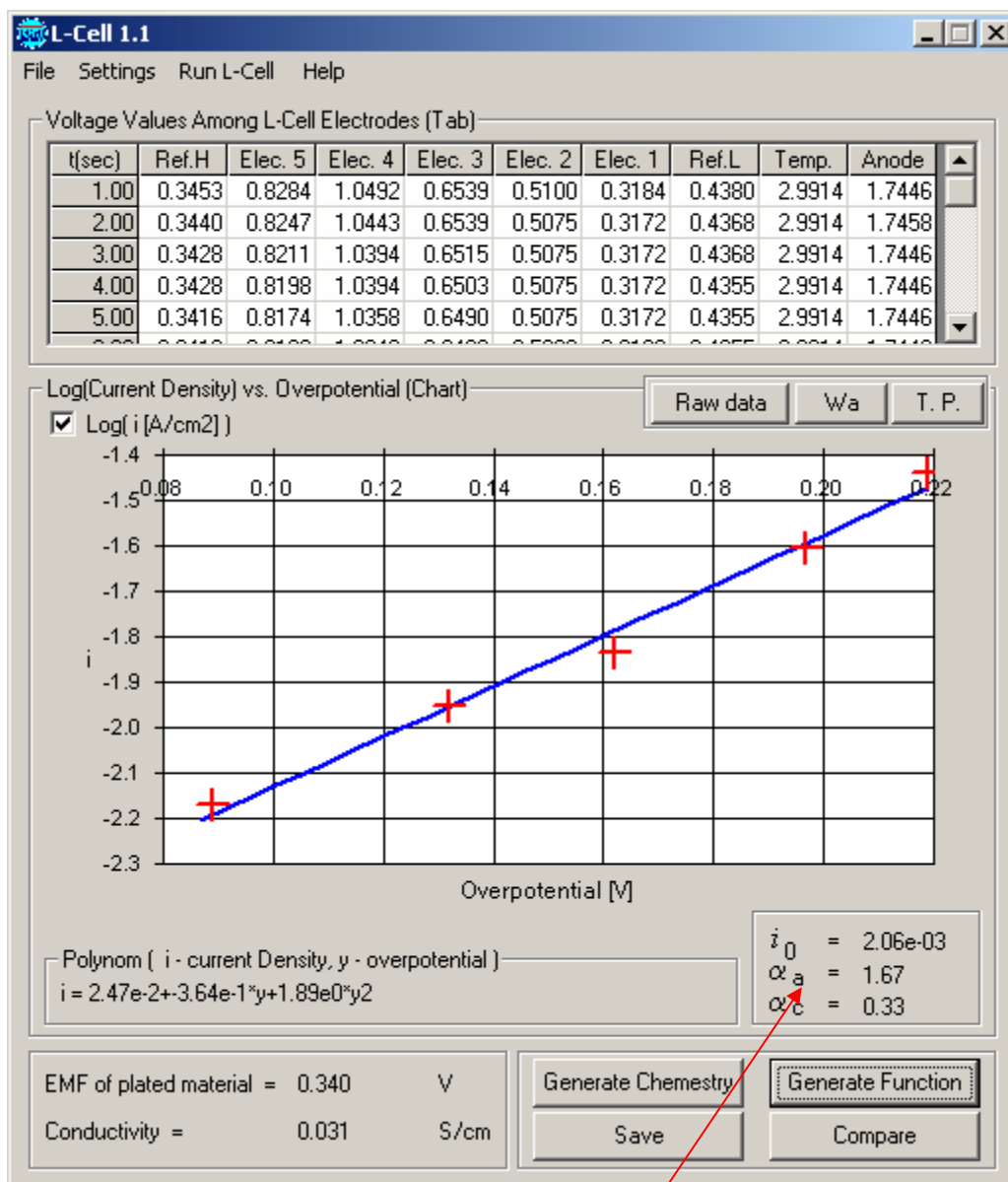
To generate the polarisation data click on the *Generate Chemistry* button.

The following data and graph will show.



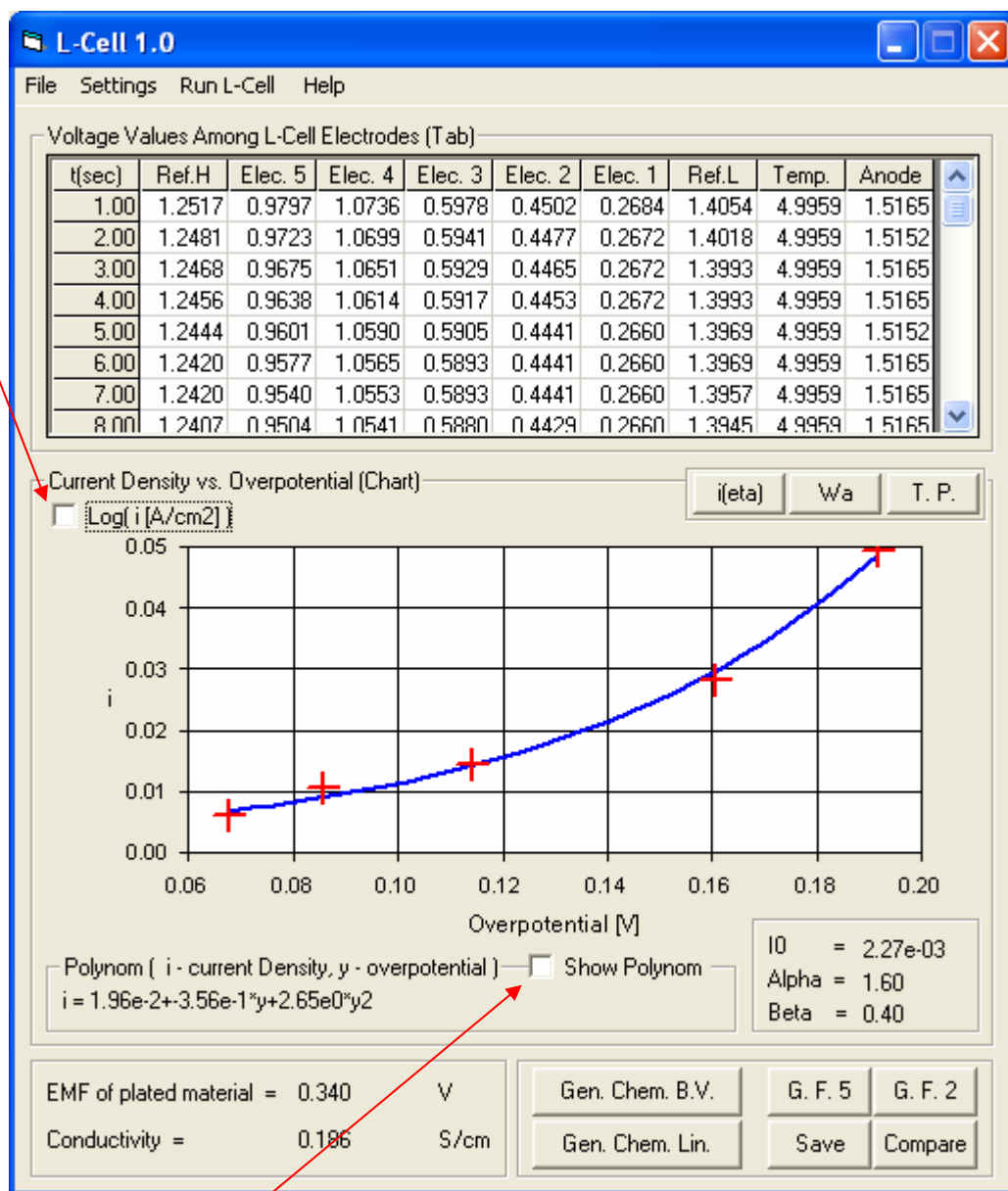
To extract the kinetics parameters click on the '*generatr function*' button.

**Note:** that the polarization data above have been corrected for mass transport effect, i.e., it represents pure kinetics. One consequence is that the current densities displayed, which correspond to the measured voltages, are higher than the measured current densities.

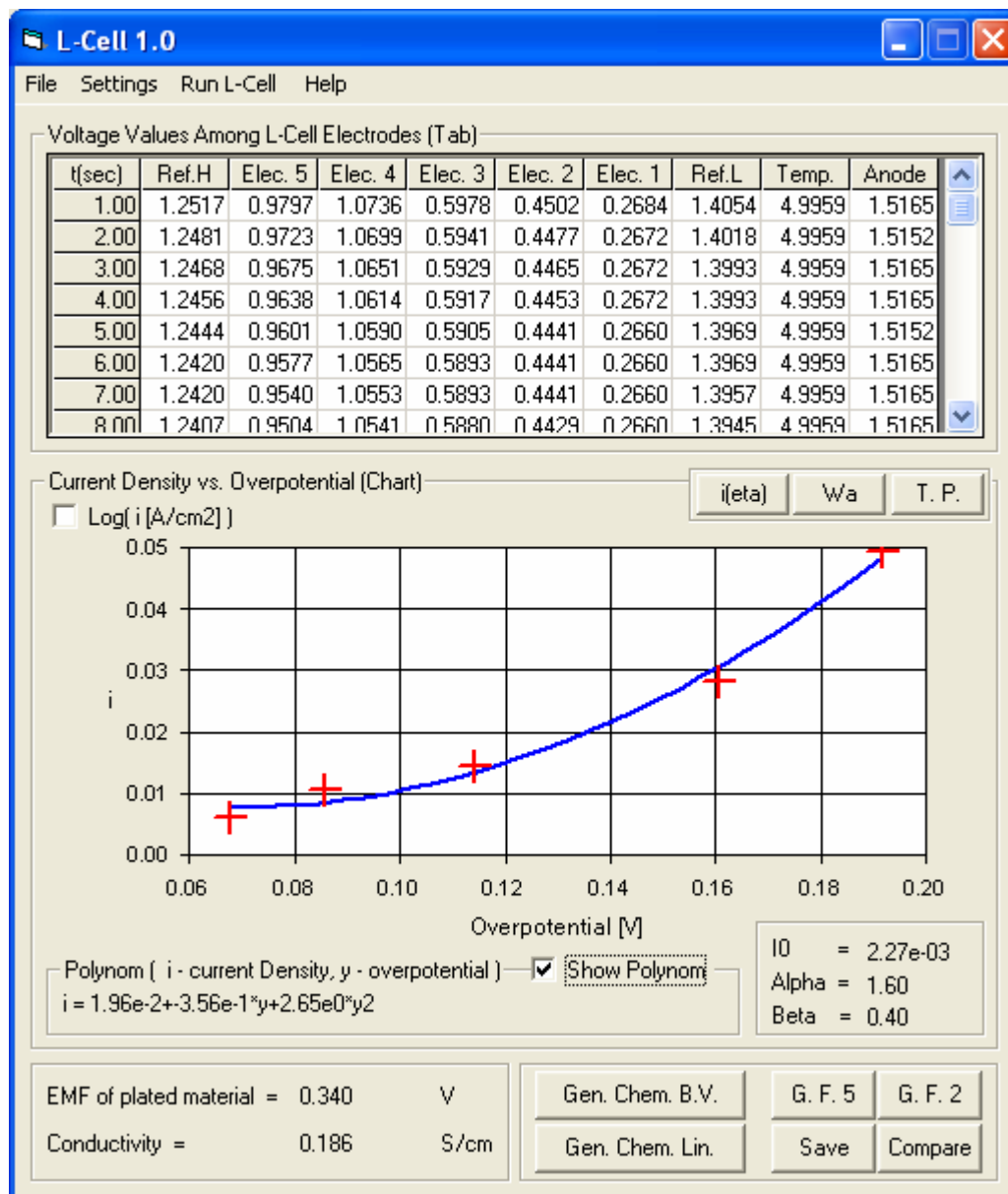


The kinetics parameters values will show at the bottom right side of the window.

To view a non-logarithmic relationship between current densities and the overpotentials uncheck the Log(i) check box.

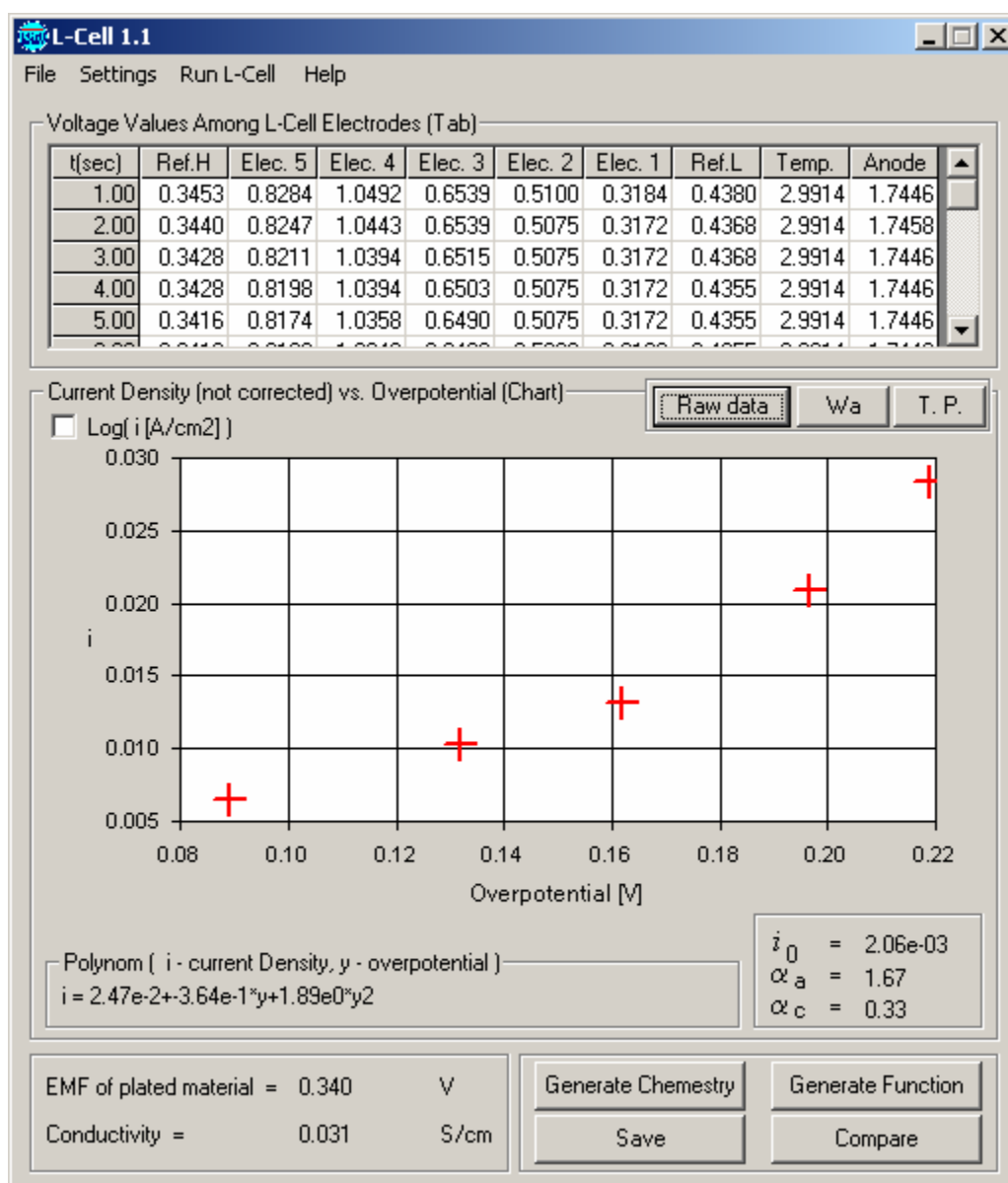


For a polynomial representation of the kinetics data (instead of the Butler-Volmer representation) click on the Show Polynom check box. The following graph will show.

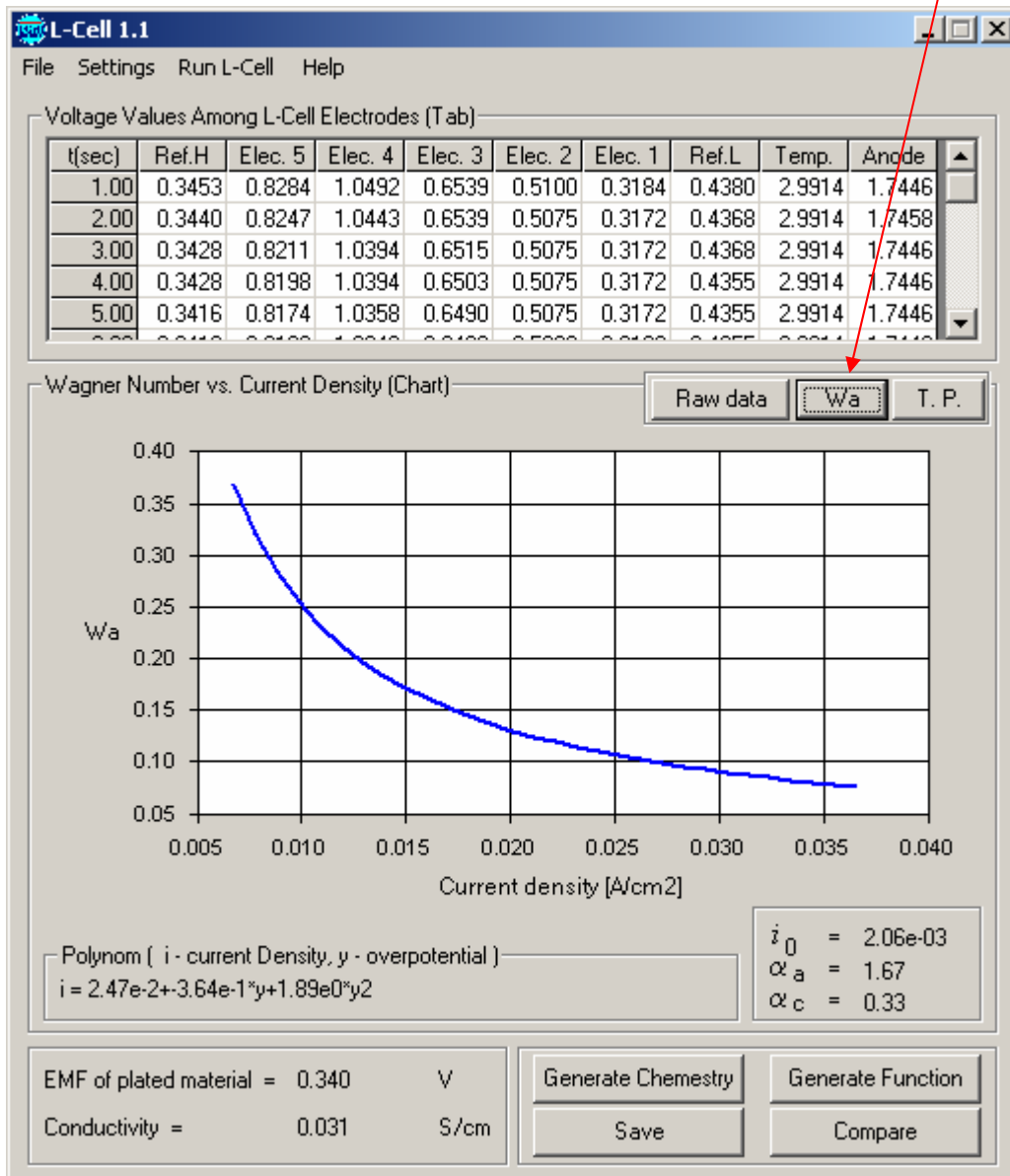


To view the raw polarization data (not corrected for mass transport) click on the i(eta) button located at the right upper corner of the screen.

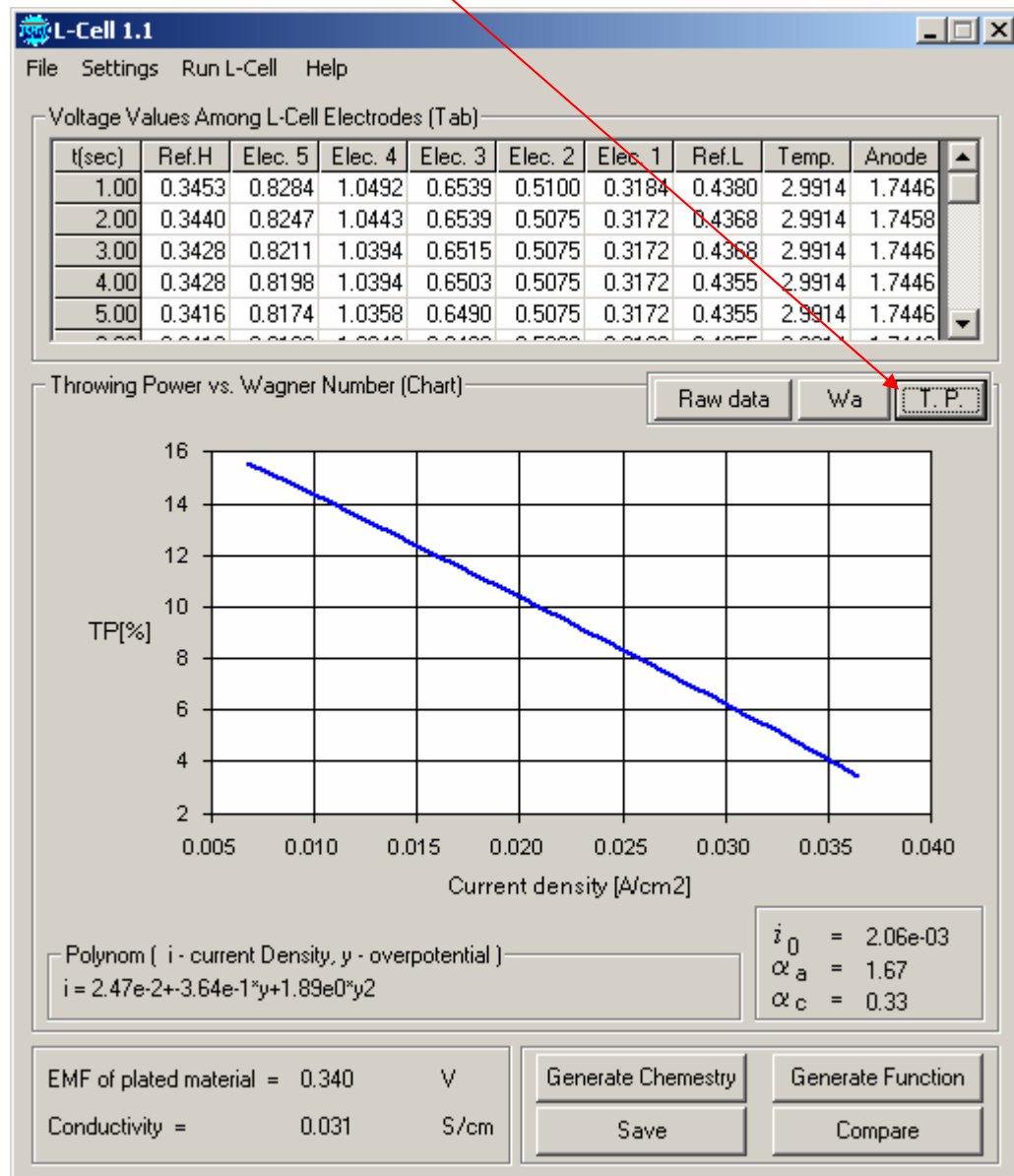
The recorded (raw) polarization data appear as follows:



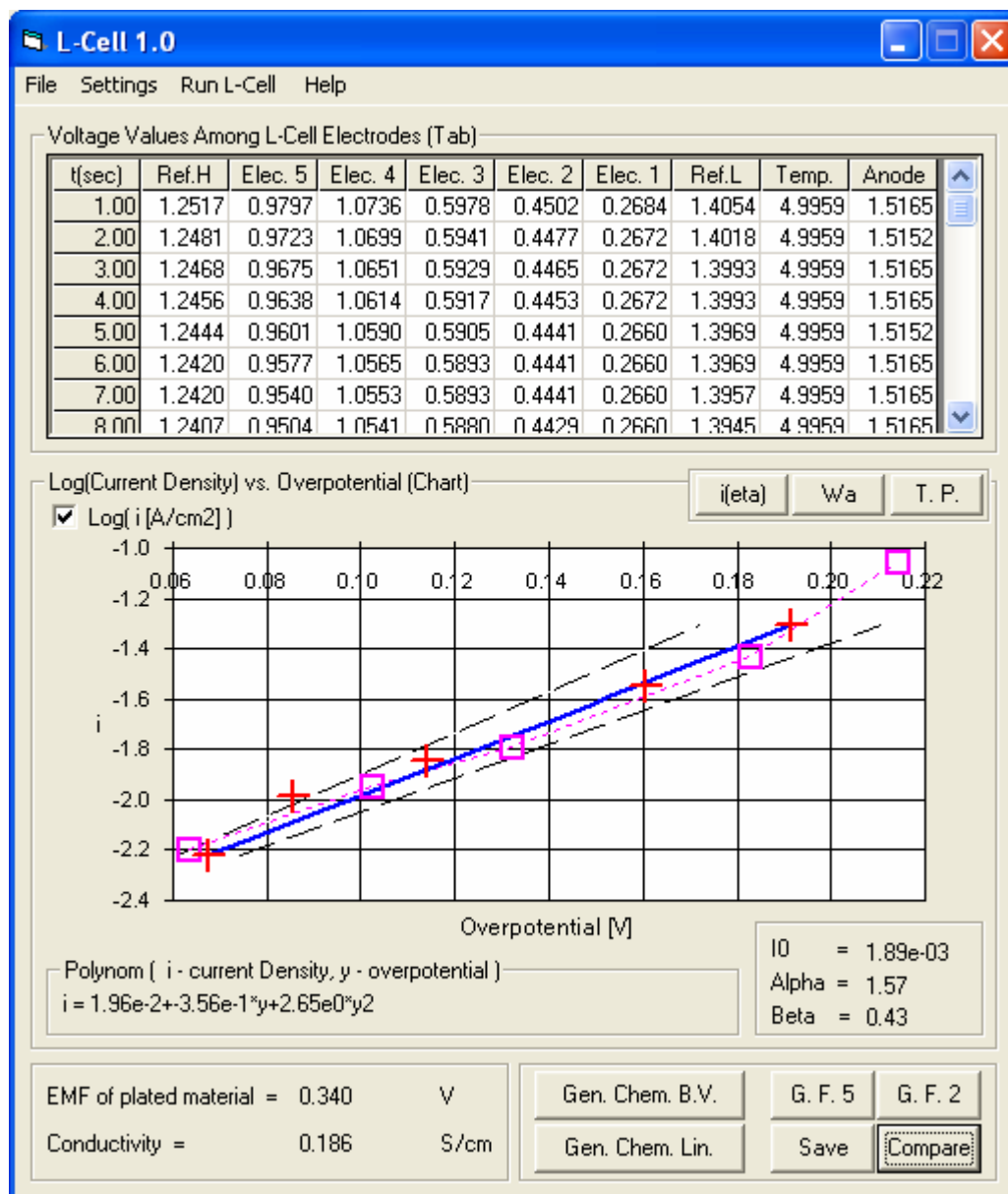
To view the Wagner number and its dependence on the current density click on the Wa button.



To view the throwing power click on the T.P button.



To compare two current data to data that has been run previously and saved click the Compare button. The screen below will appear. The current data will be show in red crosses and blue line and the compared data will be shown in pink squares and dotted line. The black dashed lines indicate a 10% variation from the current data. The 10% default value can be changed by the user in the Settings submenu.



# **BATH PERFORMANCE ANALYSIS –**

## **Interpretation of the Results**

Since variations between different commercial bath formulation can be large, it is recommended that the L-Cell not be used in an absolute mode but rather as a tool for comparing bath characteristics. To this end, it is advisable that the user establishes a standard, perhaps, a newly mixed or a well performing bath, to which other baths be compared.

### **Setting a standard:**

Mix a new electrolyte and test its performance in plating a typical part. If performance appears good,

### **Comparing a production solution to the standard:**

# BATH PERFORMANCE ANALYSIS – Generation of plated samples

The kinetics measurements are completed in one minute and therefore the plated cartridge may not provide sample with adequate deposit thickness.

In order to perform longer plating tests start as indicated above, except that in the test specification page (shown below) uncheck the 'recommended plating time' and specify an appropriate (longer) plating time.

The screenshot shows a software window titled "L-Cell Control panel". It contains several input fields and a list of instructions. The "Specify following properties" section includes dropdown menus for "Metal" (Unknown), "Type" (Unknown), and "Make" (Unknown), and a text box for "Metal concentration" (mol/L). Below this is a section with a checked checkbox for "Recommended plating time" and a "Time" field set to "60 Sec.". At the bottom, there is a "Make sure" section with a list of six steps: 1. L - Cell box is connected, 2. Pour ~50 ml solution into cell, 3. Insert template and tighten clamp, 4. Connect power manifold to cell, 5. Connect air agitation (optional), and 6. Turn power (control box) 'ON'. At the bottom right, there are "CLOSE" and "NEXT STEP" buttons.

Click on the 'next step' button' and in the following menu click 'start'. Recording the conductivity data and saving the file are optional.

You may want to check the 'air agitation' check box to provide air agitation for enhanced mass transport. The air flow, directed to the first and second segments where the current density is highest, can be adjusted using the valves in front of the cell. It is recommended to use only moderate air bubbling.

# DEFAULT SETTINGS –

All defaults values have been set and unless you make any configuration changes you are not expected to change those. Do not change those without first consulting with L-Chem.

The following windows show the default settings.

**Shunt Resistances**

Shunt Resistances

@ Electrode 1 =	<input type="text" value="29.67"/>	Ohm
@ Electrode 2 =	<input type="text" value="50.54"/>	Ohm
@ Electrode 3 =	<input type="text" value="50.43"/>	Ohm
@ Electrode 4 =	<input type="text" value="50.68"/>	Ohm
@ Electrode 5 =	<input type="text" value="50.67"/>	Ohm

**Controlling Resistances**

Controlling Resistances

@ Electrode 1 =	<input type="text" value="0.20"/>	Ohm
@ Electrode 2 =	<input type="text" value="0.20"/>	Ohm
@ Electrode 3 =	<input type="text" value="50.63"/>	Ohm
@ Electrode 4 =	<input type="text" value="90.77"/>	Ohm
@ Electrode 5 =	<input type="text" value="199.74"/>	Ohm

**Effective Parameters**

Effective Distances

Electrode To Anode =	<input type="text" value=".7625"/>	cm
Reference To Electrode =	<input type="text" value=".5245"/>	cm

Anode Wire Resistance =  Ohm  
Cathode Wire Resistance =  Ohm

Voltage Multiplier =   
Temperature Multiplier =   
Hi Reference Multiplier =   
Low Reference Multiplier =

Anode Area Fraction =   
Volume Exchange Fraction =

Conductivity linear interpolation

$$K = a * V^3 + b * V^2 + c * V + d \text{ S/cm}$$

where V - DA conductivity reading