





CONTENTS

| Chapter 1 | I. Introduction | 1 |
|-----------|--|----|
| 1-1. | Introduction | 1 |
| 1-2. | Electrochemical technique | 1 |
| 1-3. | Required system configuration | 2 |
| 1-4. | Hardware specifications | 2 |
| Chapter 2 | 2. Basic Information | 3 |
| 2-1. | Update information | 3 |
| 2-2. | Specification changes | 3 |
| 2-3. | Damage during transportation | 3 |
| 2-4. | Product warranty | 3 |
| 2-5. | Service information | 3 |
| 2-6. | Software warranty | 3 |
| Chapter 3 | 3. Installation | 4 |
| 3-1. | Shipment Inspection | 4 |
| 3-2. | Software installation | 5 |
| 3-2- | 1. Windows 11 (x64), Windows 10 (1803) (x64, x86) or later | 5 |
| 3-2-2 | 2. Windows 10 (1803) (x64, x86) or earlier | 5 |
| 3-3. | Equipment setup | 6 |
| 3-4. | Operation check | 7 |
| 3-5. | Measurement preparation | 11 |
| 3-6. | Cell control terminal | 12 |
| Chapter 4 | 4. File Menu | 13 |
| 4-1. | Open | 13 |
| 4-2. | Save As | 13 |
| 4-3. | Delete | 14 |
| 4-4. | List Data File | 15 |
| 4-5. | Convert to Text | 16 |
| 4-6. | Text File Format | 17 |
| 4-7. | Import Text File | 20 |
| 4-8. | Print | 20 |
| 4-9. | Print Multiple Files | 21 |
| 4-10. | Print Setup | 21 |
| 4-11. | Exit | 22 |



| Chapter 5 | 5. Setup Menu | 23 |
|-----------|--|----|
| 5-1. | Technique Selection | 23 |
| 5-1-1 | . Sweep technique (LSV, CV) | 24 |
| 5-1-2 | . Amperometric technique (i-t) | 26 |
| 5-1-3 | . Open circuit potential-time (OCPT) | 27 |
| 5-2. | Parameter Setting | 28 |
| 5-2-1 | . Cyclic voltammetry parameters | 29 |
| 5-2-2 | . Linear Sweep Voltammetry parameters | 31 |
| 5-2-3 | . Amperometric i-t curve parameters | 32 |
| 5-2-4 | . Open Circuit Potential - Time parameters | 33 |
| 5-3. | System Setup | 34 |
| 5-4. | Hardware - Test | 36 |
| Chapter 6 | 5. Control Menu | 37 |
| 6-1. | Measurement | 37 |
| 6-2. | Pause/Resume | 37 |
| 6-3. | Stop Run | 37 |
| 6-4. | Reverse Scan | 38 |
| 6-5. | Run Status | 38 |
| 6-6. | Repetitive Run | 40 |
| 6-7. | Macro Command | 41 |
| 6-8. | Open Circuit Potential | 45 |
| 6-9. | Filter setting | 46 |
| 6-10. | Cell | 47 |
| 6-11. | Step Functions | 49 |
| 6-12. | Preconditioning | 50 |
| 6-13. | Rotating Disk Electrode Control | 51 |
| 6-14. | Other Controls | 52 |
| 6-15. | Stripping Mode | 53 |
| Chapter 7 | 7. Graphics Menu | 54 |
| 7-1. | Preseent Data Plot | 54 |
| 7-2. | New Overlay Plot | 56 |
| 7-3. | Add Data to Overlay | 56 |
| 7-4. | Replot overlay | 57 |
| 7-5. | Manage Overlay | 57 |
| 7-6. | New Parallel Plot | 58 |
| 7-7. | Add Data to Parallel | 58 |
| 7-8. | Replot Parallel | 58 |



| 7-9. | Manage Parallel | 59 |
|-----------|--|----|
| 7-10. | Zoom In | 59 |
| 7-11. | Manual Results | 60 |
| 7-12. | Peak Definition | 61 |
| 7-13. | X-Y Plot | 62 |
| 7-14. | Levich and other Plot | 64 |
| 7-15. | Semilog Plot | 67 |
| 7-16. | Special Plot | 68 |
| 7-17. | Graph Options | 69 |
| 7-18. | Color and Legend | 71 |
| 7-19. | Font | 72 |
| 7-20. | Copy to Clipboard | 73 |
| Chapter 8 | B. Data Processing Menu | 74 |
| 8-1. | Smoothing | 74 |
| 8-2. | Derivative | 75 |
| 8-3. | Integration | 76 |
| 8-4. | Semiintegral and Semiderivative | 76 |
| 8-5. | Interpolation | 77 |
| 8-6. | Baseline Fitting & Subtraction | 78 |
| 8-7. | Linear Baseline Correction | 79 |
| 8-8. | Data Point Removing | 80 |
| 8-9. | Data Point Modifying | 81 |
| 8-10. | Background Subtraction | 82 |
| 8-11. | Signal averaging | 82 |
| 8-12. | Mathematical Operation | 83 |
| 8-13. | Fourier Spectrum | 84 |
| Chapter 9 | . Analysis Menu | 85 |
| 9-1. | Calibration Curve | 85 |
| 9-2. | Standard Addition | 86 |
| 9-3. | Data File Report | 87 |
| 9-4. | Time Dependence | 89 |
| Chapter 1 | 0. Simulation Menu | 91 |
| 10-1. | Mechanism | 91 |
| 10-1- | 1. Potential and rate constants | 93 |
| 10-1- | 2. Concentration and Diffusion coefficients dialog box | 94 |
| 10-1- | 3. Surface concentration dialog box | 94 |
| 10-1- | 4. Concentration at equilibrium dialog box | 94 |



| 10-1 | -5. | Simulation variables dialog box | 95 |
|---------|-----|---|-----|
| 10-2. | CV | Simulate | 95 |
| 10-3. | CV | Fitting | 96 |
| Chapter | 11. | View Menu | 98 |
| 11-1. | Dat | a Information | 98 |
| 11-2. | Dat | a List | 99 |
| 11-3. | Εqι | lations | 99 |
| 11-4. | Clo | ck | 99 |
| 11-5. | Тос | Ibar Options | 100 |
| Chapter | 12. | Help Menu | 101 |
| 12-1. | Ind | ex | 101 |
| 12-2. | Abo | out ALS3325 | 101 |
| Chapter | 13. | Appendix | 102 |
| 13-1. | Cor | nnection with SEC2020 spectrometer system | 102 |
| 13-2. | Cor | nnection with RRDE-3A Rotating Ring Disk Electrode Device Ver.3.0 | 103 |
| 13-3. | Tro | ubleshooting | 104 |



Chapter 1. Introduction

1-1. Introduction

Model 3325 Bipotentiostat is a versatile electrochemical measurement device. The instrument incorporates a digital function generator, data sampling system, and potentiostat / bipotentiostat. The potential range is \pm 5.0 V and the current range is \pm 50 mA. It can measure currents greater than 100 pA. It is also possible to read the data measured with its predecessor, the Model 2325 Bipotentiostat.

The handheld size of the instrument allows it to be connected to a computer using a USB cable for communication and power supply. It is ideal for a field measurement and educational device in combination with a notebook PC.

It also has a remote function for the control of the RRDE-3A Rotating Ring Disk Electrode Apparatus and SEC2020 Spectrometer system.

1-2. Electrochemical technique

Model 3325 Bipotentiostat features the following techniques:

Sweep Technique

- CV : Cyclic voltammetry
- LSV : Linear sweep voltammetry

Amperometric Technique

- i-t : Amperometry

Other Techniques

- OCPT : Open circuit potential vs. time



1-3. Required system configuration

Table 1-3-1. Required system configuration

| Operating system | Windows 10 / 11 |
|--------------------|-------------------------|
| CPU | Intel Core i3 or higher |
| RAM | 8 G byte |
| Communication port | USB port |

1-4. Hardware specifications

Table 1-4-1. Hardware specifications

| Potential range | ±5 V |
|------------------------------|-------------------------------|
| Current range | ±50 mA |
| Output voltage | ±11 V |
| Sensitivity range | ±2.5 nA ~ ±0.05 A(8 ranges) |
| Input impedance | 1x10 ¹² Ω |
| Minimum potential resolution | 100 μV |
| Maximum sampling rate 16 bit | 16 bit @ 100 kHz |
| Background current | < 80 pA |
| Current resolution | 0.3 pA |
| Scanning rate CV | 1x10 ⁻⁶ ~ 80 V/sec |
| Interface | USB 2.0 |
| Power | supplied from USB port |
| Dimensions (W×D×H) | 170 × 110 × 25 mm |
| Weight | 0.35 kg |



Chapter 2. Basic Information

2-1. Update information

Please subscribe to our newsletter to receive the latest product information and important announcements regarding BAS products. We will keep you informed of the latest product information and various events. You can subscribe to the newsletter at the following URL https://www.als-japan.com/contact/contact.html

2-2. Specification changes

The specifications are subject to change without notice to improve the performance of the equipment.

2-3. Damage during transportation

If any part of the equipment is damaged during transportation, please contact us or our distributor and shipping company within 7 days of product arrival. We will replace the damaged product with a new one.

Keep the product in the cardboard box it was packed in so that the shipping company can check the damage.Please contact us or our local distributor to return the damaged product.

2-4. Product warranty

ALS Co., Ltd warrants products manufactured by the company are tested and inspected before shipping. In the unlikely event that a defect in the product is proved within the warranty period, we will replace or repair it free of charge.

The service does not cover damage caused by disassembly of the equipment or damage or corrosion caused by chemicals.

2-5. Service information

ALS provides a skilled service staff to solve your equipment oriented problems. For further details, please contact our sales representative. (sales@als-japan.com) Please inform us the full detail, and at this time inform the serial number.

2-6. Software warranty

If a software bug is identified, it will be fixed in the next version.



Chapter 3. Installation

3-1. Shipment Inspection

After unpacking the equipment carefully, check the package contents, referring to the list of accessories. Please keep the shipping box and packing material until you have fully tested the unit to be certain that no damage was occurred during shipment.

If any item is missing, please contact us or our local distributor. We will send you a replacement as soon as we confirm that we have the item in stock.



Figure 3-1-1. Accessories content

| | Product name | Quantity |
|---|--|----------|
| А | Device | 1 |
| В | USB cable | 1 |
| С | Cell cable | 1 |
| D | Ground wire (installed in the main unit) | 1 |

| Table | 3-1-1. | List of | accessories |
|-------|--------|---------|-------------|
|-------|--------|---------|-------------|

In addition to the above, software (sold separately) is required.



3-2. Software installation

Insert the USB memory with the specific software, which is sold separately, into the computer. Double-click "als3325-inst.exe" in the USB memory to start the program installation. Click "Yes" to create a shortcut on the desktop and the als3325 folder on the C drive.

| als3325en Program Setup | \times |
|-------------------------------|----------|
| START als3325en Program Setup | |
| Yes No | |

Figure 3-2-1. Software installation

Next, check the version of Windows. Type "winver" in "Search" on the taskbar and execute the command. Windows version information will be displayed.

|--|

Figure 3-2-2. Taskbar

Install the USB driver according to the version of the PC to be used.

3-2-1. Windows 11 (x64), Windows 10 (1803) (x64, x86) or later

Open the "CP210x_Universal_Windows_Driver" folder in the USB memory. Place the cursor on silabser.inf, right-click, select "Show More Options" to open the pull-down menu, and run the installation.



Figure 3-2-3. Installation execution

3-2-2. Windows 10 (1803) (x64, x86) or earlier

Open the "USB_Driver" folder in the als3325 folder on the C: drive created in step 3-2. Double-click "CP210xVCPInstaller_x64.exe" (64bit version) or

CP210xVCPInstaller_x86.exe" (32bit version) to install the software sequentially. Doubleclick the shortcut created on the desktop to launch the software.



Figure 3-2-4. shortcut



3-3. Equipment setup

Plug the supplied USB cable into the USB port on the rear side of the device and connect it to the computer. Power is supplied from the USB cable, so there is no need to connect the power cable. Connect the ground wire to Ground.



Figure 3-3-1. Back of the equipment

Connect the cell cable to the cell cable terminal. This completes the equipment preparation.



Figure 3-3-2. Cable connection (left) and cell cable connection example (right)

Connect the cell cable to the corresponding electrode according to Table 3-3-1. Connect the cell cable after checking the operation as described in 3-4.

| Alligator clip color/ | Electrode |
|-----------------------|---------------------|
| Green | Working electrode |
| White | Reference electrode |
| Red | Counter electrode |
| Yellow | Working electrode 2 |

Table 3-3-1. Electrodes corresponding to alligator clips



3-4. Operation check

Make sure the computer is connected to the device and start the software.

Clicking the icon launches the system setup. First, select the power supply frequency according to the operating environment.

Next, click the "Communication Port Setting" button and confirm that communication with the device is performed correctly.

| System Setup | | × |
|-------------------------------|----------------------------------|--|
| Communication Port | | OK |
| C Com 1 C Com 2 | Line Frequency | Cancel |
| C Com 3 C Com 4 | C 50 Hz | Help |
| C Com 5 C Com 6 | Potential Axis Positive Left | Set Com Port |
| C Com 7 C Com 8 C Com 9 | C Positive Right | |
| C Com 10 C Com 11 | Current Axis | ADC Calibration |
| C Com 12 | C Positive Down | Data Length: 256K 💌 |
| Com Port Speed | Current Polarity | Save retrieve data during run |
| C Standard | Cathodic Positive | Auto data save after run |
| Fast | C Anodic Positive | Present Data Override Warning |
| - Windows | | 🔲 Save Text File As Well |
| English | C Oriental | Check Com port while idling Automatically detect Com port |

Figure 3-4-1. System Setup

For details, refer to 5-3 System setup. Next, select "Setup" - "Hardware - Test" from the toolbar.

| p Control | | Graphic | | | | |
|-------------------|--------------------|------------|--|--|--|--|
| <u>T</u> echnique | | | | | | |
| Parameters | | | | | | |
| <u>S</u> ystem | | | | | | |
| <u>H</u> a | rdware Tes | t | | | | |
| | <u>P</u> ai Sys | Parameters | | | | |

Figure 3-4-2. Toolbar

If the Link Failed message appears, communication is not working properly. Check the cable connection and communication port. Also, make sure that the USB driver described on page 5 is installed properly.

When performing a hardware test with the cell cable connected to the electrode, sample solution, etc. an error may occur. Please perform the test with the cable disconnected.





Figure 3-4-3. Link Failed

If all test results are OK, the device is operating properly.

| Hardware Test Results | | × |
|---|---|---------|
| Digital circuitry test results: ROM version: 20.01 ROM revision date: 18–Jan–2020 Potential offset test OK. Channel 1 offset test OK. | ^ | UK Help |
| Channel 2 offset test OK. | | |
| Sensitivity test OK. | | |
| Analog circuitry test OK. | | |
| | | |
| | | |
| | | |
| | ~ | |

Figure 3-4-4. Hardware Test

For the check of the operation in more detail, use the internal dummy cell installed in the device. From "Control" - "Cell", check the "Test with internal dummy cell" box. The internal dummy cell is a 1 M Ω resistor.

| Control Level of Stir Line OK | Cell Control | × |
|-------------------------------------|---|--|
| Cell On Collect | Active High C Active Low Immediate Stir Stir Time (sec) | Cancel Help ✓ Stir Between Runs ✓ Purge Between Runs ✓ Stir and Purge Simultaneously ✓ Test with Internal Dummy Cell Cell On Between Runs ✓ Return to Init E after Run SMDE Drops Before Run 0 Mercury Drop Collection Number of Drops |

Figure 3-4-5. Test with internal dummy cell



Select CV from the technique and set the parameters. Set the potential range from 0.5 V

to -0.5 V and the sensitivity (in this case, set to 1e-6 A / V). Click the icon and perform the dummy cell test. As a measurement result, a straight line (large slope) passing through the zero point is displayed.

| | | Parameters for Cyclic Voltammetry | > |
|---|--|---|--|
| Electrochemical Techniques Technique Selection (type 'cv' to select CV, etc.): | Х | Init E (V) 0.5 High E (V) 0.5 Low E (V) -0.5 Initial Scan Polarity Negative ▼ Scan Rate (V/s) 0.1 Sweep Segments 2 Sample Interval (V) 0.001 Quiet Time (sec) 2 Sensitivity (A/V) 1.e-006 Electrode 2 | OK Cancel Help |
| CV - Cyclic Volkammetry LSV - Linear Sweep Volkammetry H - Amperometric H Curve OCPT - Open Circuit Potential - Time | Cancel Help Tech Help Show on startup Show for new documents Use current tech on startup Always confirm parameters | Potential (V) | Off Constant E C Scan C Diff Scan |
| View as tree | Run prompt after params | | |

Figure 3-4-6. Technique selection





Figure 3-4-8. CV measurement example using an internal dummy cell



When checking the second working electrode, prepare two 100 K Ω resistors and use them as dummy cells. Connect the reference electrode (white) and the counter electrode (red) to one side of the two resistors, and connect the working electrode (green) and the second working electrode (yellow) to the other side of each resistor, respectively.



Figure 3-4-9. Connection to a resistor

Select CV from technique and set the parameters.

Set the potential range to 0.5 V to -0.5 V, the sensitivity to 1e-5 A / V, and electrode 2 to Scan. Click on the icon to perform the measurement.



Figure 3-4-10. CV measurement example using a 100 K Ω resistor



3-5. Measurement preparation

After the operation check is complete, prepare to start the measurement.

First, select the technique to be used for the measurement by clicking on the icon \square . See 5-1 for details on each technique.

| Electrochemical Techniques | × |
|--|-------------------------------|
| Technique Selection (type 'cv' to select CV, etc.): | ОК |
| CV - Cyclic Voltammetry | Cancel |
| CV - Cyclic Voltammetry | Help |
| LSV - Linear Sweep Voltammetry i-t - Amperometric i-t Curve | Tech Help |
| OCPT - Open Circuit Potential - Time | Show on startup |
| | Show for new documents |
| | 🔲 Use current tech on startup |
| | Always confirm parameters |
| | 🔲 Run prompt after params |
| 🔽 View as tree | |

Figure 3-5-1. Technique selection

Next, click on the icon 💷 to set the parameters. Figure 3-5-2 Shows the parameter setting dialog box when cyclic voltammetry is selected. Enter the parameters required for the measurement. See 5-2 for details on each parameter.

| Init E (V) | OK |
|----------------------------------|--------------|
| High E (V) | Cancel |
| Low E (V) | Help |
| Initial Scan Polarity Negative 💌 | |
| Scan Rate (V/s) | |
| Sweep Segments | |
| Sample Interval (V) 0.001 | |
| Quiet Time (sec)2 | |
| Sensitivity (A/V) 1.e-006 💌 🚽 | |
| Electrode 2 | |
| Potential (V) | ○ Off |
| | 🔿 Constant E |
| | 🔿 Scan |
| Sensitivity (A/V) 1.e-005 🛛 👻 | O Diff Scan |

Figure 3-5-2. Parameter setting

When it is ready to start measurement, click the icon **b** to start the measurement.



3-6. Cell control terminal

The cell control terminals on the rear of the Model 3325 bipotentiostat can be used to synchronize and control external devices. Use the dedicated cable to connect the device.

For connection with external devices, refer to 13-1. Connection with SEC2020 spectrometer system and 13-2. Connection with RRDE-3A Rotating Ring Disk Electrode Device Ver.3.0.



Figure 3-6-1. Cell control terminal

| Pin # | Function | Pin # | Function | Pin # | Function |
|-------|--------------------------|-------|----------|-------|------------------------------|
| 1 | i1 (+2.5V initial value) | 6 | - | 11 | Trigger In |
| 2 | i2 (+2.5V initial value) | 7 | - | 12 | Stir |
| 3 | E (+2.5V initial value) | 8 | - | 13 | Purge |
| 4 | RDE(0-10V) | 9 | - | 14 | Spectromter Control Knock |
| 5 | Ground | 10 | - | 15 | - |

Table 3-6-1. Function of each pin

Note

- 1. If the remote control is not used, do not connect anything to the terminals.
- 2. If the default current polarity is Cathodic Positive, the formula is:
 - i1 (A) = (2.5 Pin 1 output voltage)(V) x sensitivity 1 (A/V)
 - i2 (A) = (2.5 Pin 2 output voltage)(V) x sensitivity 2 (A/V) x 5

E(V) = 2.5 - Pin 3 output voltage(V)

When the current polarity is Anodic Positive, the current value is inverted



Chapter 4. File Menu

The following are the functions that can be selected from the file menu on the toolbar.

4-1. Open

Click "File" - "Open" to open a saved file.

| | | | 🚰 Open | | | | | × |
|------|---------------------|-------------|--|--------------------------------|----------|-------|-------------------------------|----|
| | | | \leftarrow \rightarrow \checkmark \uparrow \square \rightarrow This PC \rightarrow v | win10e (C:) > als3325en > Data | | ٽ ~ | 🔎 Search Data | |
| | | | Organise 🔻 New folder | | | | . · · | • |
| | | | Name | Date modified | Туре | Size | | ^ |
| File | Setup Control | Graphics Da | acv1.bin | 2018/03/08 8:00 | BIN File | 3 KB | | |
| | | | acv2.bin | 2018/03/08 8:01 | BIN File | 3 KB | | |
| D | <u>N</u> ew | Ctrl+N | be1.bin | 2018/03/08 7:52 | BIN File | 4 KB | | |
| Ê | Open | Ctrl+O | a1.bin | 2018/03/08 7:55 | BIN File | 4 KB | | |
| | <u>open</u> | Cuito | cc1.bin | 2018/03/08 7:56 | BIN File | 10 KB | | |
| | Close | Ctrl+W | cp1.bin | 2018/03/08 8:15 | BIN File | 9 KB | | |
| - | - | | 📄 cv1.bin | 2018/03/08 7:52 | BIN File | 6 KB | | |
| | Save As | Ctrl+S | 📄 cv2.bin | 2018/03/08 7:52 | BIN File | 6 KB | | |
| | Delete | | 📄 cv3.bin | 2018/03/08 7:52 | BIN File | 8 KB | | |
| | Delete | | cv4.bin | 2018/03/08 11:43 | BIN File | 10 KB | | |
| | Retrieve | | ddpa1.bin | 2018/03/08 8:07 | BIN File | 5 KB | | |
| | | | dnpv1.bin | 2018/03/08 7:58 | BIN File | 2 KB | | |
| | Lindata instrumenti | Dragram | dpa1.bin | 2018/03/08 8:06 | BIN File | 4 KB | | |
| | Update Instrument | riogram | dpv1.bin | 2018/03/08 7:57 | BIN File | 3 KB | | ~ |
| | List Data File | | File <u>n</u> ame: cv1 | .bin | | ~ | CHI binary data files (*.bin) | ~ |
| | Convert to Text | | | | | | <u>O</u> pen Cance | el |

Figure 4-1-1. Toolbar (left), open file (right)

From the dialog box, select the file to open. .bin", ".txt", and ".csv" format files can be opened. Also, by setting the file format to "All Files (*.*)", the data measured by the model 2325 bipotentiostat can be read.

The same operation can be performed by clicking on the icon.

4-2. Save As

Click "File" - "Save As" to save the file as.

| | | | Save As | | | | × |
|------|------------------------|-------------|---|-----------------------------|----------|-------|-------------|
| | | | \leftarrow \rightarrow \checkmark \uparrow \square \rightarrow This PC \rightarrow De | esktop > 3325-220711 > Data | | √ 5 √ | Search Data |
| | | | Organise 🔻 New folder | | | | E • 🕐 |
| | | | Name | Date modified | Туре | Size | |
| File | Setup Control | Graphics Da | Cv1.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| The | Setup Control | Graphics De | Cv2.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| ß | New | Ctrl+N | cv3.bin | 2020/10/23 4:55 | BIN File | 6 KB | |
| | - | | cv4.bin | 2020/10/23 5:44 | BIN File | 9 KB | |
| 1 | <u>O</u> pen | Ctrl+O | it1.bin | 2020/10/23 4:57 | BIN File | 3 KB | |
| | Close | Ctrl+W | Lsv1.bin | 2020/10/23 4:55 | BIN File | 4 KB | |
| | - | | Isv100rpmtest.bin | 2022/04/25 21:28 | BIN File | 7 KB | |
| | Save <u>A</u> s | Ctrl+S | Isv1500rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| | Delete | | Isv5000rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| | Delete | | Mcv.bin | 2020/10/23 5:47 | BIN File | 6 KB | |
| | Retrieve | | Ocpt1.bin | 2020/10/23 4:58 | BIN File | 4 KB | |
| | | | | | | | |
| | Update Instrument | Program | File name: CV.bin | | | | ~ |
| | | | Save as type: Data Files (*.bin) | | | | ~ |
| | <u>L</u> ist Data File | | | | | _ | |
| | Convert to Text | | Hide Folders | | | L | Save Cancel |

Figure 4-2-1. Toolbar (left), Save file (right)

From the dialog box that appears, specify the destination for saving the data, enter a file name, and save the file. If you use a file name that already exists, a warning message will be displayed.

The same operation can be performed by clicking on the icon.



4-3. Delete

Click "File" - "Delete" to delete a file.

| | | | 🞇 File Deletion | | | | × |
|----------|---------------------------|-------------|--|------------------------------|----------|-------|------------------------|
| | | | \leftarrow \rightarrow \checkmark \Uparrow This PC \rightarrow | Desktop > 3325-220711 > Data | | ې ق 🗸 | Search Data |
| | | | Organise 🔻 New folder | | | | E 🕶 🔲 💡 |
| | | | Name | Date modified | Туре | Size | |
| File | Setup Control | Graphics Da | Cv1.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| ß | New | Ctrl+N | Cv2.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| _ | <u>n</u> en | Current | cv3.bin | 2020/10/23 4:55 | BIN File | 6 KB | |
| 2 | Open | Ctrl+O | cv4.bin | 2020/10/23 5:44 | BIN File | 9 KB | |
| | Class | Ctrl+W | it1.bin | 2020/10/23 4:57 | BIN File | 3 KB | |
| | <u>C</u> lose | Ctri+vv | Lsv1.bin | 2020/10/23 4:55 | BIN File | 4 KB | |
| | Save As | CtrI+S | Isv100rpmtest.bin | 2022/04/25 21:28 | BIN File | 7 KB | |
| - | | | Isv1500rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| | <u>D</u> elete | | Isv5000rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| | Retrieve | | Mcv.bin | 2020/10/23 5:47 | BIN File | 6 KB | |
| | Retrieve | | Ocpt1.bin | 2020/10/23 4:58 | BIN File | 4 KB | |
| | <u>U</u> pdate Instrument | Program | | | | | |
| | <u>L</u> ist Data File | | File name: | bin | | ∽ Da | ta Files (*.bin) 🗸 🗸 🗸 |
| | Convert to Text | | | | | | Open Cancel |



From the dialog box, select the file to be deleted.



Figure 4-3-2. Deleting file

A confirmation message will be displayed, and click "Yes" to delete the file.



4-4. List Data File

From "File" - "List Data File", list the experimental conditions, results, and numerical values of the data.

| | | | 🕌 List Data File | | | | | | | × |
|----|------------------------|-------------|--|-------------------|-------------------------------------|---------------------------|---------------|--------------------|--------|---|
| | | | $\leftarrow \rightarrow \checkmark \uparrow$ | - > This PC > Des | ktop → 3325-220711 → Data | | ٽ ~ | ල 🔎 Search Data | | |
| | | | Organise 💌 | New folder | | | | | | • |
| | | | Name | ^ | Date modified | Туре | Size | | | |
| Fi | le Setup Control | Graphics Da | Cv1.bin | | 2020/10/23 4:54 | BIN File | 6 KB | | | |
| Г | New | Ctrl+N | Cv1.txt | | 2020/11/05 20:54 2020/10/23 4:54 | Text Document BIN File | 18 KB 6 KB | | | |
| | <u>O</u> pen | Ctrl+O | 🗋 cv3.bin | | 2020/10/23 4:55 | BIN File | 6 KB | | | |
| | <u>C</u> lose | Ctrl+W | 📄 cv4.bin | | 2020/10/23 5:44 | BIN File | 9 KB | | | |
| | Save <u>A</u> s | Ctrl+S | | | | | | | | |
| | <u>D</u> elete | | | | | | | | | |
| | Retrieve | | | | | | | | | |
| | Update Instrument | Program | | | | | | | | |
| | <u>L</u> ist Data File | | | File name: CV* | | | ~ | Data Files (*.bin) | | ~ |
| | Convert to Text | | | | | | | Open | Cancel | |

Figure 4-4-1. Toolbar (left), Select File (right)

Select a file from the dialog box. The selectable files are in ".bin" format.

| Data List | | × |
|--|---|----|
| Dct: 22,2020 14:54:53 Cyclic Voltammetry File: c:\als1242cx\data\cv1.bin Data Source: Experiment Instrument Model: CHI1225C Header: 0.5 mM Ferricyanide in 0.4M KCI solution Note: | ^ | OK |
| $ \begin{array}{l} \mbox{Init E (V) = 0.5} \\ \mbox{High E (V) = 0.5} \\ \mbox{Low E (V) = 0} \\ \mbox{Init P/N = N} \\ \mbox{Scan Rate (V/s) = 0.1} \\ \mbox{Scampent = 2} \\ \mbox{Sample Interval (V) = 0.001} \\ \mbox{Quiet Time (sec) = 2} \\ \mbox{Sensitivity (\Delta / V) = 1e-5} \end{array} $ | | |
| Results: Channel 1: Segment 1: Ep = 0.197V jp = 3.643e-6A Ah = 2.289e-6C A | | |
| Segment 2: Ep = 0.263V ip = -3.164e-6A Ah = -1.980e-6C | | |
| Potential/V, Current/A 0.500, -3.245e-7 0.493, -2.569e-7 0.498, -2.242e-7 0.497, -2.028e-7 0.496, -1.795e-7 | * | |
| < >> | | |

Figure 4-4-2. List of data files

The data is displayed in text format. The display format can be changed in "Text File Format". To display the data opened in the software in text format, select "View" - "Data List".



4-5. Convert to Text

Convert a binary file (.bin) to a text file (.txt) from "File" - "Convert to Text".

| File | Setup Control | Graphics Di | 🞇 File Conversion | | | | | |
|------|------------------------|-------------|--|-------------------------------|----------|------|----------------------|----|
| | Setup Control | Graphics Di | $\leftarrow \rightarrow \ ' \uparrow \blacksquare$ > This PC > V | Vindows (C:) → als3325 → Data | | | ✓ Ö シ Search Data | |
| ß | New | Ctrl+N | Organize 🔻 New folder | | | | III 🕶 🔲 | |
| Ê, | Open | Ctrl+O | Name | Date modified | Туре | Size | | |
| | Close | Ctrl+W | Cv.bin | 7/25/2022 3:36 PM | BIN File | 2 KB | | |
| | Close | Cult+vv | Isv100rpmtest.bin | 4/25/2022 9:28 PM | BIN File | 7 KB | | |
| | Save As | Ctrl+S | Isv1500rpmtest.bin | 4/25/2022 3:14 PM | BIN File | 7 KB | | |
| | Save As | Cuita | Isv5000rpmtest.bin | 4/25/2022 3:14 PM | BIN File | 7 KB | | |
| | Delete | | Mcv.bin | 10/23/2020 5:47 AM | BIN File | 6 KB | | |
| | Deleten | | cv4.bin | 10/23/2020 5:44 AM | BIN File | 9 KB | | |
| | Retrieve | | TPA2.BIN | 10/23/2020 5:42 AM | BIN File | 3 KB | | |
| | Retrieve | | TPA1.BIN | 10/23/2020 5:40 AM | BIN File | 8 KB | | |
| | | | DPA1.BIN | 10/23/2020 5:37 AM | BIN File | 5 KB | | |
| | Update Instrument | Program | DNPV.BIN | 10/23/2020 5:36 AM | BIN File | 3 KB | | |
| | | - | DDPA1.BIN | 10/23/2020 5:35 AM | BIN File | 8 KB | | |
| | | | Shacv2.bin | 10/23/2020 5:05 AM | BIN File | 3 KB | | |
| | <u>L</u> ist Data File | | Shacv1.bin | 10/23/2020 5:03 AM | BIN File | 3 KB | | |
| _ | Commentation Trends | | Acv2.bin | 10/23/2020 5:01 AM | BIN File | 3 KB | | |
| | Convert to Text | | Acv1.bin | 10/23/2020 5:00 AM | BIN File | 3 KB | | |
| | Text File Format | | File name: Cv.b | in | | | ✓ Data Files (*.bin) | |
| | | | | | | | | |
| | Import Text File | | | | | | Open Cance | :I |



| | Windows (C:) > als3325 > Data | | | ~ Ū | 🔎 Search Data | |
|-----------------------|-------------------------------|----------|------|-----|-----------------|--------|
| Organize 🔻 New folder | | | | | (iii • | |
| lame | Date modified | Туре | Size | | | |
| 🕅 Cv.txt | 9/27/2022 1:11 PM | TXT File | 1 KB | | | |
| Cv.bin | 7/25/2022 3:36 PM | BIN File | 2 KB | | | |
| CV.sim | 6/6/2022 11:53 AM | SIM File | 1 KB | | | |
| Isv100rpmtest.bin | 4/25/2022 9:28 PM | BIN File | 7 KB | | | |
| Isv1500rpmtest.bin | 4/25/2022 3:14 PM | BIN File | 7 KB | | | |
| Isv5000rpmtest.bin | 4/25/2022 3:14 PM | BIN File | 7 KB | | | |
| Mcv.bin | 10/23/2020 5:47 AM | BIN File | 6 KB | | | |
| cv4.bin | 10/23/2020 5:44 AM | BIN File | 9 KB | | | |
| TPA2.BIN | 10/23/2020 5:42 AM | BIN File | 3 KB | | | |
| TPA1.BIN | 10/23/2020 5:40 AM | BIN File | 8 KB | | | |
| DPA1.BIN | 10/23/2020 5:37 AM | BIN File | 5 KB | | | |
| DNPV.BIN | 10/23/2020 5:36 AM | BIN File | 3 KB | | | |
| DDPA1.BIN | 10/23/2020 5:35 AM | BIN File | 8 KB | | | |
| Shacv2.bin | 10/23/2020 5:05 AM | BIN File | 3 KB | | | |
| Shacv1.bin | 10/23/2020 5:03 AM | BIN File | 3 KB | | | |
| File name: Cv. | bit | | | ~ | All Files (*.*) | ` |
| | | | | | Open (| Cancel |

Figure 4-5-2. Open the file

From the dialog box, select the file to convert to text. The converted file will be saved in the same folder as the selected file. The display format can be changed in "Text File Format".

Text files can be read by spreadsheets such as Excel.



4-6. Text File Format

From "File" - "Text File Format", the display format of the text file generated by "4-5 Convert to Text" can be changed.

| | | | Text File Format X |
|----------|------------------------|-------------|--|
| | | | ✓ Memo |
| File | Setup Control | Graphics Da | DigiSim Format for CV and LSV Data |
| ß | <u>N</u> ew | Ctrl+N | Excel 3D Format for SECM Data |
| 2 | Open | Ctrl+O | Nanoscope Header for SECM data |
| | <u>C</u> lose | Ctrl+W | CP for Battery Test Compare with previous point: 0 |
| | Save <u>A</u> s | Ctrl+S | ☐ 3 column impedance data |
| | Delete | | Segment Separator |
| | Retrieve | | CV Selected Segments From 1 To 2 |
| | Update Instrument | Program | Charge |
| | <u>L</u> ist Data File | | Charge Reset Every Segment |
| | Convert to Text | | 🗖 Time |
| | Text File Format | | Mott-Shottky |
| | Import Text File | | |

Figure 4-6-1. Toolbar (left), Text file format (right)

- Memo

Select to display date, time, technique, label, note, etc.

- Parameters

Select to display experimental parameters.

- Results

Select to display experimental results (e.g., peak or waveform potential, current, area). Peak settings are defined from "Graphics" - "Peak Definition".

- Numerical Data

Select to display numerical data.

CV selected segments From...To...

By default, all CV data is converted to a text file. This option allows selecting specific segment data to be converted.

- Time

Saves time in a text file for time-based experimental data.

· XY Separator

Set the separator (comma, tab, space, line spacing) used to separate data.

| Comma | Х,Ү |
|--------------|--------|
| Tab | Х Ү |
| Space | ХҮ |
| Line spacing | X Y |



Significant digits

Set the number of significant digits. The default is 4 digits, but can be changed from 3 to 10 digits. The larger number of significant digits, the larger the file size.

- Data point interval

Set the interval (potential resolution) of the output data. File size can be reduced, but some data may be lost.

- Digisim format for CV and LSV data

Used only for CV and LSV data. When selected, text files are output in a format that can be read directly by DigiSim.

Here are the specific steps for text conversion. First, select "File" - "Text File Format" to set the display format of the text file.

| Text File Format | | × |
|---|---|----------------------|
| I ✓ Memo I ✓ Parameter: I ✓ Results I ✓ Numeric Data | XY Separator © Comma © TAB © Space © Linefeed | OK Cancel Help |
| Number of Significant Figu | ıres 4 | |
| Data Point Interval | 1 | |
| DigiSim Format for CV | and LSV Data | |
| Excel 3D Format for S | ECM Data | |
| Nanoscope Header fo | | |
| | Compare with previous (| point: 0 |
| 3 column impedance (| data | |
| Segment Separator | - | |
| CV Selected Segment | s From 1 | To 2 |
| Charge | | |
| Charge Reset Every S | egment | |
| Time | | |
| Mott-Shottky | | |

Figure 4-6-2. Text file format

Select the required item from the displayed text file format dialog. For example, when exporting to Excel, specifies the data separator from XY separator, and set the significant digits (number of digits) and data point i nterval (potential resolution).

To change the contents of the graph display, go to "Graphics" - "Graph Options" to make the setting. Peak settings are defined from "Graphics" - "Peak definition".

Select "File" - "Convert to Text" and select the data to be converted from the dialog box. The data will be automatically converted to text format. The converted text file will be saved in the same folder as the selected file.



| → * ↑ - > This PC > | Desktop > 3325-220711 > Data | | ٽ ~ | 🔎 Search Data | |
|-----------------------|------------------------------|----------|------|--------------------|--|
| Irganise 🔻 New folder | | | | . · · | |
| lame | Date modified | Туре | Size | | |
| Cv1.bin | 2020/10/23 4:54 | BIN File | 6 KB | | |
| Cv2.bin | 2020/10/23 4:54 | BIN File | 6 KB | | |
| cv3.bin | 2020/10/23 4:55 | BIN File | 6 KB | | |
| cv4.bin | 2020/10/23 5:44 | BIN File | 9 KB | | |
| it1.bin | 2020/10/23 4:57 | BIN File | 3 KB | | |
| Lsv1.bin | 2020/10/23 4:55 | BIN File | 4 KB | | |
|] Isv100rpmtest.bin | 2022/04/25 21:28 | BIN File | 7 KB | | |
|] Isv1500rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | | |
|] Isv5000rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | | |
|] Mcv.bin | 2020/10/23 5:47 | BIN File | 6 KB | | |
| Ocpt1.bin | 2020/10/23 4:58 | BIN File | 4 KB | | |
| | | | | | |
| | | | | | |
| File name: Cv | 1.bin | | ~ | Data Files (*.bin) | |
| | | | | Open Cancel | |

Figure 4-6-3. Select a file

Figure 4-6-4 Shows the text-converted data loaded in Notepad.



Figure 4-6-4. Text conversion data



4-7. Import Text File

Import the text file from "File" - "Import Text File".

| File | Setup Control | Graphics I |
|----------|-------------------|------------|
| ß | New | Ctrl+N |
| B | <u>O</u> pen | Ctrl+O |
| | <u>C</u> lose | Ctrl+W |
| | Save <u>A</u> s | CtrI+S |
| | <u>D</u> elete | |
| | Retrieve | |
| | Update Instrument | Program |
| | List Data File | |
| | Convert to Text | |
| | Text File Format | |
| | Import Text File | |

Figure 4-7-1. Toolbar (left), File format (right)

This command can import only one text file at a time. Use "Open" to import multiple files. Also, by setting the file format to "*.DY20", it is possible to import the data measured by the Model 2325 Bipotentiostat.

4-8. Print

Data can be printed from "File" - "Print".



Figure 4-8-1. Toolbar

The data displayed on the software is printed. To customize the contents of the graph display, go to "Graphics" - "Graph Options" to make the setting.

The same operation can be performed by clicking on the icon.



4-9. Print Multiple Files

Select "File" - "Print Multiple Files" and specify the data to be printed.

| File | | Graphics Da Ctrl+N | 🞇 Multiple File Print | | | | · · · · · · · · · · · · · · · · · · · |
|------|----------------------|-----------------------|-----------------------|--------------------------------|----------|------|---------------------------------------|
| | New | | ← → × ↑ 🔒 > This PC | > Desktop > 3325-220711 > Data | | 5 v | 🔎 Search Data |
| Ē | Open | Ctrl+O | Organise 🔻 New folder | | | | |
| | <u>C</u> lose | Ctrl+W | | | | | 8== • • |
| | Cove Ar | Ctrl+S | Name | Date modified | Туре | Size | |
| | Save <u>A</u> s | Cui+5 | Cv1.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| | Delete | | Cv2.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| | - | | 🗋 cv3.bin | 2020/10/23 4:55 | BIN File | 6 KB | |
| | Retrieve | | cv4.bin | 2020/10/23 5:44 | BIN File | 9 KB | |
| | | | 📑 it1.bin | 2020/10/23 4:57 | BIN File | 3 KB | |
| | Update Instrument | Program | Lsv1.bin | 2020/10/23 4:55 | BIN File | 4 KB | |
| | | - | Sv100rpmtest.bin | 2022/04/25 21:28 | BIN File | 7 KB | |
| | List Data File | | Isv1500rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| | List Data The | | Isv5000rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| | Convert to Text | | Mcv.bin | 2020/10/23 5:47 | BIN File | 6 KB | |
| | | | Ocpt1.bin | 2020/10/23 4:58 | BIN File | 4 KB | |
| | Text File Format | | | | | | |
| | Import Text File | | | | | | |
| 3 | Print | Ctrl+P | File name: | Lsv1.bin" "Cv1.bin" "it1.bin" | | ~ | Data Files (*.bin) ~ |
| | Print Multiple Files | | | | | | Open Cancel |

Figure 4-9-1 Toolbar (left), Select file (right)

From the dialog box, select the file to be printed. Multiple files can be selected by holding down the Ctrl key while selecting.

To customize the display contents, go to "Graphics" - "Graph Options" to make the setting.

4-10. Print Setup

From "File" - "Print Setup", configure printer setting. Set the printer and paper in the displayed dialog box.



Figure 4-10-1. Toolbar



4-11. Exit

Select "File"-"Exit" to exit the software.



Figure 4-11-1. Toolbar

Some system information will be saved, such as file directories, system setup, control status, macro commands, data processing options, simulation options, graphics options, colors, fonts, etc.



Chapter 5. Setup Menu

The following is a list of functions that can be selected from the setup menu on the toolbar.

5-1. Technique Selection

Select the electrochemical technique from "Setup" - "Technique". The dialog box shown in Figure 5-1-1 is displayed. Select the technique for the measurement.

| Electrochemical Techniques | | × |
|--|------------------|----------------|
| Technique Selection (type 'cv' to select CV, etc.): | 1 | OK |
| CV - Cyclic Voltammetry | | Cancel |
| CV - Cyclic Voltammetry | ī | Help |
| LSV - Linear Sweep Voltammetry i-t - Amperometric i-t Curve | | Tech Help |
| OCPT - Open Circuit Potential - Time | 🔲 Show on start | up |
| | 🔲 Show for new | documents |
| | 🗌 Use current te | ech on startup |
| | 🗌 Always confir | n parameters |
| | 🔲 Run prompt a | fter params |
| 🗌 View as tree | | |

Figure 5-1-1. Technique selection

The same operation can be performed by clicking on the icon. ${\mathbb T}$



5-1-1. Sweep technique (LSV, CV)

The Linear Sweep Voltammetry (LSV) the potential is swept linearly from initial to final potential at a constant scan rate. The current is monitored as a function of the applied potential. A simple LSV potential waveform is shown in Figure 5-1-2.





Cyclic voltammetry (CV) is a more versatile version of LSV. This technique reverses the scan direction when the final potential is reached and scan again in the opposite direction within the same potential range. The products of the electrochemical reaction generated by the forward scan can be investigated in the reverse scan. This feature is one of the main reasons why the CV technique is widely used.



Figure 5-1-3. CV potential waveform

In CV, the potential can be repeated many times within the same range. Three potential variables are required: the initial potential, and two switching potentials, high and low potential, in which the direction of the scan is reversed.

Figure 5-1-4 Shows the simplest I-E curve for CV. The asymmetry of the curve is caused by mass transfer due to diffusion. There are many variables that affect the shape of this curve. For example, slow heterogeneous electron transfer, instability of oxidized or reduced species, adsorption, etc. If the electron transfer rate in the heterogeneous system is faster than the experimental timescale and either oxidized or reduced species are stable, the



redox process is said to be electrochemically reversible. The standard redox potential of such a system is the average of the two peak potentials (Epa and Epc), and the difference between the peak potentials is 57/n (mV), where n is the number of electrons transferred per mole.





In the CV of the reversible process, the peak current is expressed by the Randles-Sevcik equation.

| quiv/mol |
|--|
| ffusion coefficient (cm ² /s) |
| an rate (V/s) |
| |

In the reversible process, ip is proportional to concentration C and scan rate $v^{1/2}$. There are many variables that affect the shape of the CV curve. The slow electron transfer rate increase the peak potential separation (Δ Ep), and the electron transfer rate constant can be calculated by examining the change in Δ Ep with scan rate. The uncompensated resistance between the working electrode and the comparative electrode also increases Δ Ep. The effect of uncompensated resistance can be reduced or eliminated by iR compensation by the electronics.

Another use of CV is to study the reaction of product species of electrode reactions.

The reactivity of the product species produced by the forward scan can be examined in the reverse scan and subsequent scans. A qualitative assessment of the reaction rate can be obtained by varying the scan rate. Due to its simplicity and speed, CV is often used as a first step in the investigation of redox systems and is considered a very powerful technique for qualitative analysis of reaction kinetcs and mechanisms. The background (capacitive) current that appears in the CV and LSV limit their usefulness as a quantitative analytical tool. On the other hand, LSV is an effective technique for the detection of trace metals by stripping voltammetry.



5-1-2. Amperometric technique (i-t)

The amperometric technique measures current as a function of time. Genarally, such techniques are used for current titration, amperometric sensors, flow cells, etc. The simplest potential waveform is the fixed potential. The i-t waveform and a typical current response are shown in Figure 5-1-5 and Figure 5-1-6, respectively.



Figure 5-1-5. i-t potential waveform



Figure 5-1-6. Typical current response of i-t



5-1-3. Open circuit potential-time (OCPT)

Open Circuit Potential - Time (OCPT) measures the potential difference between a working electrode and a reference electrode without applying current to the cell. The potential difference is recorded as a function of time. Since the counter electrode is not connected to an external cell, no current flows to the working electrode except for a bias current at the pico ampere level. The OCPT waveform is shown in Figure 5-1-7.



Figure 5-1-7. OCPT waveform



5-2. Parameter Setting

Set the experimental parameters from "Setup" - "Parameters". The parameters to be set are different for each technique.

| Parameters for Cyclic Voltammetry | × |
|--|--|
| Init E (V) □ High E (V) □ Low E (V) □ Initial Scan Polarity □ Initial Scan Polarity Negative ▼ Scan Rate (V/s) □.1 Sweep Segments 2 Sample Interval (V) □.001 Quiet Time (sec) 2 Sensitivity (A/V) 1.e-005 ▼ | OK Cancel Help |
| Potential (V)0 Differential E (V) 0 Sensitivity (A/V) 1.e-005 💌 | Off Constant E Scan Diff Scan |

Figure 5-2-1. Parameter setting

The same operation can be performed by clicking on the icon.



5-2-1. Cyclic voltammetry parameters

| Init E (V) | OK |
|----------------------------------|-------------|
| High E (V) | Cancel |
| Low E (V) 0 | Help |
| Initial Scan Polarity Negative 💌 | |
| Scan Rate (V/s) | |
| Sweep Segments | |
| Sample Interval (V) 0.001 | |
| Quiet Time (sec)2 | |
| Sensitivity (A/V) 1.e-005 💌 | |
| Electrode 2 | |
| Potential (V) | Off |
| Differential E (V) | Constant E |
| | C Scan |
| Sensitivity (A/V) 1.e-005 👤 | C Diff Scan |
| | |

The parameters for cyclic voltammetry are as follows.

Figure 5-2-2. CV parameter setting



| Table 5-2-1. CV Parameters | | | | |
|----------------------------|------------------------------|--|--|--|
| Parameter | Range | Contents | | |
| Init E (V) | -5 ~ +5 | Initial Potential | | |
| High E (V) | -5 ~ +5 | Potential scan high potential limit | | |
| Low E (V) | -5 ~ +5 | Potential scan low potential limit | | |
| Initial Scan | Positive or Negative | Scan direction at start of measurement | | |
| Scan Rate (V/s) | 1 × 10 ⁻⁶ ∼ 80 | Potential scan speed | | |
| Sweep Segment | 1 ~ 1,000,000 | Half cycle is1 segment | | |
| Sample Interval (V) | 1 × 10 ⁻³ ∼ 0.064 | Data sampling interval | | |
| Quiet Time (sec) | 0 ~ 100,000 | Quiet time before potential scan | | |
| Sensitivity (A/V) | 1 × 10 ⁻⁹ ~ 0.01 | Sensitivity scale | | |
| Scan Complete Cycle | Selected / Unselected | If the sweep segment is odd and the initial potential is different from the high and low potentials, the final potential stops at the initial potential | | |
| Electrode 2 | | | | |
| Potential (V) | -5 ~ +5 | Initial potential (used in Constant E) | | |
| Differential E (V) | -0.2 ~ +0.2 | Potential offset during DiffScan | | |
| Sensitivity (A/V) | 1 × 10 ⁻⁹ ∼0.01 | Sensitivity scale | | |
| Off | Selected / Unselected | Turn off the second working electrode | | |
| Constant E | Selected / Unselected | Hold the second working electrode at a constant potential | | |
| Scan | Selected / Unselected | Scan for both first and second channels | | |
| Differential Scan | Selected / Unselected | Scan the second channel while maintaining a constant potential difference from the first channel | | |

Table 5-2-1. CV Parameters

Note

- 1. Potential range is ± 5 V.
- 2. High and low potentials should be at least 0.01 V apart.
- 3. If high or low potential is entered outside the potential range, a warning will be displayed.
- 4. Depending on the initial potential, high potential, and low potential, the system will automatically readjust the initial scan direction.
- 5. As the number of sweep segments increases, the data sampling interval automatically increases to 0.02 V. If the scan rate is 0.5 V/s or higher, the number of sweep segments is limited by the memory size. The maximum is 64 K data points. If the scan rate is low, the specified number of sweep segments will be performed, but only the limit number of segments will be saved. It is effective to increase the sweep segment during electrode pretreatment.
- 6. When the second working electrode is Scan, the potential scan setting of the second working electrode is the same as the first working electrode potential. The potentials of the two electrodes are not independent.
- 7. The End of Scan Cycle works only when the initial potential is different from the high or low potential and the sweep segment is 3, 5, 7, or 9 (odd numbers). When End of Scan Cycle is activated, the last segment stops at the initial potential instead of the high and low potentials.



5-2-2. Linear Sweep Voltammetry parameters

The experimental parameters for linear sweep voltammetry are as follow.

| Linear Sweep Voltammetry Parameters | × |
|---|----------------------|
| Init E (V) 0 Final E (V) 0 Scan Rate (V/s) 0.1 Sample Interval (V) 0.001 Quiet Time (sec) 2 Sensitivity (A/V) 1.e-005 | OK Cancel Help |
| Electrode 2 Potential (V) 0 Sensitivity (A/V) 1.e-005 | □ On □ Scan |

Figure 5-2-3. LSV parameter setting

| Parameter | Range | Contents |
|---------------------|-------------------------------|---|
| Initial E (V) | -5 ~ +5 | Initial Potential |
| Final E (V) | -5 ~ +5 | Final potential (V) |
| Scan Rate (V/s) | $1 \times 10^{-6} \sim 80$ | Potential scan speed |
| Sample Interval (V) | $1 \times 10^{-3} \sim 0.064$ | Data sampling interval |
| Quiet Time (sec) | 0 ~ 100,000 | Quiet time before potential scan |
| Sensitivity (A/V) | $1 \times 10^{-9} \sim 0.01$ | Sensitivity scale |
| Electrode 2 | | |
| Potential (V) | -5 ~ +5 | Initial potential |
| Sensitivity (A/V) | 1 × 10 ⁻⁹ ~ 0.01 | Sensitivity scale |
| On | Selected / Unselected | Second working electrode on/off |
| Scan | Selected / Unselected | Scanning for both first and second channels |

Note

- 1. Potential range is ± 5 V.
- 2. High and low potentials should be at least 0.01 V apart.
- 3. The increase in potential is 1 mV.
- 4. When the second working electrode is only "On", the potential of the second working electrode becomes the constant potential (Constant E) at the potential set in "Potential E (V)".
- 5. When "On" and "Scan" are checked for the second working electrode, the potential scan of the second working electrode is the same as the potential scan set for the first working electrode. The potentials are not independent.


5-2-3. Amperometric i-t curve parameters

The experimental parameters for the amperometric i-t curve are as follow.

| Amperometric i-t Curve Parameters | × |
|--|----------------------|
| Init E (V) 0.1 Sample Interval (sec) 0.1 Sampling Time (sec) 400 Quiet Time (sec) 0 Scales during Run 1 Sensitivity (A/V) 1.e-005 | OK Cancel Help |
| Electrode 2 Potential (V)0 Sensitivity (A/V) 1.e-005 💌 | ☐ E2 On ☐ i2 On |

Figure 5-2-4. i-t parameter setting

Table 5-2-3. i-t parameters

| Parameter | Range | Contents |
|-----------------------|-----------------------------|---|
| Initial E (V) | -5 ~ +5 | Initial Potential |
| Sample Interval (sec) | 1 × 10 ⁻⁵ ∼ 10 | Data sampling interval |
| Sampling Time (sec) | $0.01 \sim 1 \times 10^7$ | Total measurement time |
| Quiet Time (sec) | 0 ~ 100,000 | Quiet time before starting data collection |
| Scales during Run | 1, 2, 3 | Current display scale number |
| Sensitivity (A/V) | 1 × 10 ⁻⁹ ~ 0.01 | Sensitivity scale |
| Electrode 2 | | |
| Potential (V) | -5 ~ +5 | Second working electrode potential |
| Sensitivity (A/V) | 1 × 10 ⁻⁹ ~ 0.01 | Second working electrode sensitivity scale |
| E2 On | Selected / Unselected | Second working electrode on/off control |
| i2 On | Selected / Unselected | Second working electrode current measurement on/off |

Note

- 1. Select the data sample interval according to the measurement time. For longer measurements, larger sample intervals and longer sample intervals will improve signal averaging and reduce noise. If the sample interval is shorter than 0.002 sec, the data cannot be transferred and displayed in real time. The maximum is 64 K data points.
- 2. If the sample interval is 0.002 sec or more, the data storage interval will be doubled automatically when the data exceeds the maximum data point during measurement.
- 3. When current 1 is displayed during measurement, it is automatically fitted to the data. When current 2 is displayed during measurement, it will be 1/100 or 1/10 of the full scale. When current 3 is displayed during measurement, it will be 1/100, 1/10, or 1/1 of the full scale.
- 4. If "i2 On" is not checked while "E2 On" is checked on the second working electrode, the potential input to "Potential (V)" will be applied to the second working electrode, but current data will not be acquired.



5-2-4. Open Circuit Potential - Time parameters

The experimental parameters for Open Circuit Potential - Time are as follow.

| Parameters for Open Circuit Potential - Time | ; | × |
|--|--------|---|
| Run Time (sec) | ОК |] |
| Sample Interval (sec) 0.1 | Cancel | 1 |
| High E Limit (V) 1 | Help | 1 |
| Low E Limit (V) -1 | | 1 |

Figure 5-2-5. OCPT parameter setting

| Parameter | Range | Contents |
|-----------------------|---------------------------|----------------------|
| Run Time (sec) | 1 ~ 500,000 | Measurement time |
| Sample Interval (sec) | 1 × 10 ⁻⁵ ∼ 10 | Sampling interval |
| High E limit (V) | -2.5 ~ 2.5 | High potential limit |
| Low E limit (V) | -2.5 ~ 2.5 | Low potential limit |

Table 5-2-4. OCPT parameters

Note

- 1. Up to 128,000 data points are allowed.
- 2. A warning will be displayed when the high or low potential limit is reached.
- 3. The data storage interval is selected according to the length of the experiment. The longer the measurement, the larger the data storage interval. A longer sampling interval results in more signal averaging and less noise. If the sample interval is shorter than 0.002 sec, the data cannot be transferred and displayed in real timec. The maximum is 64 K data points.
- 4. If the sample interval is 0.002 sec or more, the data exceeds the maximum data points, and the data storage interval is automatically doubled. As a result, the data points will not overflow even with unexpectedly long measurements.



5-3. System Setup

Select "Setup" - "System" to display the system setup dialog box. Configure settings according to the operating environment.

| System Setup | | × |
|---|--|--|
| Communication Port- | 1 | OK |
| C Com 1 C Com 2 C Com 3 C Com 4 | Line Frequency | Cancel Help |
| C Com 5 C Com 6 C Com 7 C Com 8 | Potential Axis Positive Left Positive Right | _Set Com Port |
| C Com 9 C Com 10 C Com 11 C Com 12 | Current Axis Positive Up Positive Down | ADC Calibration |
| Com Port Speed C Standard © Fast | Current Polarity Cathodic Positive Anodic Positive | Save retrieve data during run Auto data save after run Present Data Override Warning Save Text File As Well |
| Windows © English | C Oriental | Save Text File As Well Check Com port while idling Automatically detect Com port |

Figure 5-3-1. System setup

The same operation can be performed by clicking the icon.

- Communication Port

Manually select the communication port for connecting the PC to the device. If "Automatically detect communication port" is selected, the software scans USB ports and assigns a valid Com# to the communication port.

- Communication Port Speed (Com Port Speed)

Set the communication speed between the PC and the device. The fast setting allows for real-time data transfer at high scan rate/short sampling interval. The standard setting provides excellent compatibility.

- Line Frequency

Set the power supply frequency according to the operating environment. For measurement methods affected by power supply frequency, this setting is useful to reduce interference.

Potential Axis

Switch the positive potential axis to the left or right.

Current Axis

Allows switching the positive current axis up or down.

Current Polarity

Select positive current from cathode current or anode current. If setting is not made prior to measurement, it will not be reflected in the experimental results (peaks and waveforms). For the display in the standard IUPAC format, set the potential axis to Positive Right, the current axis to Positive Up, and the current polarity to Anodic Positive.



· Windows

Select English to use English or Oriental to use Japanese. If you select Oriental, the text (characters) will be larger than if you select English. In the English window, the technique selection field may be truncated. The Oriental window does not support English system fonts. For example, English μ is displayed as u.

- Data Length

The default data length is 256 K. Increasing the data length requires a large amount of RAM, which can slow down the system and prohibit other programs from running during the measurement. If the change is not required, it is recommended to do not change it. If you change the data length, exit the software and restart it, otherwise the program may crash. If data is acquired and saved with a longer data length but read back with a shorter data length, the program may also crash. Once the data length has been set, do not change the data length.

- Save retrieve data during run

Checking this option will restore the data stored on the hard disk during the measurement. If an experiment is not completed successfully due to external interference, interruption, or missed communication, data can be recovered partially. This is useful for experiments with slow scan rate.

- Present Data Override Warning

If experimental data has not been saved before making a new measurement or opening a file that exists on disk, the unsaved data will be ignored. This option causes the system to display a warning message before data is lost.

- Save Text File As Well

It is usually saved as binary files (.bin). Binary files contain more information (experiment control information) and the data size is small. If checked, when saving a binary file, it will also be saved as a text file.

- Set Com port

Set the communication port for connecting the PC and the device.

ADC Calibration

The analog-to-digital converter (ADC) calibration factors are stored in the non-volatile memory of the instrument. ADC calibration is performed before shipment. If the ADC calibration is successful, the new calibration factors will replace the old ones. Otherwise, the old one is retained.

| Information | × |
|----------------------|-----------------|
| ADC offset check and | save successful |
| | ОК |

Figure 5-3-2. ADC calibration factor



5-4. Hardware - Test

Select "Setup" - "Hardware-Test" to check the operation of the device. When selected, the hardware self-test is executed and the results are displayed after the test.

| Hardware Test Results | | × |
|---|---|------------|
| Digital circuitry test results: ROM version: 20.01 ROM revision date: 18–Jan–2020 Potential offset test <u>OK</u> . Channel 1 offset test <u>OK</u> . Channel 2 offset test <u>OK</u> . Sensitivity test <u>OK</u> . Analog circuitry test <u>OK</u> . | ^ | UK Help |
| | ~ | |



| Hardware Test Results | > | K Hardware Test Results | × |
|---|-----------|---|----|
| Digital circuitry test results: ROM version: 13.05 ROM revision date: 28-Oct-2013 Check Code: 1 E offset (V) = -4.5416 i1 offset (1e-1A/V) = -4.5461 i1 offset (1e-2A/V) = -4.5463 i1 offset (1e-3A/V) = -4.5454 i1 offset (1e-5A/V) = -4.5482 i1 offset (1e-5A/V) = -4.5482 i1 offset (1e-5A/V) = -4.5444 Sensitivity (1e-2A/V) error = -100.00% Sensitivity (1e-2A/V) error = -30.35% Sensitivity (1e-5A/V) error = -30.33% Sensitivity (1e-5A/V) error = -30.35% Sensitivity (1e-5A/V) error = -30.35% Sensitivity (1e-5A/V) error = -58.67% | ∧ ОК Неір | ii offset ($1e-1A/V$) = -4.5.451 ii offset ($1e-2A/V$) = -4.5.456 ii offset ($1e-2A/V$) = -4.5.459 ii offset ($1e-3A/V$) = -4.5.454 ii offset ($1e-6A/V$) = -4.5.454 ii offset ($1e-6A/V$) = -4.5.442 Sensitivity ($1e-2A/V$) error = -100.00% Sensitivity ($1e-2A/V$) error = -90.78% Sensitivity ($1e-3A/V$) error = -90.78% Sensitivity ($1e-3A/V$) error = -90.38% Sensitivity ($1e-3A/V$) error = -90.38% Sensitivity ($1e-6A/V$) error = -90.38% Sensitivity ($1e-6A/V$) error = -90.35% Sensitivity ($1e-6A/V$) error = -93.39% 2nd gain stage error = -93.39% 3rd gain stage error = -100.00% Analog circuitry test error! See details above. | ОК |

Figure 5-4-2. Hardware - Test error example

- Digital circuitry test results

Display the software version and revision date of the device.

- Potential and current offset test

Display the results of the potential and current offset test.

- Sensitivity scale test

The sensitivity scale has 6 ranges, and an error is displayed if it exceeds a certain level.

Gain test

There are 3 gain ranges. If the error exceeds 1%, an error will be displayed.

- Analog circuitry test results

Display the results of the analog circuitry tests. If no error is detected, only "Analog circuitry test OK" is displayed. If an error is displayed, repeat the test several times to confirm that the errors are the same. Record the error details and contact your distributor.

Note

1. When performing a hardware test with the cell cable connected to the electrode, sample solution, etc. an error may occur.



Chapter 6. Control Menu

The following is a list of functions that can be selected from the control menu on the toolbar.

6-1. Measurement

This command is used to start running the experiment from "Control" - "Run Experiment". It is used to skip settings for quiet time, deposition time, and preconditioning.

| trol | Graphics | DataProc | An |
|------------------|--|---|--|
| <u>R</u> un | Experiment | | |
| Paus | e / Resume | | |
| <u>S</u> top Run | | | |
| Re <u>v</u> e | rse Scan | | |
| Run | Status | | _ |
| | <u>R</u> un Paus <u>S</u> top Re <u>v</u> e | <u>Run Experiment</u> Pause / Resume | <u>Run Experiment</u> Pause / Resume <u>S</u> top Run Re <u>v</u> erse Scan |

Figure 6-1-1. Toolbar

After the measurement is executed, the subsequent confirmation is automatically performed before the measurement starts. The connection with the device is checked, and if the connection cannot be confirmed, the command ends and an error message is displayed. Next, the experimental parameters are checked, and if the settings are not appropriate, the command ends and an error message is displayed.

The measurement can be displayed in real time, but if the data acquisition speed exceeds the data transmission rate, it will be displayed after the measurement is finished.

The same operation can be performed by clicking on the icon.

6-2. Pause/Resume

This command is used to pause and resume measurement from "Control" -"Pause/Resume". This command is unavailable for time-based measurement (i-t).

The same operation can be performed by clicking on the icon.

6-3. Stop Run

This command is used to stop measurement from "Control" - "Stop Run". It can be used to stop measurement in progress, repeated measurement, or macro command.

The same operation can be performed by clicking on the icon.



Figure 6-3-1. Toolbar



6-4. Reverse Scan

From "Control" - "Reverse Scan," this command is used to reverse the potential scan during CV measurement. Pressing this command reverse the sweep segment. If used during measurement, data analysis such as peak search will be unavailable. This command does not work for other techniques.

The same operation can be performed by clicking on the icon.

| Cor | ntrol | Graphics | DataProc | An |
|-----|------------------|----------|----------|----|
| ► | Run Experiment | | | |
| Ш | Pause / Resume | | | |
| | <u>S</u> top Run | | | |
| ¢ | Re <u>v</u> e | rse Scan | | |
| | R <u>u</u> n | Status | | |

Figure 6-4-1. Toolbar

6-5. Run Status

It is used to make settings related to measurement from "Control" - "Run Status".

| | Run Status | × |
|-----------------------------------|--|---------------------------------|
| | Use Open Circuit E as Init E Smooth After Run | ОК |
| | - Abort Quiet Time | Cancel |
| | • No | Help |
| | C Current (A) : | Run |
| | C Current (A) < | |
| | | Control Level of Stir Line |
| | Abort Experiment at Level | Active High |
| | ● No | C Active Low |
| Control Graphics DataProc An | C Current Overflow | |
| Run Experiment Pause / Resume | C Current (A) > 0.002 | 🔲 External Trigger Run |
| Stop Run | C Current (A) < 0 | 🔽 Stir Between Runs |
| Reverse Scan | C Charge (C) > 100000 | 🔽 Purge Between Runs |
| R <u>u</u> n Status | C 10s Change < 0 | 🔲 Knock |
| Re <u>p</u> etitive Runs | | |

Figure 6-5-1. Toolbar (left), Run Status (right)

- Use Open Circuit E as Init E

Execute the open circuit potential before measurement and use the measured potential as the initial potential.

Smooth After Run

Smoothing is performed automatically after measurement. This option can also be turned on/off from "Data Processing" - "Smooth". From data processing, the smoothing mode can be set to Least Square or Fourier Transform.



Abort Quiet Time

When the current or charge reaches a specific value (or overflows), the quiet time before data sampling is stopped. If No is selected, measurement is performed to the end.

- Abort Experiment at Level

When the current or charge reaches (or overflows) a specific value, the measurement is ended automatically. The electrode is protected against overcurrent.

No : Measurement is performed until the end.

Current Overflow : If the current overflows, the measurement is stopped.

Current(A)> : If the current is higher than the specified value, measurement is stopped.

Current(A)< : If the current is smaller than the specified value, measurement is stopped.

Charge(C)> : If the charge is higher than the specified value, the measurement is stopped.

10s Change< : If the signal change after 10 seconds is less than the specified value, the measurement is stopped.

After 10 seconds from the start of measurement, the signal change is checked at each sampling interval. The response time is the same as the sampling interval. After data is read, it compares it to the data point sampled 10 seconds earlier and checks the difference. If the difference is less than the specified value (A for current or V for potential), the measurement is stopped. This option is valid only for time-based measurements (i-t).

- Control Level of Stir Line

Select the signal of the stirring line from Active High and Active Low.

- External Trigger Run

The measurement is started by the external trigger signal from the cell control terminal on the back of the device. See 3-6. for pin assignments.

- Stir Between Runs

Select to perform stirring between measurements.

- Purge Between Runs

Select to perform a purge between measurements.



6-6. Repetitive Run

| | Repetitive Runs | × |
|---|--|--|
| | Number of Runs Time Interval Between Runs (sec) 0 Prompt Before Each Run © None © Manual © External Trigger Data Averaging When Done | OK Cancel Help Repetitive Run |
| Control Graphics DataProc An Run Experiment Pause / Resume Stop Run Reverse Scan Run Status | Result Report File Enable Peak Shape: Default Data Type: Original | |
| Repetitive Runs M Macro Command | Base Filename | Browse |

Repetitive Run can be set from "Control" - "Repetitive Run".

Figure 6-6-1. Toolbar (left), Repetitive Run (right)

- Number of Runs

Enter the Number of Runs. The parameter range is 1 to 999,999.

- Time Interval Between Runs

Specify the delay time between two consecutive measurements. This parameter is ignored if the Prompt Before Each Run is enabled.

- Prompt Before Each Run

If Manual is selected, a message box is displayed before each measurement (except the first measurement) and the instrument waits for a response.

Selecting External Trigger puts the instrument in a state of waiting for an external signal before each measurement. The external trigger signal is active low and is connected to pin 13 of the cell control terminal on the back of the instrument.

When manual or external trigger is selected, the time interval setting has no effect.

- Data Averaging When Done

Repeated measurement data is averaged and saved as file name 0.

- Result Report File

Select Enable to create a file. For the peak waveform, select from Default, Gaussian, Diffusive, or Sigmoidal. In the case of CV and LSV, select the diffusive waveform.

Select the data format from Original, Semi-deriviative, and derivative. Normally, select Original. For better quantification, differential and semi-derivative processing can be used to find accurate peak potentials.

- Base Filename

Click Browse or enter a Base Filename. You can enter up to 5 characters. The data will be saved after each measurement. The number of measurements will be added to the base filename. For example, Base Filename N, where N is the number of measurements (1 to 9999). If no base filename is specified, a warning will be displayed. If continued, the measurement will start without saving the data.



6-7. Macro Command

Macro commands can be set from "Control" - "Macro Command". A series of commands can be executed in a specified order, which is useful for performing measurements automatically.

| | | Macro Command | × |
|---|--|--|--|
| | | Macro Command Editor: | |
| | Graphics DataProc An Run Experiment Pause / Resume Stop Run Stop Run Reverse Scan Run Status Repetitive Runs Repetitive Runs | <pre># Welcome to Macro Command! # Use this dialog box to execute a series of commands. # This is analogous to batch files in Windows or shell scripts in Unix/Linux. # Click the 'Heip' button for more information. # " # Beep three times. for: 3</pre> | OK Cancel Help Read Save Test Run Macro Run on OK |
| м | Macro Command | | |
| | Open Circuit Potential | < > | |

Figure 6-7-1. Toolbar (left), Macro Command (right)

- Read

Saved macro commands can be loaded.

- Save

Save the file edited by the macro command.

- Test

Test the macro command. If the macro command is correct, a message indicating that the test was successful is displayed. If an error is detected, the details of the error will be displayed.

Run Macro

Execute the macro command. The macro command and parameter range are automatically checked before execution. If an error is detected, the macro command is terminated and a warning message is displayed.

- Macro Command Editor

Enter commands in the edit box. Each command is entered in one line at a time. It is case-insensitive and spaces are ignored.

If a parameter is required after the command, use a colon ":" or equal sign "=" to separate the parameters. The text following the semicolon ";" or the hash symbol "#" is ignored (inline comment).



| Table 6-7-1 | . Explanation | of command | functions | (basic) | 1 |
|-------------|---------------|------------|-----------|---------|---|
|-------------|---------------|------------|-----------|---------|---|

| Command | Parameter | Explanation | |
|------------------|------------|--|--|
| tech | string | Selection of electrochemical technique. | |
| run | | Start run. | |
| save | string | Save data to file. When used in a For Next loop, the file name can only be entered with 5 characters and the loop number (1-999) is added to the file name. | |
| tsave | string | Save data as a text file | |
| end | | Exit macro, the following lines will be ignored. | |
| for | 1 - 999 | In the case of next loop, only one layer is allowed. | |
| next | | For next loop end | |
| delay | 1 - 32,000 | Delay between commands | |
| purge | 1 - 32,000 | Purge for specific time | |
| stir | 1 - 32,000 | Stirring for specific time | |
| trigon | | Turn on trigger before run | |
| trigoff | | Turn off trigger before run | |
| knockon | | Turn on drop knocker. Also used to send trigger pulse to external device before run | |
| konckoff | | Turns off drop knocker . No pulse before run | |
| cellon | | Cell on between runs | |
| celloff | | Cell off between runs | |
| specon | | For SEC2020 Spectromoter, set Cell Control port pin 14 High | |
| specoff | | For SEC2020 Spectromoter, pull Cell Control port pin 14 low | |
| rdeon | | Turn on RDE | |
| rdeoff | | Turn off RDE | |
| rderunon | | RDE on during run | |
| rderunoff | | RDE off during run | |
| rdebetweenrunon | | RDE on between runs | |
| rdebetweenrunoff | | RDE off between runs | |
| rdequieton | | RDE on during quiet time | |
| rdequietoff | | RDE off during quiet time | |
| rpm | 0 ~ 10000 | RDE rotation rate (rpm) | |
| rpmincr | 0 ~ 1000 | RDE rotation rate increment (rpm) | |
| rpmsqrt | 0 ~ 100 | Square root of rotational rate | |
| rpmsqrtincr | 0 ~ 10 | Square root of rotational rate increment | |
| dummyon | | Control > Cell > Test on in internal dummy cell | |
| dummyoff | | Control > Cell > Test Off in internal dummy cell | |
| ei | -5 ~ +5 | Initial potential | |
| eh | -5 ~ +5 | High limit of potential in CV | |
| el | -5 ~ +5 | Low limit of potential in CV | |
| ef | -5 ~ +5 | Final potential for sweep technique | |
| eio | | Use OCP as initial potential. Entering an initial potential (ei) turns the command off. | |
| eho | | Use OCP as high potential. Entering high potential (eh) turns the command off. | |



| elo | | Use OCP as low potential. Entering low potential (el) turns the command off. |
|-------------------|---------------------------|---|
| efo | | Use OCP as final potential. Entering final potential (ef) turns the command off. |
| eiincr | -1 ~ +1 | Increment the initial potential in for next loop. Should be |
| efincr | -1 ~ +1 | reset to zero after the loop. Increment the final potential in for next loop. Should be reset to zero after the loop. |
| ehincr | -1 ~ +1 | Increment the high potential in for next loop. Should be reset to zero after the loop. |
| elincr | -1 ~ +1 | Increment the low potential in for next loop. Should be reset to zero after the loop. |
| fullcycleon | | CV full cycle on |
| fullcycleoff | | CV full cycle off |
| efon | | Final potential on |
| efoff | | Final potential off |
| V | 1e-6 ~ 80 | Scan rate |
| incre | 0.01 ~ 0.05 | Potential increment |
| pn | 0.01 0.00 | Initial scan polarity |
| cl | 1 ~ 1e6 | Number of segments in CV |
| si | 0.00~ 0.064 | Sample interval |
| | 1e-9 ~ 0.01 | Sensitivity |
| sens | 16-9 ~ 0.01 | Automatic sensitivity for slow CV or LSV |
| autosens | 0 . 100.000 | Quiet time before run |
| qt | 0 ~ 100,000 0.01 ~ 1e7 | Total measurement time for i-t curve |
| st | 0.01 ~ 167 | |
| pcon | | Turn on preconditioning Turn off preconditioning |
| pcoff | | |
| initeon | | Return to initial potential after run |
| Initeoff | | Remain at final potential after run |
| pce1 | -5 ~ +5 | Potential for first stage of preconditioning |
| pce2 | -5 ~ +5 | Potential for second stage of preconditioning |
| pce3 | -5 ~ +5 | Potential for third stage of preconditioning |
| pct1 | 0 ~ 6,400 | Time for first stage of preconditioning |
| pct2 | 0 ~ 6,400 | Time for second stage of preconditioning |
| pct3 | 0 ~ 6,400 | Time for third stage of preconditioning |
| For bipotentiosta | it | |
| e2on | | Turn on second working electrode (no data recording) |
| e2off | | Turn off second working electrode |
| i2on | | Turn on second working electrode and record data |
| i2off | | Turn off second working electrode |
| e2 | -5 ~ +5 | Second channel potential |
| e2scan | | Scan the second channel at the same time as the first channel |
| e2d | -0.2 ~0.2 | Difference potential between second and first channel during potential scan |
| e2dscan | | Scan the second channel while maintaining constant potential difference from the first channel |
| sens2 | 1e-9 ~ 0.01 | Sensitivity of the second working electrode |



| Command | Parameter | Explanation |
|---------|--------------|---------------------------|
| tech | CV | Select cyclic voltammetry |
| ei | -5 ~ +5 | Initial potential (V) |
| eh | -5 ~ +5 | High potential (V) |
| el | -5 ~ +5 | Low potential (V) |
| pn | 'p' or 'n' | Initial Scan Polarity |
| V | 1e-6 ~ 80 | Scan rate (V/s) |
| cl | 1 ~ 1e6 | Sweep segments |
| si | 1e-3 ~ 0.064 | Sample interval (V) |
| qt | 0 ~ 1e5 | Quiet time (s) |
| sens | 1e-9 ~ 0.01 | Sensitivity (A/V) |

Table 6-7-2. Explanation of command functions (CV)

Table 6-7-3. Explanation of command functions (LSV)

| Command | Parameter | Explanation |
|---------|--------------|---------------------------------|
| tech | lsv | Select linear sweep voltammetry |
| ei | -5 ~ +5 | Initial potential (V) |
| ef | -5 ~ +5 | Final potential (V) |
| V | 1e-6 ~ 80 | Scan rate (V/s) |
| si | 1e-3 ~ 0.064 | Sample interval (V) |
| qt | 0 ~ 1e5 | Quiet time (s) |
| sens | 1e-9 ~ 0.01 | Sensitivity (A/V) |

Table 6-7-4. Explanation of command functions (i-t)

| Command | Parameter | Explanation |
|---------|-------------|----------------------------------|
| tech | i-t | Selection amperometric i-t curve |
| ei | -5 ~ +5 | Initial potential (V) |
| si | 1e-5 ~ 10 | Sample interval (V) |
| st | 0.01 ~ 1e7 | Run time |
| qt | 0 ~ 1e5 | Quiet time (s) |
| sens | 1e-9 ~ 0.01 | Sensitivity (A/V) |

Table 6-7-5. Explanation of command functions (OCPT)

| Command | Parameter | Explanation |
|---------|-------------|--------------------------------------|
| tech | ocpt | Select Open Circuit Potential - Time |
| st | 1 ~ 5e5 | Run time (s) |
| si | 1e-5 ~ 10 | Sample interval (s) |
| eh | -2.5 ~ +2.5 | High potential limit (V) |
| el | -2.5 ~ +2.5 | Low potential limit (V) |



| Measurement metho | od using i-t technique | Measurement method using LSV technique |
|---|---|---|
| 1 electrode i-t measurement method | 2 electrode i-t measurement method | |
| tech: i-t st=30 cellon ei = 0 run save: file0 for: 7 ei=-0.2 run save: file1a ei=0 run save: file1b next | tech:i-t eiincr = 0 ei=-0.5 st = 2 qt=0 e2 = 0 e2on cellon for:10 run save: file eiincr=0.1 next celloff eiincr = 0 | tech:lsv e1=-0.2 ef=0.8 v=0.1 sens=1e-5 run save: lsv100 v=0.2 run save: lsv200 v=0.5 run save: lsv500 v=1 run save: lsv1000 |

Table 6-7-6. Examples of macro commands

6-8. Open Circuit Potential

From "Control" - "Open Circuit Potential", open circuit potential is measured. The open circuit potential is the potential between working electrode and reference electrode while no current is flowing through the cell. It gives the initial conditions before start the measurement. The compound under study can distinguish whether is oxidizable or reducible.

After the measurement, the Open Circuit Potential value is displayed in a dialog box.



Figure 6-8-1. Toolbar (left), Open Circuit Potential (right)



6-9. Filter setting

From "Control" - "Filter Setting," set the filter settings.

A capacitor can be connected to the feedback resistor of the i/E converter to form a lowpass RC filter. This can remove high-frequency noise.

These filters are useful for reducing noise during measurements. Set the filter parameters from the dialog box.

| | | Filter Setting | | × |
|------|--|-----------------------------|-----------------------------|--------------|
| | | i/E Conv 1 Filter: 32 Hz | i/E Conv 2 Filter: 32 Hz | OK Cancel |
| Cor | ntrol Graphics DataProc An Run Experiment | Selection: | Selection: | Help |
| | Pause / Resume | Automatic | Automatic | |
| - | Stop Run | Automatic | Automatic | |
| Ð | Re <u>v</u> erse Scan | None | None | |
| | R <u>u</u> n Status | 32K Hz 3200 Hz | 32K Hz 3200 Hz | |
| | Re <u>p</u> etitive Runs | 320 Hz | 320 Hz | |
| м | Macro Command | 32 Hz 3.2 Hz | 32 Hz 3.2 Hz | |
| | Open Circuit Potential | 0.32 Hz | 0.32 Hz | |
| ~~.₹ | <u>F</u> ilter Setting | 0.032 Hz | 0.032 Hz | |
| Ð | <u>C</u> ell | | | |

Figure 6-9-1. Toolbar (left), Filter setting (right)

- i/E Conv 1/2 Filter

The gray box displays the filter settings for the actual i/E (current - voltage) converter.

- Selection

Select the i/E converter filter cutoff frequency.

This is the RC filter combined with the i/E (current - voltage) converter. The filter setting should take into account the timescale of the measurement and the type of experiment, selecting Automatic will set the default setting. If you are not sure which setting to select, select Automatic. Some cutoff frequencies may not be selectable. This is because for a given sensitivity scale, the feedback resistance of the i/E converter is fixed, and when this is combined with the RC filter, it may be impossible to reach certain cutoff frequencies. For example, a low (high) sensitivity scale may make it impossible to select a relatively low (high) cutoff frequency. Changing the sensitivity scale may allow the desired cutoff frequency to be selected.

The second column applies to bipotentiostat dual channel measurements.

The same operation can be performed by clicking on the icon.



6-10. Cell

From "Controls" - "Cell", set the purge, stir, and electrochemical cleaning settings.

| | | Cell Control | × |
|-----|----------------------------|--|---------------------------------|
| | | Control Level of Stir Line • Active High C Active Low | OK Cancel |
| Cor | ntrol Graphics DataProc An | Stir Time (sec) 10 | ✓ Stir Between Runs |
| ► | <u>R</u> un Experiment | Stir | Purge Between Runs |
| н | Pause / Resume | laura diata Diwan | 🔽 Stir and Purge Simultaneously |
| | <u>S</u> top Run | Immediate Purge | Test with Internal Dummy Cell |
| Ð | Re <u>v</u> erse Scan | Purge Time (sec) 240 | Cell On Between Runs |
| | R <u>u</u> n Status | Purge | Return to Init E after Run |
| | Re <u>p</u> etitive Runs | Immediate Cell On | SMDE Drops Before Run 0 |
| м | Macro Command | E 1 (V) 0 🗖 On | Mercury Drop Collection |
| | Open Circuit Potential | E 2 (V) 0 0 0n | Number of Drops |
| ~~₹ | <u>F</u> ilter Setting | Time (sec) 10 | |
| ٩ | <u>C</u> ell | Cell On | Collect |
| | Step Functions | | |

Figure 6-10-1. Toolbar (left), Cell (right)

- Control Level of Stir Line

The stir line control signal can be set as active high (BAS) or active low (PAR).

- Immediate Stir

Specify the immediate stir time (from 1 to 32767). Click the stir button to activate immediate stirring.

- Immediate Purge

Specify the immediate purge time (from 1 to 32767). Click the Purge button to activate immediate purging.

Immediate Cell On

Specify the cell potential (from -3.276 to +3.276) and Cell On time (from 1 to 32767). Click the Cell On button to stabilize the electrode at the set potential and time.

- Stir Between Runs

Check this box to enable stir between runs. This option can also be turned on or off using "Control" - "Run Status".

- Purge Between Runs

Check this box to enable purge between runs. This option can also be turned on or off using "Control" - "Run Status".

- Stir and Purge Simultaneously

Checking this box to enable stir and purge at the same time.

- Test with Internal Dummy Cell

A 1 M Ω resistor is provided as an internal dummy cell. Checking this box, the cell will not be turned on, but an internal dummy cell will be used for the measurement. This option is usefull for testing the instrument.



Cell On Between Runs

For the default, it is turned on only during measurement and turned off after the measurement ends. Check this box to keep the cell connected after the run. Be careful when this option is enabled. During Cell On, connecting or disconnecting the cell using improper procedures may damage the electrode. When connecting, connect the reference electrode and counter electrodes first, and when disconnecting, disconnect the working electrode first.

- Return to Init E after Run

After run, the potential returns to the initial potential. Otherwise, the potential will remain at the final value of the previous run. It only makes sense to enable this option if Cell On Between Runs is also checked.

The same operation can be performed by clicking the icon.



6-11. Step Functions

This command is used to generate a continuous square waveform from "Control" - "Step Functions". This command is used for electrode treatment and cleaning.

| Cor | ntrol Graphics DataProc Ar | | |
|----------|---|-----------------|----------|
| | <u>R</u> un Experiment Pause / Resume | Step Functions | × |
| а С | <u>S</u> top Run Re <u>v</u> erse Scan | Start E (V) | 0K |
| | R <u>u</u> n Status | Duration (s) | 0 Cancel |
| | Re <u>p</u> etitive Runs | Step E1 (V) | 0 Help |
| м | Macro Command | Step Time 1 (s) | 0.1 Bun |
| | Open Circuit Potential | Step E2 (V) | 0 |
| ~~∓ ⊕ | <u>F</u> ilter Setting Cell | Step Time 2 (s) | 0.1 |
| *9 | Step Functions | Step Segment | 2 |
| | Preco <u>n</u> ditioning | | |

Figure 6-11-1. Toolbar (left), Step function (right)

| Parameter | Range | Contents |
|-------------------|-------------------|------------------------------------|
| Start E (V) | -5 ~ 5 | Starting potential |
| Duration (s) | 0 ~ 100000 | Duration at start potential |
| Step E 1/2 (V) | -5 ~ 5 | 1st (E1) / 2nd (E2) step potential |
| Step Time 1/2 (s) | 0.0001 ~ 100000 | Duration of 1st / 2nd step |
| Step Segment | 1 ~ 2,000,000,000 | Half cycle is set as 1 segment |

 Table 6-11-1. Step function parameters

- Run

Pressing the "Run" button activates the step function generator. A status box will be displayed showing the number of steps, remaining steps, and time. Press the stop button to cancel this function. No data will be collected or displayed.

NOTE

- 1. The potential range between the start potential and the first and second step potentials must be less than 13.1 V.
- 2. If the start potential duration is less than 0.001 seconds, the start potential will be ignored.
- 3. Steps are applied in the sequence of step potential 1, step potential 2, step potential 1, step potential 2, The final potential will be step potential 1 for an odd number of segments and step potential 2 for an even number of segments.



6-12. Preconditioning

From "Control" - "Preconditioning", this command is used to treat the working electrode before run. This is useful, for example, for electrode cleaning. Preconditioning is performed before deposition or quiet time. The working electrode will be conditioned with up to 3 potential steps.

| | | Preconditioning before Run | × |
|-------------------|--|---|------------------------------------|
| | | Enable Preconditioning | ОК |
| | ntrol Graphics DataProc An <u>R</u> un Experiment Pause / Resume <u>S</u> top Run | First step Potential (V) Time (s) | Cancel Help |
| U M | Re <u>v</u> erse Scan R <u>u</u> n Status Re <u>p</u> etitive Runs <u>M</u> acro Command | Second step Potential (V) 0 Time (s)0 | |
| ~∵ ≢ © | Open Circuit Potential <u>F</u> ilter Setting <u>C</u> ell Step Functions Preconditioning Rotating Disk Electrode | Third step Potential (V) 0 Time (s)0 | Electrode 2 Stir On Purge On |

Figure 6-12-1. Toolbar (left), Preconditioning (right)

- Enable Preconditioning

Preconditioning is performed before deposition or quiet time. Uncheck this box to disable preconditioning.

- Potential

The parameter range is from -5 to 5.

- Time

The parameter range is from 0 to 6,400. Set to 0 to skip a given step. If the time setting is specified with a non-zero value less than 1 msec, the time control may not be accurate.

- Stir On

Check this box to to enalbe stir during preconditioning.

- Purge On

Check this box to enable purge during preconditioning.



6-13. Rotating Disk Electrode Control

This command is used to set the rotation rate of the rotating disk electrode from "Control" - "Rotating Disk Electrode Control".

| Cor | ntrol Graphics | DataProc | An | | |
|-----|-------------------------|----------|----|---------------------------------|--------|
| ► | <u>R</u> un Experimen | t | | | |
| | Pause / Resume | | | | |
| | <u>S</u> top Run | | | | |
| Ð | Re <u>v</u> erse Scan | | | | |
| | R <u>u</u> n Status | | | | |
| | Repetitive Runs | | | Rotating Disk Electrode Control | × |
| м | Macro Commar | d | | | OK |
| | Open Circuit Po | tential | | Rotation Speed (rpm) | 0K |
| ~~₹ | <u>F</u> ilter Setting | | | Rotate during Deposition Time | Cancel |
| ூ | <u>C</u> ell | | | Rotate during Quiet Time | |
| | Step Functions | | | | Help |
| | Preconditionin | g | | 🔲 Rotate during Run | |
| | Rotating Dis <u>k</u> E | lectrode | | Rotate between Run | |
| | Other | | | | |

Figure 6-13-1. Toolbar (left), Rotating Disk Electrode Control (right)

- Rotate Speed (rpm)

Specify the speed of the rotating disk electrode. The parameter range is from 0 to 10,000 rpm. The Cell Control port of the instrument outputs a voltage of 0 to 10 V, which corresponds to a rotation speed of 0 to 10,000 rpm.

- Rotate during Deposition Time

Check this box to turn the rotator on during deposition time in stripping mode.

- Rotate during Quiet Time

Check this box to turn the rotator on during quiet time.

- Rotate during Run

Check this box to turn the rotator on during run.

Rotate between Run

Check this box to turn the rotator on between measurements.



6-14. Other Controls

From "Control" - "Other...", this command is used to control the spectrometer when performing spectroelectrochemical measurements. It works with CV, LSV, and i-t techniques.

The spectrometer is controlled using the output from Pin#14 of the cell control terminal on the back of the instrument. See 3-6 for pin assignments.

| Con | trol Graphics DataProc A | | |
|-----|---------------------------------|----------------------|--------|
| ► | <u>R</u> un Experiment | | |
| Ш | Pause / Resume | | |
| - | Stop Run | | |
| ₽ | Re <u>v</u> erse Scan | | |
| | R <u>u</u> n Status | | |
| | Re <u>p</u> etitive Runs | | |
| м | Macro Command | | |
| | Open Circuit Potential | Other Controls | |
| ~~₹ | <u>F</u> ilter Setting | | |
| Ð | <u>C</u> ell | Spectrometer Control | OK |
| | Step Functions | | |
| | Preco <u>n</u> ditioning | (* OII | Cancel |
| | Rotating Dis <u>k</u> Electrode | O On | Hala |
| | Other | | Help |
| | Stripping Mo <u>d</u> e | 、 | |

Figure 6-14-1. Toolbar (left), Other controls (right)

- Spectrometer Control

When a measurement is executed with "On" is selected, the TTL signal output is at a High level (5 V) during the measurement and at a Low level (0 V) during all other times. When "On" is selected, the Cell command functions "SMDE drop before measurement" and "mercury drop collection" are not available.

The output of Pin#14 is High level (5 V) in the initial state. When the measurement (OCPT is recommended) is performed with "Spectrometer control" turned on, the output state of Pin#14 switches to Low level (0 V). Thereafter, the TTL signal is output at High level (5 V) during measurement and at Low level (0 V) except during measurement.

Once turned On, it will remain in the Spectrometer Control On state as long as power is supplied to the device. Reset "Spectrometer Control" to its initial state by disconnecting the USB cable from the device or by turning off the PC.



6-15. Stripping Mode

From "Control" - "Stripping Mode", enable the stripping mode and specify the deposition conditions. Stripping mode is only available for LSV techniques. It is not available when polarographic mode is selected.

Figure 6-15-1. Toolbar (left), Stripping mode (right)

Stripping Mode Enabled

Check this box enables stripping mode, which inserts a deposition step before the quiet time. The deposition potential and time can be specified from this dialog box. After the deposition period and before quiet time, the potential returns to the initial potential. The potential scan during the stripping step starts from the initial potential.

- Purge During Deposition

Check this box to purge the solution during deposition.

- Stir During Deposition

Check this box to stir the solution during deposition.

Deposition Potential, Quiet Potential

Select the initial potential or the deposition potential (D)/quiet potential (Q). If an initial potential is not selected, enter a value in the field at the bottom of the dialog box.

- Deposition E

If the deposition potential (D) is selected at the top of the dialog box, enter the potential during the deposition period. If the initial potential is selected, it will be invalid.

- Deposition Time

The parameter range is from 0 to 100,000.

• Quiet E (V)

If "Quiet potential (Q)" is selected in the "Quiet potential" field at the top, enter the potential during the quiet time. If the initial potential is selected, it will be invalid.



Chapter 7. Graphics Menu

The following are the functions that can be selected from the Graphics menu on the toolbar.

7-1. Preseent Data Plot

From "Graphics" - "Present Data Plot", plot the current active data. This is useful, for example, after using overlay or parallel plotting.

When you move the cursor to the X-axis or Y-axis, it becomes a double-headed arrow. Click and drag on the X or Y axis to change the scale.

The same operation can be performed by clicking on the icon. 🔛

| | Axis Options | × |
|---|--|----------------------|
| | Grid and Inv □ Current Density Invert □ Electrode Area: 1 Axis Label Expression □ E vs Reference Electrode Scientific □ Electrode: Ag/AgCl | OK Cancel Help |
| Graphics DataProc Analysis | LongAutcShortAutcFreeze:1.328e-006To:1.342e-006Title:Unit: | |
| New <u>O</u> verlay Plots <u>A</u> dd Data to Overlay Replot Overla <u>v</u> Manage Overlay | Grid: Change Point ▼ Color Thickness XYAxis: Change 1 ▼ | |
| New <u>P</u> arallel Plots Add Data to Para <u>l</u> lel Replo <u>t</u> Parallel Manage Parallel | Y axis Title Rotation on Printer 270 degree Axis Label: Arial Narrow Regular Axis Title: Times New Roman Bold | Change Change |

Figure 7-1-1. Toolbar (left), Axis Options (right)

Double-click on an axis opens the Axis Options dialog box. Settings in this dialog box can also be made using the Graph Options, Color and Legend, Font commands. The specific settings are as follows.

- Axis Label Expression

The format of the axis values can be choose from Scientific (for example, 1e-6A) or Engineering (for example, μ A). If Oriental is selected in the system setup, the μ symbol will not be displayed correctly.

- Long/Short

These options allow you to customize long (major) and short (minor) ticks. Select from the pull-down menu (Auto, 1, 2, to 20) on the right side.

To manually adjust the axis display range, check the "Fixed" checkbox and enter the range in the box on the right.



Double-clicking on the graph field opens the Insert Text dialog box.

| Insert Text | × |
|---|------------|
| Text: Erase | OK |
| X: -0.0445821 Y: 1.33794e-001 Rotation: 0 | Cancel |
| Font Style Size Color | Help |
| Arial Narrow Regular 6 Change | Default |
| ✓ Text Display Enabled | Monochrome |

Figure 7-1-2. Insert Text

- Text

Enter text to be displayed. The text is removed from the plot when you clear the existing text field and exit the dialog box.

- Erase

Clear the text field.

X/Y

Change the position of the text. X and Y are the coordinates of the upper left corner of the first letter. The default value indicates where you double-clicked.

- Rotation

Rotate the text clockwise from 0 to 360 degrees.

- Font

Click the Change button to specify the font, style, size, and color of the text. These settings apply to all text inserted on the current graph.

- Text Display Enabled

Uncheck it from the box to hide the text.

- Default

Set the current font as the default for all data files.

- Monochrome

Set the text color to black.



7-2. New Overlay Plot

"Graphics" - "New Overlay Plot" allows to plot multiple data on a single graph. This function is useful for comparing data. To select multiple files, hold down the Ctrl key while selecting the required files. To add more data to the overlayed plot, or to overlay data from other directory or drive, use the "7-3 Add Data to Overlay command".

| | 🚰 Overlay Data Display | | | | |
|-------------------------------|--|---------------------------------|----------|------|--------------------|
| | \leftarrow \rightarrow \checkmark \uparrow \square \rightarrow This PC \rightarrow Des | ktop > 3325-220711 > Data | | ٽ ~ | . ○ Search Data |
| | Organise 🔻 New folder | | | | |
| | Name | Date modified | Туре | Size | |
| | Cv1.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| Graphics DataProc Analysis | Cv2.bin | 2020/10/23 4:54 | BIN File | 6 KB | |
| Present <u>D</u> ata Plot | 📄 cv3.bin | 2020/10/23 4:55 | BIN File | 6 KB | |
| These in Data Hot | 📄 cv4.bin | 2020/10/23 5:44 | BIN File | 9 KB | |
| New Overlay Plots | it1.bin | 2020/10/23 4:57 | BIN File | 3 KB | |
| New Overlay Flots | Lsv1.bin | 2020/10/23 4:55 | BIN File | 4 KB | |
| Add Data to Overlay | 📄 Isv100rpmtest.bin | 2022/04/25 21:28 | BIN File | 7 KB | |
| Deplet Overlay | 📄 Isv1500rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| Replot Overla <u>v</u> | Isv5000rpmtest.bin | 2022/04/25 15:14 | BIN File | 7 KB | |
| Manage Overlay | Mcv.bin | 2020/10/23 5:47 | BIN File | 6 KB | |
| | Ocpt1.bin | 2020/10/23 4:58 | BIN File | 4 KB | |
| New <u>P</u> arallel Plots | | | | | |
| Add Data to Para <u>l</u> lel | | | | | |
| Replo <u>t</u> Parallel | File name: "cv4.b | n" "Cv1.bin" "Cv2.bin" "cv3.bin | | ~ | Data Files (*.bin) |
| Manage Parallel | | | | | Open Cancel |

Figure 7-2-1. Toolbar (left) and dialog box (right)

The scale of the plot will automatically adjust to the scale of the current active data. The data added to the overlay will be plotted if it is within the range of the active data, but if it is outside the range, please adjust the scale manually.

It can be customized by using the Graph Options, Color and Legend, Font commands. This feature does not check the type of data. The user checks the significance of the overlay, axis units, etc.

7-3. Add Data to Overlay

"Graphics" - "Add Data to Overlay" is used to add more data to a graph that has been overlayed, or to add data from other directory or drive to the overlay plot.

The scale of the plot will automatically adjust to the scale of the current active data. If both X and Y values are within the plot scale, then the data points will be plotted. The graph can be customized by using the Graph Options, Color and Legend, Font commands. This feature does not check data technique.

It is not available if you do not have an overlayed plot open.



Figure 7-3-1. Toolbar



7-4. Replot overlay

From "Graphics" - "Replot Overlay", the most recent overlay plot is displayed. This is useful, for example, after using the Present Data Plot.

New <u>P</u>arallel Plots... Add Data to Para<u>l</u>lel... Replo<u>t</u> Parallel Manage Parallel...

Graphics DataProc Analysis

New Overlay Plots...

Add Data to Overlay ...

Present Data Plot

Replot Overla<u>v</u> Manage Overlay...

Figure 7-4-1. Toolbar

7-5. Manage Overlay

From "Graphics" - "Manage Overlay" allows you to replace the selected data.

| | | Manage Overlay Files | × |
|------------|----------------------------|--|----------------------|
| Gra | phics DataProc Analysis | Cv1.bin C:\als3325\Data\Cv2.bin C:\als3325\Data\cv3.bin C:\als3325\Data\cv4.bin | OK Cancel Help |
| <u>الم</u> | Present <u>D</u> ata Plot | | |
| | New <u>O</u> verlay Plots | | Add Move Up |
| | Add Data to Overlay | | Move Down |
| | Replot Overlay | | Remove |
| | Manage Overlay | | |
| | New <u>P</u> arallel Plots | | |
| | Add Data to Parallel | | |
| | Replo <u>t</u> Parallel | | |
| | Manage Parallel | 1 | |

Figure 7-5-1. Toolbar (left), Manage Overlay (right)

- Addition

Use to add data to be overlayed.

- Move Up/Move Down

The order of the overlayed data can be changed. This order is also reflected in the display on the graph.

- Remove

Use to delete data to be overlayed.

7-6. New Parallel Plot

From "Graphics" - "New Parallel Plot", multiple data can be plotted side by side. This is useful for displaying different techniques and data you wish to compare.

To select multiple files, hold down the Ctrl key while selecting the desired files. To add further data, or to add data from other directoriy or drive, use the "7-7 Add Data to Parallel command.

The scale of the graph will automatically adjust to the individual data. It can be customized by using the Graph Options, Color and Legend, Font commands. This function does not check the type of data.

7-7. Add Data to Parallel

From "Graphics" - "Add Data to Parallel", multiple data can be added to an existing parallel plot, or added data from other directory or drive.

The scale of the graph will be automatically adjusted to the individual data. It can be customized by using the Graph Options, Color and Legend, Font commands. This function does not check the type of data.

It is not available when a parallel plot is not open.

7-8. Replot Parallel

From "Graphics" - "Replot Parallel", the most recent parallel plot is displayed. This is useful, for example, after using the Preseent Data Plot.



Figure 7-6-1. Toolbar



Figure 7-7-1. Toolbar



Figure 7-8-1. Toolbar



7-9. Manage Parallel

From "Graphics" - "Manage Parallel", selected data is replaced. For details on how to use this function, please refer to 7-5 Manage Overlay.

Graphics DataProc Analysis Image: Present Data Plot Present Data Plot New Overlay Plots... Add Data to Overlay... Add Data to Overlay... Replot Overlay Manage Overlay... New Parallel Plots... Add Data to Parallel... Replot Parallel

Figure 7-9-1. Toolbar

7-10. Zoom In

The plot can be enlarged from "Graphics" - "Zoom In".

Once in zoom mode, the mouse cursor appears as an up arrow in the data plot area. To zoom in, left-click and drag the area to be enlarged.

To zoom back out and exit zoom mode, click the Zoom In command or toolbar button again.

The same operation can be performed by clicking on the icon. $\textcircled{\mathbf{Q}}$

 Present Data Plot

 New Overlay Plots...

 Add Data to Overlay...

 Replot Overlay

 Manage Overlay...

 New Parallel Plots...

 Add Data to Parallel...

 Replot Plots...

 Manage Overlay...

 Wew Parallel Plots...

 Add Data to Parallel...

 Replot Parallel

 Manage Parallel...

Graphics DataProc

Analysis

Figure 7-10-1. Toolbar







7-11. Manual Results

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From "Graphics" - "Manual Results", manually set and display peaks, wave potentials, currents, and area. The linear baseline for a peak or wave can be visually examined.

Once in manual mode, the cursor appears as an up arrow in the data plot area. In order to report a peak or wave correctly, you must use the Peak Definition command to define the peak shape as Gaussian, diffusive, or sigmoidal.



Figure 7-11-1. Toolbar

For Gaussian peaks -

The baseline is determined by two points spanning the peak. Left-click at one point, drag to the other point, and release the left mouse button. A vertical line from the peak to the baseline will appear, with the corresponding numerical value reported at the right side of the plot.

-For diffuse peak

The baseline is determined by extending the foot before the peak. Left-click at the foot, drag past the peak potential, and release the left mouse button. A vertical line from the peak to the baseline appears, with corresponding numerical value reported at the right side of the plot. Note that in this case, half of the peak area is reported.

For sigmoidal wave

Two baselines are required. One at the foot of the wave and the other at the plateau portion of the wave. Left-click and drag to draw each baseline. A vertical line will appear connecting the two baselines and crossing the middle of the wave apper, with the corresponding numerical values reported at the right side of the plot.

The same operation can be performed by clicking on the icon. ${}^{I\!\!\!\!\Delta}$



7-12. Peak Definition

From "Graphics" - "Peak Definition", you can define the peak shape as Gaussian, diffuse or sigmoidal. Also, the report peak (or wave) potential, half peak (or wave) potential, peak (or wave) current, and peak area can be defined.



Figure 7-12-1. Toolbar (left), Peak definition (right)

- Peak Shape

Define peak shape as Gaussian, Diffusive, or Sigmoidal. The default peak shape is assigned based on electrochemical technique. This setting can be changed.

- Report option

It can be set to report peak (or wave) potential, hal peak (or wave) potential, peak (or wave) current, and/or peak area.

- Peak or wave search potential range

Adjust the peak potential range to be searched according to the peak wave. For wide peaks or wave, the potential range should be larger, and vice versa. The potential range should include both sides of the peak or wave.

For cyclic voltammetry data, segments can be selected using the Graph Options command in the Graphics menu.

The same operation can be performed by clicking on the icon.



7-13. X-Y Plot

From "Graphics" - "X-Y Plot", create X-Y data and perform linear fitting to the data. After editing the data, click the OK button.

The resulting plot is temporary and will disappear when other data display commands are executed. It can be customized by using the Graph Options, Color and Legend, Font commands.

| | | X-Y Plot | × |
|--------------------|---|-------------------------|----------------------|
| | phics DataProc Analysis | X, Y Data Array Editor: | ОК |
| ₽ E | Present <u>D</u> ata Plot | | Cancel |
| | New <u>O</u> verlay Plots | | Help |
| | <u>A</u> dd Data to Overlay Replot Overlay | | Read |
| | Manage Overlay | | Save |
| | New <u>P</u> arallel Plots | | Overlay Plot |
| | Add Data to Para <u>l</u> lel | | Parallel Plot |
| | Replo <u>t</u> Parallel | | Plot |
| | Manage Parallel | | 1100 |
| Ð, | <u>Z</u> oom In | | |
| $\mathbf{\Lambda}$ | <u>M</u> anual Results | × 🗆 | Linear Curve Fitting |
| ☆ | Pea <u>k</u> Definition | X Axis Title: | Unit: |
| | <u>X</u> -Y Plot | Y Axis Title: | Unit: |
| | Levich and other Plot | Header: | |
| | S <u>e</u> milog Plot | Note: | |
| | Special Plots | No.e. | |

Figure 7-13-1. Toolbar (left), X-Y Plot (right)

X,Y Data Array Editor

Enter XY data points. Use comma or space to separate x and y values, one pair per line.

- Read

Use this command to read data from file.

- Save

Use this command to save data to file. XY titles, units, header, and note will be saved along with the data.

Overlay Plot

Overlays data in the X-Y data array editor. Select the data from the displayed dialog box. To display both data markers and lines, save the data with different names for each and select proper legend with the Color and Legend, command. The scale of the plot will automatically adjust to the data in the XY data array editor. Use the Graph Options command to fix the scale.

Parallel Plot

Plot data in parallel. Select the data from the displayed dialog box. The graph scale is automatically adjusted to the data loaded. Use the Graph Options command to fix the scale.



Plot

Use this command to plot the data entered into the XY Data Array Editor. The scale of the graph is automatically adjusted.

- X, Y Axis Title

Enter a tittle for the X and Y axis.

- Unit

Enter the unit for the X and Y axis.

- Header

Enter the desired title (header) for the data. If it does not displayed on the top of the plot, check the Header box using the Graph Options command.

- Note

Input field for experimental conditions and comments. They will not be displayed on the plot, but will be saved as data information. It is used to review the experiment later.

Linear Curve Fitting

Creates an approximate curve using the least squares method and displays the best fitting line on the plot.



7-14. Levich and other Plot

From "Graphics" - "Levich and other Plot", plot the data according to the selected type.

The resulting plot is temporary and will disappear when other data display command is executed. It can be customized by using the Graph Options, Color and Legend, Font commands.

| Gra | phics DataProc Analysis | | |
|---------|---|---|----------------|
| ₽V I | Present <u>D</u> ata Plot | | |
| | New <u>O</u> verlay Plots | | |
| | <u>A</u> dd Data to Overlay Replot Overlay | | |
| | Manage Overlay | Levich and other Parameters Plot | |
| | New <u>P</u> arallel Plots | Peak Potential Window | OK |
| | Add Data to Para <u>l</u> lel | Ep From (V) | Cancel |
| | Replo <u>t</u> Parallel | Ep To (V) | Help |
| | Manage Parallel | Use current at two Es | Select File |
| € | Zoom In | Type of Plot | ter to report- |
| ∆ ☆ | <u>M</u> anual Results Pea <u>k</u> Definition | C ip vs Scan Rate C Concentration (mol/L) | 0.001 |
| | <u>X</u> -Y Plot | | 4 0.01 |
| | Levich a <u>n</u> d other Plot | C Levich Plot (i-sqrt(rpm) C Electron Transfer No | 1 |
| | S <u>e</u> milog Plot | C Koutecky-Levich Plot O Diffusion Coefficient (cm^2/s) | 5e-006 |
| | Special Plots | | |

Figure 7-14-1. Toolbar (left), Levich and other Plot (right)

- Peak Potential Window

Enter the potential range to search for peaks. Plot using the first peak detected. When creating Levich plot or Koutecky-Levich plot, enter the potential at which the diffusion limiting current is obtained and the potential at the background current in the peak potential window. Check Use current at 2 points Es.

Type of Plot

Choose from Peak Current vs. Scan Rate, Peak Current vs. Square Root of Scan Rate, Peak Potential vs. Logarithm of Scan Rate, Levich Plot, and Koutecky-Levich Plot.

For reversible surface reactions, peak current is proportional to the scan rate.

| For thin layer cells | |
|-----------------------|---|
| Reversible system: | $i_p = \frac{n^2 F^2 v V C_0^*}{4RT}$ |
| Irreversible system: | $i_p = \frac{n\alpha n_a F^2 v V C_0^*}{2.718 RT}$ |
| For surface reactions | |
| Reversible system: | $i_p = \frac{n^2 F^2 v A \Gamma_0^*}{4RT}$ |
| Irreversible system: | $i_p = \frac{n\alpha n_a F^2 v A \Gamma_0^*}{2.718 RT}$ |



For reversible diffusion systems, the peak current is proportional to the square root of the scan rate.

Reversible system:
$$i_p = (2.69 \times 10^5) n^{3/2} A D_o^{1/2} v^{1/2} C_o^*$$
Irreversible system: $i_p = (2.99 \times 10^5) n (\alpha n_a)^{1/2} A D_o^{1/2} v^{1/2} C_o^*$

The peak potential should be independent of scan rate for reversible systems. The changes in peak potential with scan rate indicate a slow reaction rate or chemical complexity.

Forward Reaction
$$(C_r E_r)$$
: $E_{p/2} = E^O - \frac{0.007}{n} - \frac{0.029}{n} \log k_b + \frac{0.029}{n} \log v$

 k_b : Reverse reaction rate constant of the forward reaction

Backward reaction:

$$E_p = E^O + \frac{RT}{nF} \ln \frac{D_O^{1/2}}{D_R^{1/2}} - 0.78 \frac{RT}{nF} + \frac{RT}{2nF} \ln \frac{kRT}{nFv}$$

k : Chemical reaction rate constant

Levich equation:
$$i_L = 0.62 n F A D^{2/3} \omega^{1/2} v^{-1/6} C^*$$

 $\boldsymbol{\omega}$: Angular rotation rate of electrode

Koutecky-Levich equation: $i^{-1} = i_k^{-1} + (0.62nFAD^{2/3}\omega^{1/2}v^{-1/6}C^*)^{-1}$

- Linear Curve Fitting

An approximate curve is created by the least squares method, and the best fitting line is displayed on the plot.

- Intercept = 0

The approximate straight line in the plot passes through the origin.

- Report Parameters

Display the parameters on the plot diagram.

Enter known parameters, select a parameter to report

Select one parameter to calculate from concentration (mol/L), electrode diameter (mm), kinematic viscosity (cm^2/s), electron transfer number, and diffusion coefficient (cm^2/s). Otherwise, enter known parameters.

- Select Files

Select the data to be plotted (CV or LSV data only). Select at least three files with data acquired at different scan speeds.



Operation method of peak variable plot [Plot type: Koutecky-Levich].

1. Enter the potentials at which the diffusion limited current can be obtained and the potential at the time of the background current in the peak potential window, respectively. Check the "Use current at two Es" checkbox.

| Levich and other Parameters Plot | | > |
|----------------------------------|--|----------------|
| Peak Potential Window | | ОК |
| Ep From (V) 0 | Linear Curve Fitting | Cancel |
| Ep To (V) 0.5 | Intercept = 0 | Help |
| Use current at two Es | 🔲 Report Parameter | Select Files |
| Type of Plot | Enter known parameters, select a param | eter to report |
| 🔿 ip vs Scan Rate | C Concentration (mol/L) | 0.002 |
| ◯ ip vs Sqrt (Scan Rate) | C Electrode Diameter (mm) | 4 |
| C Epivs log (Scan Rate) | C Kinetic Viscosity (cm ² /s) | 0.01 |
| C Levich Plot (i-sqrt(rpm) | Electron Transfer No | 1 |
| Koutecky-Levich Plot | O Diffusion Coefficient (cm^2/s) | 6.73e-006 |

Figure 7-14-2. Enter the parameters

- 2. Select Koutecky-Levich Plot from "Type of Plot".
- 3. Select the parameter to be calculated from "Enter known parameters, select a paramete to report". In this case, the "Electron Transfer No." was selected, and other known values were entered.
- 4. Check "Linear Curve Fitting" to plot an approximate straight line. If "Intercept = 0" is checked, the approximate straight line in the plot will pass through the origin. If Koutecky-Levich Plot is selected and Intercept = 0 is not checked (intercept ≠ 0), the calculated activation dominant current ik (A) is displayed on the plot. Check the "Report Parameter" to see each parameter displayed on the plot.



Figure 7-14-3. Parameter display

- 5. Select 3 or more data from file selection.
- 6. The result will be displayed on the plot. The slope of the approximate straight line, intercept, correlation coefficient, and parameters are displayed. The calculated parameter is displayed at the bottom.



7-15. Semilog Plot

From "Graphics" - "Semilog Plot", create a current-voltage semilog plot. This command is useful for data analysis for steady-state responses. It converts the response of a diffusive or peak shapped response by semi-integral or full integral to a sigmoidal curve data analysis, and display a semilog plot.

For a reversible reaction, the intercept of the potential axis is the half wave potential and the slope is 0.059/n V. Deviation from the expected slope indicate a slow kinetic or complexity of the electrode reaction.

The resulting plot is temporary and will disappear when other data display commands are executed. It can be customized by using the Graph Options, Color and Legend, Font commands.

| Gra | phics DataProc Analysis | | |
|-----|--------------------------------|--------------------------------|-----------|
| ₽ | Present <u>D</u> ata Plot | | |
| | New <u>O</u> verlay Plots | | |
| | Add Data to Overlay | | |
| | Replot Overla <u>v</u> | | |
| | Manage Overlay | | |
| | New <u>P</u> arallel Plots | | |
| | Add Data to Para <u>l</u> lel | | |
| | Replo <u>t</u> Parallel | | |
| | Manage Parallel | Current-Potential Semilog Plot | |
| Ð | <u>Z</u> oom In | _ | |
| A | <u>M</u> anual Results | Potential Window | <u>ок</u> |
| ₽ | Pea <u>k</u> Definition | E From (V) 🚺 | Cancel |
| | <u>X</u> -Y Plot | E To (V) | Help |
| | Levich a <u>n</u> d other Plot | | Пар |
| | S <u>e</u> milog Plot | E. Linear Course Fibling | |
| | Special Plots | Linear Curve Fitting | |

Figure 7-15-1. Toolbar (left), Semilog Plot (right)

Potential Window

Enter the potential range to search for peaks. Plot using the detected peaks.

Enter the potential window of the data to be plotted. The potential window should be $\pm 0.059/n$ V of the half wave potential. Data points that exceed the specified potential window will be ignored.

- Linear Curve Fitting

Creates an approximate curve using the least squares method and display the best fitting line on the plot.




7-16. Special Plot

From "Graphics" - "Special Plot", the polarization resistance is calculated by analyzing linear sweep voltammetry data.

The resulting plot is temporary and will disappear when other data display commands are executed. It can be customized by using the Graph Options, Color and Legend, Font commands.

| Gra | phics DataProc Analysis | | |
|------------|----------------------------|------------------------------|----|
| ₽ <u>~</u> | Present <u>D</u> ata Plot | | |
| | New <u>O</u> verlay Plots | | |
| | Add Data to Overlay | | |
| | Replot Overla <u>v</u> | | |
| | Manage Overlay | | |
| | New <u>P</u> arallel Plots | | |
| | Add Data to Parallel | | |
| | Replo <u>t</u> Parallel | | |
| | Manage Parallel | | |
| Ð | <u>Z</u> oom In | Polarization Resistance Plot | × |
| Λ | Manual Results | | |
| ☆ | Pea <u>k</u> Definition | Potential Window OK | |
| | <u>X</u> -Y Plot | E (V, i=0) | i. |
| | Levich and other Plot | Window (V) 0.03 | 1 |
| | S <u>e</u> milog Plot | Help | |
| | Special Plots | | |

Figure 7-16-1. Toolbar (left), Special Plot (right)

- E (V, I = 0)

The potential at which the current value is 0 A is automatically detected and displayed.

- Window (V)

Specify the potential range to be displayed. If the measurement point of 0 A cannot be found, an error message is displayed.

After entering the parameters and clicking OK, a polarization resistance plot is displayed and the polarization resistance and correlation coefficient are calculated.



Figure 7-16-2. Special Plot display example LSV (left), Polarization resistance plot (right)



7-17. Graph Options

From "Graphics" - "Graph Options," set graph heading, display the XY grid, invert the axes, etc.

- Screen

Configure settings for the plot display on the screen. Uncheck the corresponding boxes to hide header, axis, baseline, parameters, or results on screen. The header is the title that appears at the top of the plot and displays what have been entered in the header field. The baseline is drawn to visually define a peak or wave.

- Printer

Uncheck the corresponding box to hide the header, axis, baseline, parameters, or results on printed output.

Grid and Inv

Uncheck the corresponding box to hide the X or Y grid or to disable axis inversion. To invert the axis polarity permanently, use the System command in the Setup menu.

- X, Y Freeze

Check the corresponding box to fix the X or Y axis range. Enter minimum and maximum values in the input fields to the right. Unchecking the box will return to the original scale. If the box is not checked, the contents of the field will not be reflected. Due to the program requirement for an integer number of ticks, an axis may be unable to be frozen at the entered scale.

| Graphics | | DataProc | Analysis | | | | | |
|--------------|---------------------------|---------------------------|----------|--|--|--|--|--|
| ₩ | Present <u>D</u> ata Plot | | | | | | | |
| | New (| New <u>O</u> verlay Plots | | | | | | |
| | <u>A</u> dd D | ata to Over | lay | | | | | |
| | Replo | t Overla <u>v</u> | | | | | | |
| | Mana | ge Overlay | | | | | | |
| | New I | Parallel Plots | | | | | | |
| | Add [|)ata to Para <u>l</u> | lel | | | | | |
| | Replo | <u>t</u> Parallel | | | | | | |
| | Mana | ge Parallel | | | | | | |
| € | <u>Z</u> oom | In | | | | | | |
| \mathbb{A} | <u>M</u> anu | al Results | | | | | | |
| ₩ | Pea <u>k</u> Definition | | | | | | | |
| | <u>х</u> -ү рі | ot | | | | | | |
| | Levich | a <u>n</u> d other | Plot | | | | | |
| | S <u>e</u> mil | og Plot | | | | | | |
| | Special Plots | | | | | | | |
| 8 | <u>G</u> raph Options | | | | | | | |
| | Color and Legend | | | | | | | |
| fF | <u>F</u> ont | | | | | | | |
| e | Сору | to Clip <u>b</u> oar | d | | | | | |

Figure 7-17-1. Toolbar

| Graph Options | | | × | | |
|---|--|--|--|--|--|
| Screen F Header Axis Baseline F Param F Result | Printer V Header Axis Baseline V Param Result | Grid and Inv ✓ × <u>G</u> rid ✓ Y <u>G</u> rid □ × Invert □ Y Invert | OK Cancel <u>H</u> elp | | |
| □ × <u>F</u> reeze 0 □ Y <u>F</u> reeze -5e- □ Y Freeze durin | | Cursor Lock to Data Points C Lock © Free | | | |
| ☐ X <u>I</u> itle: ☐ Y <u>I</u> itle: X <u>S</u> cale: 1 Segments: 1 D <u>a</u> ta: Current | T <u>o</u> : 1 | Unit: | Electrode Area: 1 Current Density E vs Reference Electrode Ref. Electrode: Ag/AgCl CV Time Display | | |
| Hea <u>d</u> er: 0.5 mM Ferricyanide in 0.4M KCl solution <u>N</u> ote: | | | | | |

Figure 7-17-2. Graph Options





Y-axis freeze during measurement

Fix the Y-axis during measurement. If not fixed, the scale is automatically adjusted according to the measured value.

- X, Y Title

The default XY axis title for displayed for each electrochemical technique, but to customize it, check the box and enter the desired title in the field.

Unit

Enter the unit or dimension for the axis title (for example, the current unit is A, the dimension symbol is I, the time is s, and the dimension symbol is T). If X, Y axis title is checked, it will be enabled.

X, Y Scale

Change the plot size by changing the numerical value from the default full size of 1. The input range is 0.1 to 1.

- Segment

Enter the range of segments to be displayed.

- Data

Select the data to be plotted. Available selections depend on the technique.

Cursor Lock to Data Points

This option controls the behavior of the mouse cursor in the plot area. When "Lock" is selected, the cursor will be locked to the data point. Moving the mouse in the X direction, the cursor will move along the data curve. This is particularly useful for identifying data points. When Free is selected, the cursor moves freely in the XY plane.

- Electrode Area

This value is used to calculate the current density.

- Current Density

Check this box to display the current density.

- E vs Reference Electrode

Check this box to add a reference electrode name after the potential axis title.

- Reference electrode

Enter the reference electrode used for the measurement. If "E vs Reference Electrode" box is checked, it will be displayed after the potential axis title.

CV Time Display

Change the X axis from potential to time display, which is only displayed for CV and LSV measurement data.

- Header

Input field for header. To display this header at the top of your plot, check the Header box under Screen and/or Printer options in this dialog box.

Note

Input field for experimental conditions and comments. It will not be displayed on the plot, but will be recorded as data information. It is used to review the experiment conditions later.

You can perform the same operation by clicking the icon. \mathbb{B}

7-18. Color and Legend

From "Graphics" - "Color and Legend", specify the color and legend.

- Color

Set the color of the curves, grid, axes, and background. Curve 0 represents active data and curves 1-9 represent overlayed plots. Click the "Change" button to select the color. For the change of the text color, see 7-19. Font.

- Legend

Specify the type of marker or line for data curves and grid.

- Size

Specify the size of the marker or the thickeness of the line.

- Interval

Change the data interval of the plot. Setting this value to N plots every Nth data point. The default value is 1. This is useful for overlay plots.

- Default

Clicking the "Default" button will reset the plot to its initial state.

| Graphics DataProc Analysis | | | | | | | |
|----------------------------|--------------------------------|--|--|--|--|--|--|
| ₽ N | Present <u>D</u> ata Plot | | | | | | |
| | New Overlay Plots | | | | | | |
| | Add Data to Overlay | | | | | | |
| | Replot Overla <u>v</u> | | | | | | |
| | Manage Overlay | | | | | | |
| | New <u>P</u> arallel Plots | | | | | | |
| | Add Data to Para <u>l</u> lel | | | | | | |
| | Replo <u>t</u> Parallel | | | | | | |
| | Manage Parallel | | | | | | |
| € | <u>Z</u> oom In | | | | | | |
| \mathbb{A} | <u>M</u> anual Results | | | | | | |
| ♣ | Pea <u>k</u> Definition | | | | | | |
| | <u>X-Y Plot</u> | | | | | | |
| | Levich a <u>n</u> d other Plot | | | | | | |
| | S <u>e</u> milog Plot | | | | | | |
| | Special Plots | | | | | | |
| 88 | <u>G</u> raph Options | | | | | | |
| | <u>C</u> olor and Legend | | | | | | |
| fF | <u>F</u> ont | | | | | | |
| 8 | Copy to Clip <u>b</u> oard | | | | | | |

Figure 7-18-1. Toolbar

| Color Selec | tion | | | | | × |
|-------------|-----------|------------|-----|--------|-----------|------------|
| | Color | Legend | | Size | Interval | ок |
| Curve 0: | Change | Solid Line | - | 1 💌 | 1 💌 | Cancel |
| Curve 1: | Change | Solid Line | - | 1 🔻 | 1 💌 | Help |
| Curve 2: | Change | Solid Line | - | 1 💌 | 1 💌 | Default |
| Curve 3: | Change | Solid Line | - | 1 💌 | 1 🔻 | |
| Curve 4: | Change | Solid Line | - | 1 💌 | 1 💌 | Monochrome |
| Curve 5: | Change | Solid Line | - | 1 💌 | 1 🔻 | |
| Curve 6: | Change | Solid Line | - | 1 💌 | 1 🔻 | |
| Curve 7: | Change | Solid Line | • | 1 💌 | 1 💌 | |
| Curve 8: | Change | Solid Line | - | 1 💌 | 1 💌 | |
| Curve 9: | Change | Solid Line | • | 1 💌 | 1 💌 | |
| Grid: | Change | Point | - | | | |
| | Color | | Col | or | Thickness | |
| Bkgnd: | Change | X Y Axis: | | Change | 1 💌 | |
| Color Bar: | Two Color | • | | | | |

Figure 7-18-2. Color selection







7-19. Font

From "Graphics" - "Font", set the font, style, size, and color of the text used on the plot.

Click the change button in Figure 7-19-2 and change the settings from the window shown in Figure 7-19-3.



Figure 7-19-3. Font

The Y axis title should read from bottom to top. However, different printers have been found to follow different character rotation conventions, so rotation angle needs to be adjust to obtain the correct orientation for the printer. Clicking the "Default" button will reset the printer to its initial state. Clicking the "Monochrome" button will change the color of each item to black.

The same operation can be performed by clicking on the icon. $\mathbf{\hat{F}}$



7-20. Copy to Clipboard

From "Graphics" - "Copy to Clipboard", copy the plot displayed on the software to the clipboard. This command can also be used while a measurement or digital simulation is running. It has the same effect as the Alt+ PrintScreen keyboard shortcut.



Figure 7-20-1. Toolbar



Chapter 8. Data Processing Menu

Introduces the functions that can be selected from the data processing menu on the toolbar.

8-1. Smoothing

From "Data processing" - "Smoothing", smooth the currently active data.

| Smoothing | | | | × | |
|-----------|---|---|------------------|---|----------------------|
| | aProc Analysis Sim View Window Smooth Derivatives Integration | Method Selection C Least Square Smo C Fourier Transform S | _ | | OK Cancel Help |
| jdk D | Semiinteg and Semideriv Interpolation Baseline Fi <u>t</u> ting & Subtraction Linear Baseline <u>C</u> orrection | Least Square Point: FT Cutoff (1/s or 1/V): | 7 • 30 | Smooth After Run Override Present Data | |

Figure 8-1-1. Toolbar (left), Smoothing (right)

- Method selection

Select least squares or Fourier transform smoothing option.

- Least Squares Point

Least square smoothing requires an odd number of points between 5 and 49. More points will result in better smoothing, but may also cause distortion.

The program performs least square smoothing using the algorithm of Savitzky and Golay. For more details about this algorithm, please refer to "Smoothing and Differentiation of Data by Simplified Least Squares Procedures", Anal. Chem., 36, 1627-1639 (1964).

FT Cutoff (1/s or 1/V)

Specify the filter cutoff frequency. For a time-based experiment, the Fourier conjugate frequency has units of 1/s or Hz, and for voltammetry, it is 1/V (the number of signal cycles allowed in a potential range of 1 volt). A lower cutoff gives better smoothing, but may cause distortion.

This is done by the D.E. Smith et al. algorithm. For details of this algorithm, refer to "Some Observations on Digital Smoothing of Electroanalytical Data Based on the Fourier Transformation", Anal. Chem., 45, 277-284 (1973).

In general, Fourier transform smoothing is very effective. If the signal band is separated from the noise band, distortion is minimized. However, this method is more time consuming than least-squares smoothing.

- Smooth After Run

Check this box to automatically perform smoothing after a measurement. This function can also be switched from "Control" - "Measurement status".

Override Present Data

Check this box to overwrite the converted data with the currently active data. If unchecked, the converted data will not be overwritten and the original data will be displayed when you select "Preseent Data Plot".



8-2. Derivative

Deriviate the current data from "Data Processing" - "Derivative". The first derivative gives the Y-axis as Y-units/X-units, but the units may not be displayed accurately.

| De | erivative | Х |
|--|---|---|
| DataProc Analysis Sim View Window Smooth ✓ Derivatives Integration Integration ∫d* Semiinteg and Semideriv Intergolation Mark Baseline Fifting & Subtraction Intergolation Intergolation | Derivative Order: 1st Order Derivative OK 1st Order Derivative Cancel 2nd Order Derivative Help 3rd Order Derivative Sth Order Derivative 3rd Order Derivative Least Square Point: 7 Override Present Data | |

Figure 8-2-1. Toolbar (left), Derivative (right)

- Derivative Order

Selects the order of derivatives from 1st, 2nd, 3rd, 4th, and 5th order.

- Least Squares Point

Select an odd number of points between 5 to 49. The more points, the better the smoothing, but can also cause more distortion. Numeric differentiation amplifies high-frequency noise, so a relatively large number of points is required.

The program performs numerical differentiation using the algorithm of Savitzky and Golay. For more details about the Savitzky and Golay algorithm, please refer to "Smoothing and Differentiation of Data by Simplified Least Squares Procedures", Anal. Chem., 36, 1627-1639 (1964).

- Override Present data

Check this box to overwrite converted data with currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.



8-3. Integration

The current data can be integrated from "Data Processing" - "Integration".

After integration, the Y-axis becomes Y-units x X-units. For example, for voltammetry, the Y-axis is AV, and for i-t, the Y-axis is AS (coulomb).

| DataProc Analysis Sim View Window | Integration | × |
|---|-------------------------|--------|
| Smooth <u>Operivatives</u> Integration | | OK |
| ∫d ^{1/2} S <u>e</u> miinteg and Semideriv Interpolation | 🔽 Override Present Data | Cancel |
| Baseline Fi <u>t</u> ting & Subtraction Linear Baseline <u>C</u> orrection | | Help |

Figure 8-3-1. Toolbar (left), Integration (right)

Click OK to perform the integration.

Override Present Data

Check this box to overwrite the converted data with the currently active data.

If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.

Semiintegral and Semiderivative 8-4.

From "Data Processing" - "Semi-Integral" and "Semi-Differential", the current data can be semi-integrated or semi-derivatized.

Semi-derivatives can be used to convert diffusive peaks to a Gaussian peak, improving resolution and facilitates measurement. The diffusive peak is converted to a sigmoidal wave, which becomes a time-independent steady state current plateau. It can be used for polarographic theory data interpretation, including current-voltage semilog analysis.

| | Semi-derivative and Semi-integral | × |
|--|---|----------------------|
| DataProc Analysis Sim View Window ✓ ∑mooth ✓ Derivatives Integration | Order Selection © Semi-Derivative © Semi-Integral | OK Cancel Help |
| Baseline Fitting & Subtraction Linear Baseline <u>C</u> orrection | ✓ Override Present Data | |

Figure 8-4-1. Toolbar (left), Semiintegral and Semiderivative (right)

Order Selection

Select semi-integral or semi-derivative.

Override Present Data

Check this box to overwrite converted data with currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.



8-5. Interpolation

Insert data points into the current data from "Data Processing" - "Interpolation".

| DataProc Analysis Sim View Window | Interpolation | × |
|--|--------------------|--------------|
| Smooth ✓ Derivatives Integration ∫d ⁴ s S <u>e</u> miinteg and Semideriv | Data Insertion 🔀 💌 | OK Cancel |
| Interpolation Baseline Fitting & Subtraction Linear Baseline Correction | | Help |

Figure 8-5-1. Toolbar (left), Interpolation (right)

- Data Insertion

The larger the multiplier, the more points are inserted. If the number of data points exceeds the memory size, a warning will be displayed and the command will terminate. The multiplier will be a power of 2 only, and the Fourier transform will be used.

- Override Present Data

Checking this box overwrites the currently active data with the transformed data. If unchecked, the data will not be overwritten with the transformed data and the original data will be displayed when "Preseent Data Plot" is selected.



8-6. Baseline Fitting & Subtraction

From "Data Processing" - "Baseline Fitting & Subtraction," fit a baseline and subtract the fitting curve from the currently active data. Very useful for peak evaluation. Only works with certain peaks and experimental methods.

If you have multiple segments, such as CV data, select the segment to use from the Graph Options command in the Graph menu.

| | Baseline Fitting & Subtraction | × |
|--|--|--|
| | Two Sides of Peak Foot | ОК |
| | From 0.097 To 0.291 From 4.54534e-0 To 0 From 0 To 0 | Cancel Help View Baseline View Difference |
| | Baseline Fitting Algorithm and Orders | |
| DataProc Analysis Sim View Window | Baseline Fitting Orders: 6 | |
| Smooth | Orthogonal L.S. C Least Square | |
| ✓ Derivatives | | |
| Integration ∫d ^k ² S <u>e</u> miinteg and Semideriv Inter <u>p</u> olation | Save to Override Original Data | |
| Baseline Fitting & Subtraction Linear Baseline <u>C</u> orrection | | |

Figure 8-6-1. Toolbar (left), Baseline Fitting & Subtraction (right)

- Two Sides of Peak Foot

To fit a baseline, you must specify two peak ranges avoiding the peaks. If the fitted results are not ideal, it may be necessary to adjust the peak range or fitting order.

If there are multiple peaks, specify a range for each peak. Up to five can be set. Specify a large potential or time range to cover several peaks.

- Baseline Fitting Algorithm and Orders

For fitting, orthogonal least squares and least squares algorithms are available. The first order stands for linear fitting, aX+b; second order stands for aX2+bX+c fitting.

- Save to Override Original Data

If "No Action" is selected, the original data and the fitted baseline will be plotted and the original data will remain unchanged. If "Difference" is selected, the original data will be replaced by the difference data. When "Baseline" is selected, the fitted baseline data will be saved.

· View Baseline

Plot the original data and the fitted baseline together and evaluate the results. Select "Preseent Data Plot" to display the original data.

To hide the baseline, uncheck Baseline in the "Graph Options " screen.

View Difference

Plot the difference between the original data and the fitted baseline to evaluate the results. Select "Preseent Data Plot" to display the original data.

Click on the icon to perform the same operation.



8-7. Linear Baseline Correction

From "Data Processing" - "Linear Baseline Correction," visually correct the baseline of the currently active data. It corrects the slope of the baseline and shifts the DC level of the curve.

| | Proc Analysis Sim View Window | | Baseline Correction | × | |
|----------|---|--|---------------------|-----------------------|--------|
| r | <u>S</u> mooth <u>D</u> erivatives Integration | | | ОК | |
| , | S <u>e</u> miinteg and Semideriv Inter <u>p</u> olation | | | Override Present Data | Cancel |
| <u> </u> | Baseline Fi <u>t</u> ting & Subtraction Linear Baseline <u>C</u> orrection | | | Help | |

Figure 8-7-1. Toolbar (left), Linear Baseline Correction (right)

To perform the correction, click the OK button and the cursor will change to an up arrow.

To correct the baseline slope, left-click and drag at the start point to define a sloped baseline. When the left click is released, this line is subtracted from the data. X values not covered by the line are not affected.

To shift the DC level, left-click and drag to create a horizontal line that defines the zero. It is not necessary to draw a line covering the entire X-axis range.

If there are multiple segments, such as CV data, select the segment to use from the Graph Options command in the Graph menu.

The Baseline Correction command can be perfored once by starting it once. For multiple corrections, use the command repeatedly.

- Override Present Data

Check this box to overwrite the converted data with the currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.



8-8. Data Point Removing

From "Data Processing" - "Data Point Removing", remove unnecessary data points from the beginning or end of the currently active data.

| Dat | aProc Analysis Sim View Window | | |
|-------------------|---|-------------------------|--------|
| 9. 1 | | | |
| V | <u>D</u> erivatives Integration | Data Point Removing | × |
|]q _r ₹ | S <u>e</u> miinteg and Semideriv Inter <u>p</u> olation | Remove Beginning Points | OK |
| Þ | Baseline Fi <u>t</u> ting & Subtraction Linear Baseline <u>C</u> orrection | Remove Ending Points | Cancel |
| | Data Point <u>R</u> emoving | Override Present Data | Help |
| | Data Point <u>M</u> odifying | | |

Figure 8-8-1. Toolbar (left), Data Point Removing (right)

- Remove Beginning Points

Enter the number of data points to be removed from the beginning of the data. If the number entered is zero, no data will be removed.

- Remove Ending Points

Enter the number of data points to be removed from the end of the data. If the number entered is zero, no data will be removed.

- Override Present Data

Check this box to overwrite converted data with the currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.





8-9. Data Point Modifying

From "Data Processing" - "Data Point Modifying", the data point of the currently active data can be visually changed.

| Dat | taProc Analysis Sim View Window | | |
|--------------|------------------------------------|-------------------------|--------|
| 944) 1944 | <u>S</u> mooth | | |
| N | <u>D</u> erivatives | | |
| | Integration | | |
| Jqka | S <u>e</u> miinteg and Semideriv | Data Point Modifying | × |
| | Inter <u>p</u> olation | | |
| P | Baseline Fitting & Subtraction | | ОК |
| | Linear Baseline <u>C</u> orrection | Segment: 1 | |
| | Data Point <u>R</u> emoving | | Cancel |
| | Data Point <u>M</u> odifying | 🔽 Override Present Data | |
| | Background Subtraction | | Help |
| | Signal A <u>v</u> eraging | | |

Figure 8-9-1. Toolbar (left), Data Point Modifying (right)

To modify a data point, click the OK button, and the cursor will change to an up arrow.

When the cursor is moved in the data display range, a crosshair line will appear at the corresponding position of the X-axis of the cursor. Move the cursor horizontally and left-click at the data point to be changed, drag to move the data point vertically, and release the left-click. The old data point will be erased and the data point will appear at the new position.

- Segment

Select the segment to be changed. The valid range depends on the electrochemical technique used.

- Override Present Data

Check this box to overwrite the converted data with the currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.



8-10. Background Subtraction

From "Data Processing" - "Background Subtraction", the difference between two sets of data can be calculated. For example, first measure with a blank solution and save the data. Next, measure with a sample. Background Subtraction calculates and plots the difference between this data.

When using this command, the data must be of the same experimental type and the same X data array. Select the data to subtract from the dialog box that appears and calculate the difference with the currently active data.

| | | 🞇 Background Subtraction | | | × |
|-------------------|---------------------------------|--|--------------------|-------------------|--------|
| Dat | taProc Analysis Sim View Window | \leftarrow \rightarrow \checkmark \uparrow \square \ll als3325 \rightarrow | Data v さ | 🔎 Search Dat | ta |
| y N | <u>S</u> mooth Derivatives | Organize 🔻 New folder | | | = |
| - V | Integration | Name | Date modified | Туре | Size |
| [d ^k 2 | Semiinteg and Semideriv | Cv1.bin | 10/23/2020 4:54 AM | BIN File | 6 KB |
| 2.4 | Interpolation | Cv2.bin | 10/23/2020 4:54 AM | BIN File | 6 KB |
| ~ | - | 📄 cv3.bin | 10/23/2020 4:55 AM | BIN File | 6 KB |
| 1 | Baseline Fitting & Subtraction | 🖹 cv4.bin | 10/23/2020 5:44 AM | BIN File | 9 KB |
| | Linear Baseline Correction | it1.bin | 10/23/2020 4:57 AM | BIN File | 3 KB |
| | Data Point <u>R</u> emoving | Lsv1.bin | 10/23/2020 4:55 AM | BIN File | 4 KB |
| | Data Point Modifying | Mcv.bin | 10/23/2020 5:47 AM | BIN File | 6 KB |
| | Background Subtraction | Ccpt1.bin | 10/23/2020 4:58 AM | BIN File | 4 KB |
| | Signal Averaging | File name: | Cv.bin v | Data Files (*.bin |) ~ |
| | Mathematical Operation | | | | |
| | Eourier Spectrum | | | Open | Cancel |

Figure 8-10-1. Toolbar (left), dialog box (right)

8-11. Signal averaging

From "Data Processing" - "Signal Averaging", perform signal averaging between the currently active data and saved data. The currently active data is always included in the averaging. It is calculated by simply adding several sets of data and dividing the result by the number of data sets. Data sets whose X values differ from the currently active data will be ignored.



Figure 8-11-1. Toolbar



8-12. Mathematical Operation

From "Data Processing" - "Math Operation", perform mathematical operations on the currently active data. The operations available are addition, subtraction, multiplication, division, exponentiation, logarithm, square root, and reciprocal, and can be applied to both X and Y data values.



Figure 8-12-1. Toolbar (left), Mathematical Operation (right)

- Operation Selection

Select the operation to be applied to the data. If addition, subtraction, multiplication, or division is selected, a second operand must be specified in the appropriate field.

- Data Selection

Select either the X data array or the Y data array as the target of the mathematical operation.

- Operand for Add, Sub, Mul, Divi:

Enter when addition, subtraction, multiplication, or division is selected in operation selection.

- Override Present Data

Check this box to overwrite the converted data with the currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.



8-13. Fourier Spectrum

The Fourier spectrum of the current data can be obtained from "Data Processing" - "Fourier Spectrum".

| | Fourier Spectrum | × |
|---|---|----------------------|
| DataProc Analysis Sim View Window Smooth | C nth comp C 1/sec (Hz) C 1 / V | OK Cancel Help |
| Linear Baseline <u>C</u> orrection Data Point <u>R</u> emoving Data Point <u>M</u> odifying <u>B</u> ackground Subtraction Signal A <u>v</u> eraging Mathematical <u>O</u> peration <u>Fourier Spectrum</u> | Y Expression Magnitude ○ Real ○ Imag Model and a present Data | |

Figure 8-13-1. Toolbar (left), Fourier Spectrum (right)

- X Scale

Select the X data array for the Fourier spectrum from nth comp.(nth component), 1/s(Hz) scale, or 1/V scale. Generally, nth comp. is used. The physical meaning is derived from the experimental parameters.

The 1/s(Hz) and 1/V scales have distinct physical meanings. For time-based experiments, the 1/s(Hz) scale must be used; 1/V is invalid; for voltammetry experiments, the 1/V should be used since the 1/s(Hz) scale may not be valid. CV and LSV can use both the 1/s(Hz) and 1/V scales.

- Y Scale

The Y data array contains the Fourier coefficients and can be either linear or logarithmic scale.

· Y Expression

Select the Fourier spectrum graph display method from Magnitude (amplitude), Real (real part) and Imaginary (imaginary part).



Figure 8-13-2. Example of Fourier spectrum display

- Override Present Data

Check this box to overwrite the converted data with the currently active data. If unchecked, the data will not be overwritten with the converted data and the original data will be displayed when "Preseent Data Plot" is selected.



Chapter 9. Analysis Menu

Introduces the functions that can be selected from the analysis menu on the toolbar.

9-1. Calibration Curve

Create a calibration curve from "Analysis" - "Calibration Curve".

| | Calibration C | urve | | | × |
|---------------------------|---------------|---------------|-------------|----------------|-----------|
| | | Concentration | Peak Height | | OK |
| | Standard 1: | 0 | 0 | | Cancel |
| | Standard 2: | 0 | 0 | | |
| | Standard 3: | 0 | 0 | Slope: 0 | Help |
| | Standard 4: | 0 | 0 | Intercept: 0 | Read |
| | Standard 5: | 0 | 0 | Correlation: 0 | Save |
| | Standard 6: | 0 | 0 | | |
| Analysis Sim View Windo | Unknown: | 0 | 0 | | Calculate |
| Calibration Curve | × Axis Title: | | | X Axis Unit: | Plot |
| Standard Addition | Y Axis Title: | | | Y Axis Unit: | |
| Data File Report | Header: | | | | |
| Time Dependence | Note: | | | | |
| Special Anal <u>y</u> sis | | 1 | | | |

Figure 9-1-1. Toolbar (left), Calibration Curve (right)

- Standard

Enter the concentration and peak height/current obtained from the standard solution.

- Unknown

Enter the peak height of the unknown species to calculate the unknown species concentration.

- X Axis / Y Axis Title

Enter the X-axis and Y-axis title to be plotted.

- X Axis / Y Axis Unit

Enter the X-axis and Y-axis unit or dimensions to be plotted.

- Header

Enter the desired title (header) for the data. If it is not displayed at the top of the graph, check the header box using the Graph Option command.

Note

Enter any comments about the plot. They are not displayed on the graph, but is recorded as data information. It will be used later when reviewing the contents of the experiment.

- Read

Use this command to read data from file.

Save

Use this command to save data to file. XY titles, units, header, and note will be saved along with the data.



- Calculate

Use this command to calculate the slope, intercept and correlation coefficient of the calibration curve. If an unknown peak height is given, an unknown concentration is also calculated.

- Plot

Use this command to plot the calibration curve. The plot will be automatically scaled according the data range.

9-2. Standard Addition

From "Analysis" - "Standard Addition", calculate the concentration of unknown species using the standard addition method. In the standard addition method, first measure the unknown species and record the peak height. Next, the peak height is measured again while adding the standard solution. Usually, the amount added is much smaller than the total volume to maintain sample composition. The increased concentration should be equivalent to the unknown species.

| | Standard Add | lition | | | × |
|--------------------------|--------------------------|---------------|-------------|-----------------|-----------|
| | | Concentration | Peak Height | | ОК |
| | Unknown: | 0 | 0 | | Cancel |
| | Addition 1: | 0 | 0 | Slope: 0 | |
| | Addition 2: | 0 | 0 | | Help |
| Analysis Sim View Windo | Addition 3: | 0 | 0 | Correlation: 0 | Read |
| <u>Calibration Curve</u> | Addition 4: | 0 | 0 | | Save |
| Standard Addition | × Axis Title: | I | | X Axis Unit | Calculate |
| Data File Report | Y Axis Title: Header: | | | Y Axis Unit: | Plot |
| Time Dependence | Note: | | | | |
| Special Analysis | NUCC. | 1 | | | |

Figure 9-2-1. Toolbar (left), Standard Addition (right)

- Unknown

Enter the peak height of the unknown species to allow calculation of its concentration.

- Addition #

Enter the concentration and peak height/current after adding the standard solution.

- X Axis / Y Axis title

Enter the X-axis and Y-axis title to be plotted.

Enter the X-axis and Y-axis unit or dimensions to be plotted.

- Header

Enter the desired title (header) for the data. If it is not displayed at the top of the graph, check the header box using the Graph Option command.

Note

Enter any comments about the plot. They are not displayed on the graph, but is recorded as data information. It will be used later when reviewing the contents of the experiment.

- Read

Use this command to read data from file.

⁻ X Axis / Y Axis Unit



Save

Use this command to save data to file. XY titles, units, header, and note will be saved along with the data.

- Calculate

Use this command to calculate the slope, intercept and correlation coefficient of the calibration curve. If an unknown peak height is given, an unknown concentration is also calculated.

- Plot

Use this command to plot the calibration curve. The plot will be automatically scaled according the data range.

9-3. Data File Report

From "Analysis" - "Data File Report", enter the peak potential range, slope, and intercept of the calibration curve to report the concentration or peak current of the component to be analyzed for the saved data file.

| Data File Report | | | | × | | |
|---------------------------|---------------------------|-------------------|-----------|------------------|------------|--------|
| | | Species 1 | Species 2 | Species 3 | Species 4 | OK |
| | Species: | | | | | Cancel |
| | Ep From (V): | | 0 | 0 | 0 | Help |
| | Ep To (V): Slope: | | 0 | | | Read |
| | Intercept: | 0 | 0 | 0 | 0 | Save |
| Analysis Sim View Windo | Peak Shape: Data Type: | Gaussi Origina | | , | , | Report |
| Calibration Curve | Number of S | | | Report Data Type | | 1 |
| <u>Standard Addition</u> | Concentratio | n Unit: | | Conc. | C Current | |
| Data File Report | Report Filena | ame: | | *S* Conc. | -S Callent | |
| Time Dependence | Header: | | | | | |
| Special Anal <u>y</u> sis | Note: | | | | | |

Figure 9-3-1. Toolbar (left), Data File Report (right)

- Species

Enter the name of the species to be analyzed. Up to four compounds can be searched and reported.

- Ep From and Ep To

Enter the peak potential range. When the program detects peaks, the first peak within the specified range will be selected. The ranges for each species should not overlap.

Slope

Enter the slope of the calibration curve for each species to calculate the concentration of the compound. If the report data format is concentration and the slope is zero, a warning (Slope can not be zero or negative) is displayed.

- Intercept

Enter the intercept of each calibration curve to calculate the concentration of a compound.

- Peak Shape

Select the most appropriate peak shape from Gaussian, Diffusive, and Sigmoidal.





Data type

Select original data, semi-derivative, or first derivative to report the concentration.

Number of Species

Enter the number of species for the data file report.

Concentration Unit

Enter the concentration unit or dimension (e.g. ppm or M).

Report Filename

Enter a filename to save the report text file. If the entered file name exists, an overwrite warning will be displayed. If no filename is specified, the report will be shown but not saved.

Report Data Type -

Either concentration or peak current can be reported.

Header _

Enter the desired title (header) for the data. If it is not displayed at the top of the graph, check the header box using the Graph Option command.

Note

Enter any comments about the plot. They are not displayed on the graph, but is recorded as data information. It will be used later when reviewing the contents of the experiment.

Read

Use this command to read data from file.

Save

Use this command to save data to file. XY titles, units, header, and note will be saved along with the data.

Report -

Use this command to generate the data file report.



9-4. Time Dependence

From "Analysis" - "Time Dependence", generate reports and plots of peak heights and concentrations as a function of time from saved data file. Find the peak heights or concentrations for each data and sort them in chronological order relative to the first experiment.

| | Time Dependence | × |
|---------------------------|---|----------------|
| | Ep From (V): | OK |
| | Ep To (V): 0 | Cancel |
| | Slope: 0 | Help |
| | Intercept: 0 | Read |
| | Peak Shape: Gausian 💌 Report Data Type | Save |
| | | Current Report |
| | Report Name: | |
| | Y Axis Title: Y Axis U | nit: Plot |
| | Header: | |
| | Note: | |
| | Combine Mulitiple Time based Data Files into One Time B | ased File |
| Analysis Sim View Windo | Folder and Base Filename: | |
| Calibration Curve | Number of Files: 10 | Folder Browse |
| Standard Addition | Combine to One Point per File | 1 older browse |
| Data File Report | | |
| Time Dependence | Combine | |
| Special Anal <u>y</u> sis | | |

Figure 9-4-1. Toolbar (left), Time Dependence (right)

- Ep From and Ep To

Enter the peak potential range. Ep From and Ep To cannot be equal. When the program detects a peak, the first peak in the specified range is selected.

- Slope

Enter the slope of various calibration curves to calculate the concentration of a compound. If the report data format is concentration and the slope is zero, a warning (Slope can not be zero or negative) message is displayed.

- Intercept

Enter the intercept of each calibration curve to calculate the concentration of a compound.

- Peak Shape

Select the most appropriate peak shape from Gaussian, Diffuse, and Sigmoid.

- Data Type

Select original data, semi-derivative, or first derivative to report concentration.

Enter a filename to save the report text file. If the entered file name exists, an overwrite warning will be displayed. If no filename is specified, the report will be shown but not saved.

- Report Data Type

Either concentration or peak current can be reported.

- Y Axis Title

Enter the Y-axis title to be plotted.



- Y Axis Unit

Enter the Y-axis unit or dimension to be plotted.

- Header

Enter the desired title (header) for the data. If it is not displayed at the top of the graph, check the header box using the Graph Option command.

· Note

Enter any comments about the plot. They are not displayed on the graph, but is recorded as data information. It will be used later when reviewing the contents of the experiment.

- Combine Multiple Time based Data Files into One Time Based File

Combine multiple timebase files (including CV, LSV, i-t) into one data file. The files to be combined are File1, File2, File3, File4, and numbered after the same file name. Also, they must be ordered by the same technique. Time is calculated based on the year, month, day, hour, minute, and second of the starting point.

- Folder and Base Filename

If multiple timebase files are named File1, File2, File3, File4, ... then enter "File" for the base filename.

- Number of Files

If the files to be combined are File1, File2, File3, File4, File5, the number of files will be 5.

- Folder Browse

Browse to the folder where the timebase files are stored and click on one of the files. Delete the number after the file name displayed in the [Folder name and Base file name] field.

- Combine to One Point per File

Check the checkbox to average each data file into a single point.

- Combine

Combine data files. A text file with the specified filename will appear in the data files folder. Press OK to exit the dialog box.

- Read

Use this command to read data from file.

- Save

Use this command to save data to file. XY titles, units, header, and note will be saved along with the data.

- Report

Use this command to generate the data file report.

- Plot

Use this command to generate the time dependence plot.



Chapter 10. Simulation Menu

Introduces the functions that can be selected from the simulation menu on the toolbar.

10-1. Mechanism

From "Simulation" - "Mechanism", set the reaction mechanism, concentration of each reaction species, kinetic, experimental parameters, and other variables. Each parameter must be set before running the simulation. You can also check the equilibrium concentrations.

| | Digital Simulation | × |
|---------------------|--|-------------|
| | Mechanism Edit Mechanism Select | ОК |
| | A + e = B E | Cancel |
| | E A | Help |
| | EEEE | Read |
| | EC EC | Save |
| | ECE ECEC | Exp Param |
| | Square Schei 🗡 | Kinetics |
| | System under study Diffusive 💌 | Conc |
| | Dimensionless current | Equilibrium |
| Sim View Window | Initial concentration at the equilibrium | Variables |
| Mechanism | Display concentration profile during run | |
| CV <u>S</u> imulate | Conc Range: 1 Dist Range: 1 | |
| CV <u>F</u> itting | Time Delay Loop (usec): 0 | |

Figure 10-1-1. Toolbar (left), Mechanism (right)

- Mechanism Edit

Select from the list of mechanism selections and the corresponding reaction will be displayed. It is also possible to edit by entering directly. Each chemical species uses letters A to Z, where e represents the electron transfer process. Upper and lower case letters are treated as identical and spaces are ignored.

Up to 5 reactions and 6 combinations of chemical species can be simulated.

| Reduction | A + e = B | Dissociation | A = B + C |
|---------------|-----------|-------------------|---------------|
| Oxidation | A - e = B | Combination | A + B = C |
| Isomerization | A = B | General Reactions | A + B = C + D |



Mechanism Select

It can be choose from 10 different mechanisms. Mechanisms not included in the list can be obtained from predefined mechanisms. For example, EEC, ECC, and CEC can be obtained from ECEC by defining the reaction rate parameter to be zero. Defining the non-uniform electron transfer rate ko to zero has no effect on the corresponding electron transfer step. If the forward and reverse rate constants for a chemical reaction are defined to zero, the corresponding chemical reaction is unaffected.

Model 3325 can edit and set the mechanism from "User Input", but cannot operate.

- Dimensionless current

Checking this box, the system will calculate the dimensionless current. This is useful for comparison with other theoretical results. If the box is not checked, currents are calculated according to concentration, electrode area, and timescale.

Initial concentration at the equilibrium

Check this box to calculate the equilibrium concentration and use that value as the initial conditions for the simulation. The equilibrium condition is calculated according to the kinetic parameters and the input concentration. If the box is not checked, the input concentration is used as the initial condition.

- Display concentration profile during run

Check this box to display concentration profiles and voltammograms during the simulation. This is very useful to understand the reaction mechanism.

For the voltamogram, the axis scale reflects the value set in Sensitivity. If the sensitivity is too high, the voltammogram will be displayed as a flat line. If the sensitivity is too low, data points will be scattered. However, the data display will automatically adjust after the run.

For concentration profiles, relative concentrations and relative distances are used. The total concentration for all species is set to 1 by default and the concentrations during simulation are based on this value. The unit distance is set to 6*sqrt(Dt) by default, where D is the diffusion coefficient and t is the total time of any given experiment. To change the concentration and distance ranges, see the next two sections below.

- Conc Range

Enter the concentration range. The input range is 0.001 to 100,000, and the defaults is 1.

- Dist Range

Enter the distance range. The input range is 0.001 to 10, and the default is 1.

- Time Delay Loop

The simulation run time depends on the research question and the type of computer used. If the run time is too short, it will not be able to visualize changes in the concentration profile clearly. By taking a longer time delay loop variable, the simulation changes can be displayed slowly. The parameters range is from 0 to 1 x 10E8.

- Read

Load a *.sim file containing the parameters required for the simulation.

- Save

Saves all commands required for the simulation.

- Exper Param

Set the experimental parameters for the simulation. It can also be set from "Setup" -"Parameter Setting ". The Cyclic Voltammetry dialog box will appear to set the parameters to be used.



Kinetics

Figure 10-1-2. The Potential/ Rate Constants dialog box appears to set electron transfer rate parameters such as standard heterogeneous rate constants, standard redox potentials, and charge transfer coefficients. It also allows to enter the forward and backward rate constant for chemical reactions.

- Conc

Set the diffusion coefficient and concentration of the chemical species. If Diffusive (diffusive system) is selected for the system under study, the Concentration Diffusion Coefficient (Figure 10-1-3), and if Surface (adsorption system) is selected, the Surface Concentration (Figure 10-1-4) dialog box will appear.

- Equilibrium

Figure 10-1-5 The Concentration at Equilibrium dialog box appears, displaying the concentration of each chemical species for any given reaction rate condition.

- Variables

Figure 10-1-6 The Simulation Variables dialog box appears, to enter parameters such as temperature, electrode area, etc.

10-1-1. Potential and rate constants

Enter the kinectic parameters for the CV simulation in this dialog box.

| Potentials and | l Rate Constants | | | × |
|----------------|------------------|------|-----------|--------|
| Reactions | | | | OK |
| A + e = B | ko 🚺 | Eo O | alpha 0.5 | Cancel |
| B = C | kf 0 | kb 0 | | Help |
| | | | | |
| | | | | |

Figure 10-1-2. Potential and rate constants

Depending on the type of reaction, enter up to three parameters.

For charge transfer reactions, enter the rate constant ko, the standard redox potential Eo, and the charge transfer coefficient alpha. For other chemical reactions, specify the forward and reverse rate constants.

Some kinetic parameters may be predetermined. This is because the concentration of n species can be determined by the n-1 reactions and the initial concentration. Since there are more equations than species, some of the equations must be linearly dependent. If the equilibrium constant is determined and assigned arbitrarily, the system will never reach equilibrium.

The software automatically detects this situation and assigns the optimal linearly dependent parameters. Reactions with unknown kinetic parameters are placed at the bottom of the mechanism list and the program automatically assigns them.

If the equilibrium constant involves a chemical reaction, the backward rate constant is determined by the software. In that case, the forward rate constant must be entered.



10-1-2. Concentration and Diffusion coefficients dialog box

When select Diffusive (diffusive system) for the system under study, this dialog box is used to specify the concentration and diffusion coefficients.

| Concentration | and Diffusi | on Coefficie | nts | × |
|---------------------------------|---------------------|--------------|-------------------------------|--------------|
| Reactions A + e = B B = C | Species A: B: | Conc/M | D/(cm2/s) 1e-005 1e-005 | OK Cancel |
| | с: | 0 | 1e-005 | Help |



Enter various concentrations and diffusion coefficients.

10-1-3. Surface concentration dialog box

When Surface (adsorption system) is selected for the system under study, this dialog box is used to specify the surface concentration.

| Surface Concer | ntration | | × |
|---------------------------------|---------------------------|-----------------------------------|----------------------|
| Reactions A + e = B B = C | Species A: B: C: | Conc/(mol/cm2) 0.001 0 0 | OK Cancel Help |

Figure 10-1-4. Surface concentration dialog box

Enter the various surface concentrations.

10-1-4. Concentration at equilibrium dialog box

This dialog box displays the concentration at equilibrium.

| Concentration | at Equilibri | um | × |
|---------------|--------------|---------------|--------|
| Reactions | Species | Concentration | ОК |
| A+e=B B=C | A: | 0.001 | Cancel |
| | B: | 3.53429e-012 | Help |
| | C: | 3.53429e-012 | |
| | | | |
| | | | |

Figure 10-1-5. Concentration at equilibrium dialog box

It displays the equilibrium concentration of each species calculated by CV simulation.





10-1-5. Simulation variables dialog box

| Simulation Variables | × |
|------------------------|--------|
| Temperature (Deg C) 🗵 | OK |
| Electrode Area (cm2) 1 | Cancel |
| Capacitance (uF) 1 | Help |

This dialog box sets parameters used for CV simulation.

Figure 10-1-6. Simulation variables dialog box

- Temperature

Enter the temperature. Thermodynamic and kinetic parameters depend on temperature.

Electrode Area

Enter the electrode area (cm2). The current is proportional to the electrode area for planar diffusion and surface reactions.

- Capacitance

Enter the electrode double layer capacitance (uF). The charging current is added to the total current reported during the simulation.

10-2. CV Simulate

Simulate CV data from "Simulation" - "CV Simulate".

| Sim | View | Window |
|-----|-------------------|--------|
| | <u>M</u> echan | ism |
| (| CV <u>S</u> imu | late |
| | CV <u>F</u> ittir | ng |

Figure 10-2-1. Toolbar

Before running the simulation, set the reaction mechanism, concentration, kinetc parameters, and experimental parameters from the "Mechanism" command.

Next, make sure that the device and computer are properly connected and run the Hardware - Test. After the first fit, you do not need to have the instrument connected anymore.

Select the "CV Simulate" command to run the simulation. Graphics during the simulation can be copied to the clipboard.



10-3. CV Fitting

From "Simulation" - "CV Fitting", parameters are fitted to the existing CV data for evaluation.



Figure 10-3-1. Toolbar

Make sure that the device and computer are properly connected and run the Hardware -Test. After the first fit, you do not need to have the instrument connected anymore. Next, open the CV data to be fitted, which can be either experimental or simulated data.

Selecting the "CV Fitting" command opens the Figure 10-1-1 (right) Digital Simulation dialog box. Click OK to open the Figure 10-3-2 CV Fitting Parameters dialog box.

| Fitting Potential | - | s | ikip 1 | • | Error | 1e-007 | OK | _ |
|-------------------|---------|------------|--------|---------------|-------|------------|-------|---|
| Fic_en(v)[0.5 | | | | | | E . | Cance | 1 |
| Fit_el(v)0 | | | Сар | acitance(uF) | 1 | Fix | Help | |
| | | Potentials | and R | ate Constants | | | | |
| Reactions | | Fix | | | Fix | | Fix | |
| A + e = B | ko 0.01 | | Eo | 0.23 | | alpha 0.5 | | |

Figure 10-3-2. CV Fitting Parameters

- Fitting Potential Range

Initial values are entered automatically. It can also be specified manually.

- Skip

Omit data points to be fitted to save time.

- Error

Fitting ends when the error falls below the set Error parameter.

· Capacitance

Double layer capacitance is also available as a variable or constant, as charging current can also have a significant impact on CV data.

- Potentials and Rate Constants

The reaction mechanism selected in Figure 10-1-1 (right) and the associated potentials and rate constants are displayed. The kinetic parameters do not need to be entered. Initial values are automatically entered, but can also be entered manually if the correct values are known.

If parameters are modified, checking Fix prevents values from being changed during fitting and reduces the time required for fitting since the total number of unknown variables is reduced.



Click OK to execute the fitting. The fitting range is enclosed by a blue line, with the fitting data plotted in green and the input data in red. Fitting errors and elapsed time are displayed in the upper right corner.



Figure 10-3-3. Example of CV fitting execution

The fitting process will end when the specified Error is reached; alternatively, it can be terminated at any time by clicking the Stop button or by pressing the Esc key. The resulting parameters are displayed.



Figure 10-3-4. CV Fitting result



Chapter 11. View Menu

Introduces the functions that can be selected from the view menu on the toolbar.

11-1. Data Information

From "View" - "Data Information", information on the currently active data can be checked.

| | | Data Inform | ation | | × |
|------|--------------------------|--------------------------------|------------------------------------|---|----|
| View | w Window Help | Filename: Source: Model: | CV_s.bin Experiment CHI1225C | Data Proc Performed: Smoothing 1st Derivative 2nd Derivative 3rd Derivative | OK |
| | Data Information | Date: Time: | 6-Sep-2022 | 4th Derivative 5th Derivative | |
| | Data <u>L</u> isting | ROM Vers: | 20.01 | Integration Semi-Derivative Semi-Integral | |
| | <u>E</u> quations | Prog Vers: Serial #: | 22.05 J2728 | Interpolation Baseline Correction Data Point Removing | |
| | <u>C</u> lock Toolbar | | , | Data Point Modifying Bkgnd Subtraction Signal Averaging X Math Operation | |
| | Toolbar Options | Header: | | | |
| | | | | | |

Figure 11-1-1. Toolbar (left), Data information (right)

- Filename

Displays the name of the currently opened file. Displays "*_Unsaved" if the file has not been saved.

- Source

Displays the Experiment or Simulation.

- Model

Displays the model name of the instrument used to acquire the data.

- Date

Displays the date the data was acquired.

- Time

Displays the time the data was acquired.

- ROM Vers

Displays the firmware version

- Prog Vers

Displays the program version.

- Serial #

Displays the serial number of the instrument from which data was acquired.

- Data Proc Performed

Displays the data processing performed on the open file.

- Header

Displays the contents of the header entered in the graph option.

- Note

Displays the contents of the note entered in the graph option.



11-2. Data List

From "View" - "Data List", the experimental conditions and results of the currently active data are displayed. The format can be changed from "File" - "Text File Format".

| | | Data List | × |
|-----|-------------------------|---|----------|
| | | Sept. 6, 2022 13:52:32 Cyclic Voltammetry File: cv_s.bin Dotals Source: Experiment Instrument Model: CHI1225C Header: Note: | <u> </u> |
| | | Init E (V) = 0.6 High E (V) = 0.6 Low E (V) = 0.1 Init P/N = N Scan Rate (V/s) = 0.1 Segment -2 Sample Interval (V) = 0.001 Quiet Time (sce) = 2 Sensitivity (A/V) = 1e-5 | |
| Vie | w Window Help | Results: | |
| | Data Information | Channel 1: Segment 1: | |
| | Data <u>L</u> isting | Ep = 0.241V ip = 1.104e-5A Ah = 7.057e-6C | |
| | Equations | Segment 2: Ep = 0.305V ip = -1.073e-5A | |
| | <u>C</u> lock | Ah = -6.942e-6C Potential/V, Current/A | |
| ~ | Toolbar | 0.600, -2.824e-7 0.599, -2.432e-7 0.598, -2.278e-7 | |
| | Toolbar <u>O</u> ptions | 0.596, -2.298-7 0.596, -2.127e-7 | |
| ~ | <u>S</u> tatus Bar | < · · · · · · · · · · · · · · · · · · · | |

Figure 11-2-1. Toolbar (left), Data list (right)

11-3. Equations

From "View" - "Equations", opens the help browser to view equations related to the electrochemical technique.

For details of the theoretical formula, see "Electrochemical Methods," A.J. Bard and L.R. Faulkner, Wiley, New York, 1980.

11-4. Clock

 View
 Window
 Help

 Data
 Information...

 Data
 Listing...

 Equations...

 Clock...

 ✓
 Ioolbar

 Toolbar

 ✓
 Status Bar

Figure 11-3-1. Toolbar

From "View" - "Clock", the current date and time are displayed.

| Vie | w Window Help | | | |
|---|-------------------------|---|-------------------|----|
| | Data Information | | | |
| | Data <u>L</u> isting | | | |
| | Equations | Г | | |
| | <u>C</u> lock | | Clock | × |
| Image: A set of the set of the | Toolbar | | Date: 11-0ct-2022 | OK |
| | Toolbar <u>O</u> ptions | | Time: 16:54:17 | |
| ~ | <u>S</u> tatus Bar | | rine. [10:04:11 | |

Figure 11-4-1. Toolbar (left), Clock (right)



11-5. Toolbar Options

Customize the toolbar by selecting "View" - "Toolbar Options" - "Add or Remove Buttons".

| | Customize | × |
|--|---|-------|
| | Commands Toolbars Menu Options Categories: Commands: File New Setup Open Control Close DataProc Save As | |
| ew Window Help | Sim Delete View Retrieve Window Help Update Instrument Program New Menu List Data File All Commands Convert to Text | ~ |
| Data Information Data Listing Equations Clock Toolbar | Description: | |
| Toolbar Options Add or Remove Buttons Status Bar | | Close |

Figure 11-5-1. Toolbar (left), Toolbar options (right)

With the dialog box shown in Figure 11-5-1 (right) open, it can easily add, delete, or rearrange buttons by dragging and dropping the buttons to be customized.

| | ntrol Graphics DataProc Analy | sis Sim View Window Help 記氏水谷智川作園 ※水豚グ | Electrode | C | × • • × |
|--------------------|---|---|-----------|---|------------|
| | Customize Commands Toolbars Menu Categories: File Setup Control Graphics DataProc Analysis Sim View Window Help New Menu All Commands Description: Other Controls | Options Commands: M Macro Command Open Circuit Potential Filter Setting Cell Step Functions Preconditioning Rotating Disk Electrode Other Stripping Mode | × | | |
| For Help, press F1 | | cv | | | |

Figure 11-5-2. Example of customization

Pressing the Reset button on the toolbar tab restores the default toolbar.

NOTE

- 1. If the toolbar does not display properly, restart the software.
- 2. Toolbar settings are saved in an ini file. Note that editing the ini file manually may cause unexpected behavior.



Chapter 12. Help Menu

Introduces the functions that can be selected from the help menu on the toolbar.

12-1. Index

Open the Help menu from "Help" - "Index", to check how to handle each function.



Figure 12-1-1. Toolbar (left), Index (right)

Help functions can also be used from the icon. \mathbb{N} Click on the icon \mathbb{N} , and the cursor will change to " $\mathbb{Q}_{\mathbb{P}}$ ". Clicking each command in this state will displays the corresponding help topic.

12-2. About ALS3325

From "Help" - "About ALS3325", software version and copyright information are displayed.

| | | | About ALS 3 | 3325 | × |
|------|-----------------------|----|--------------|-------------------------------------|----|
| | | | ALS | ALS 3325 Bi-Potentiostat | OK |
| | | | ⊂ CHI | Version 22.06 10/23/2022 | |
| | | | | Distribution: 1994 - 2022 BAS Inc. | |
| Hel | | | | 1310033 1-28-12 Mukojima, Sumida-Ku | |
| iici | | | | E-mail: sales@bas.co.jp | |
| | Help Topics | F1 | | | |
| | <u>O</u> ld Help | | | | |
| Ю | <u>A</u> bout ALS3325 | | | | |
| | | | L | | |

Figure 12-2-1. Toolbar (left), About ALS3325 (right)



Chapter 13. Appendix

13-1. Connection with SEC2020 spectrometer system

How to connect the Model 3325 Bipotentiostat to the SEC2020 is introduced.

Firmly insert the CB-VS Remote cable (Pin 15) into the cell control terminal of Model 3325 and fix it with the thumbscrews. Check the labels on the pins where the wires come out and connect K (Pin#14, Spectrometer Control) and G (Pin#5, Ground) to the Trigger In (brown) and Ground (black) pins of the trigger/light source control cable supplied with the SEC2020, respectively.

SEC2020 Spectrometer system SEC2020 Model 3325 Spectrometer system **Bipotentiostat** Trigger/Light source \mathbf{v} control cable 111 Trigger In (brown) Ground (black) Trigger In(brown) K(Pin#14) G(Pin#5) Ground(black) CB-VS Remote cable (15pin)

For Model 3325 pin assignments, see 3-6. Cell control terminal.

Figure 13-1-1. SEC2020 spectrometer system (left), connection example (right) Table 13-1-1. SEC2020 Pin assignments

| CB- | VS Remo | te cable (15 pin) (013818) | SEC2020 Trigger/Light source control cable |
|-------|---------|----------------------------|--|
| Label | Pin # | Function | |
| к | 14 | Spectrometer Control | Trigger In (brown) |
| G | 5 | Ground | Ground (black) |

Note

- 1. Please also refer to the instruction manuals for the SEC2020 Spectrometer system and CB-VS remote cable (15 pin).
- 2. If you turn on the Model 3325 before connecting the trigger/light source control cable, it will not operate properly. Please turn on the power after completing the device setting.



13-2. Connection with RRDE-3A Rotating Ring Disk Electrode Device Ver.3.0

How to connect the Model 3325 Bipotentiostat to the RRDE-3A is introduced.

Firmly insert the CB-VS remote cable (15 pin) into the 15 pin cell control terminal of the Model 3325 and fix it with the thumbscrews. Check the labels on the pins where the wires are attached, and for the rotation control, connect R (Pin#4, RDE) and G (Pin#5, Ground) to the remote terminals IN and GND (right next to IN) on the back of the RRDE-3A, respectively. If also the control gas purge is required, connect P (Pin#13, Purge) to PURGE and connect GND (right next to IN) and GND (right next to PURGE) with a jumper.

For Model 3325 pin assignments, see 3-6. Cell control terminal.



Figure 13-2-1. Connection example of RRDE-3A and Model 3325

| CB- | VS Remo | te cable (15 pin) (013818) | RRDE-3A |
|-------|---------|----------------------------|--------------------------|
| Label | Pin # | Function | Remote terminal |
| R | 4 | RDE | IN |
| G | 5 | Ground | GND (right next to IN) |
| Р | 13 | Purge | PURGE |

Table 13-2-1. RRDE-3A Pin assignments

Note

1. Please also refer to the instruction manual for the RRDE-3A rotating ring disk electrode apparatus and CB-VS Remote cable (15 pin).



13-3. Troubleshooting

| Trouble | Cause | Solution |
|---|--|--|
| | Power is not turned on | Connect the USB cable [see 3-3] |
| | Defective USB cable | Check and replace the USB cable |
| Communicaton failure | USB driver is not installed properly | Install the USB driver [see 3-2] |
| Communication failure | Communication ports do not match | Set the communication port from "Setup" - "System Setup " |
| | Computer problem | Check on another computer |
| | Static electricity | Restart the computer and reconnect the USB cable |
| Application error | | Restart the computer and software |
| Hardware test erro | | Repeat the test several times, record the details of the error, and contact the sales agent [see 5-4]. |
| No current response | Cell cable not connected, cable damaged | Check the cell cable |
| | High impedance of reference electrode | Check if there are air bubbles inside the reference electrode or in the liquid junction |
| Lots of noise in the data | Electrically noisy environment | Use Faraday cage |
| Lots of hoise in the data | Weak signal | Increase the sensitivity scale and set the filter |
| | Computer problem | Check on another computer |
| Recorded data out of range | High sensitivity scale | Set the sensitivity scale lower |
| Recorded data out of range | Hardware problems | Perform a hardware test [see 5-4] |
| Overflow error | High sensitivity scale | Set the sensitivity scale lower |
| Simulation and fitting programs do not run | Device not recognized | Connect the device and run the hardware test [see 10-2 and 10-3] |