

Introduction to spectroelectrochemistry and basic measurement examples

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1. Introduction

- **What is Spectroelectrochemistry(SEC)?**

It is a measurement which combined the spectroscopy measuring method with the electrochemical measuring method simultaneously.
- **Background**

It is often difficult to understanding the phenomenon that actually occurs at the electrode / solution interface simply by measuring the relationship between the electric current and the amount of electricity and the electric potential.

Therefore, spectroelectrochemical methods have been developed.

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1. Introduction

- **History**

In 1954, Shikata¹⁾ has said that “ (polarography and electromagnetic absorption spectrum) **research must be interconnected** ”.

And Foreseeing the importance and development of spectroelectrochemistry.

In 1964, Kuwana²⁾ group announced in-situ measurement of the product performed using a transparent electrode.

Development and improvement were made by many researchers then.

UV-VIS absorption spectroscopy is the most well-known method.

And with the development of spectroscopy technique, such as reflection, Raman, EPR spectroscopy are applied .

1) **Masuzo Shikata**, One of the founders of polarography.
 2) **Kuwana, T.**, Darlington, R. K., and Leedy, D. W. 1964, Anal. Chem. 36, 2023. "Electrochemical Studies Using Conducting Glass Indicator Electrodes".

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2. Principle and Features

■ Principle

Techniques on which the SEC is based are:

Electrochemistry, which studies the interaction between electrical energy and chemical changes. This technique allows us to analysis reactions that involve electron transfer processes (redox reactions).

Spectroscopy, which studies the interaction between electromagnetic radiation and matter (absorption, dispersion or emission)

The diagram consists of two overlapping circles. The left circle is blue and labeled 'Spectroscopy'. Inside it are the terms: UV-VIS, IR, Luminescence, Raman, and EPR. The right circle is orange and labeled 'Electrochemistry'. Inside it are: Chronoamperometry, Chronopotentiometry, and Voltammetry. The intersection of the two circles is labeled 'SEC' in red.

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2. Principle and Features

■ Features

By SEC measurement we can know

- The reaction on the electrode surface, the interface of an electrode and solution, and the electronic state of a molecule.
- The absorbance of reactant, product and mediator.

■ Applications

- Thereby, to a substance that difficult analyzed by electrochemically, the redox potential can also be computed from the absorbance, if the reactants or products have spectrum characteristic.
- Identification of the molecular species, when an electrode system is at an equilibrium situation or a stationary state.
- The chemical species near electrodes can be identified by absorbance and spectrum.

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3. Typical Spectroelectrochemical techniques

3.1 Typical UV-VIS absorption SEC

Transmission

A. Conventional cell OTE B. Thin-layer cell <0.2mm C. Long optical path thin-layer cell

Reflection

D. Internal reflection E. Specular reflection

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3. Transmission method

3.2 Electrode for SEC

The most common transparent electrode is glass or quartz coated with a conductive film of oxide or metal.
Grid or porous electrodes are also used as transparent electrodes.

Indium-Tin Oxide, ITO

Pt (Au) mesh electrode

Grid electrode (Au, Pt, GC)

The light transmittance comparison a quartz glass as a reference.

*** Reference data: The light transmittance is 50-55% for Platinum mesh electrode.**

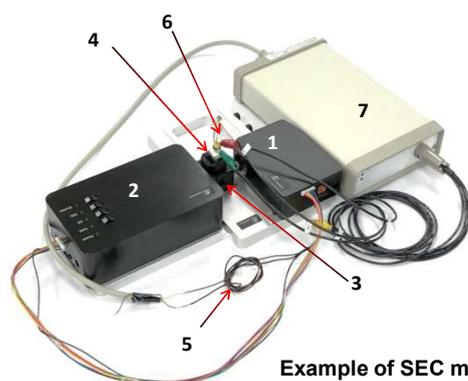
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4. Combination method

4.2 Actual Instrument setup

Electrochemistry, Model 2325 Bi-Potentiostat output external TTL signal.

Spectroscopy, SEC2020 spectrometer system receive external Input signal and acquire spectrum.



4. SEC-CT Thin Layer Quartz Glass Spectroelectrochemical cell kit (Pt)

Example of SEC measurement setup using Model 2325 Bi-potentiostat and SEC2020 spectrometer system



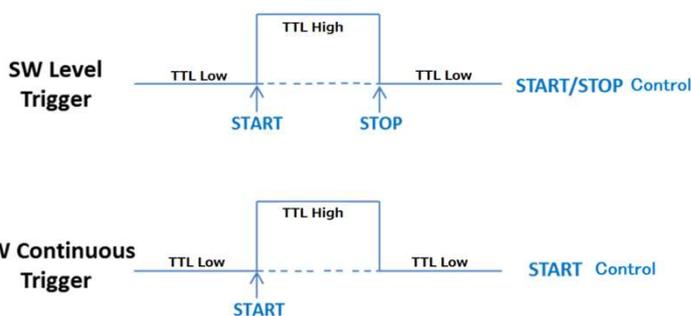
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4. Combination method

4.3 Trigger Mode

SEC2020 spectrometer system offer you two ways for synchronized measurement.



Model 2325 Bi-potentiostat can output TTL signal as the mode of SW Level Trigger



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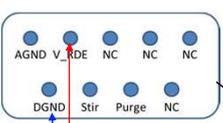
4. Combination method

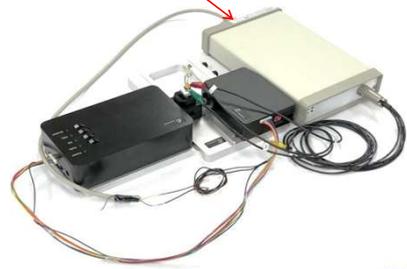
4.3 Connection for synchronized measurement

I/O port of SEC2021 Spectrometer



I/O port of Model 2325





Example of connection for synchronized setup using Model 2325 Bi-potentiostat and SEC2020 spectrometer system



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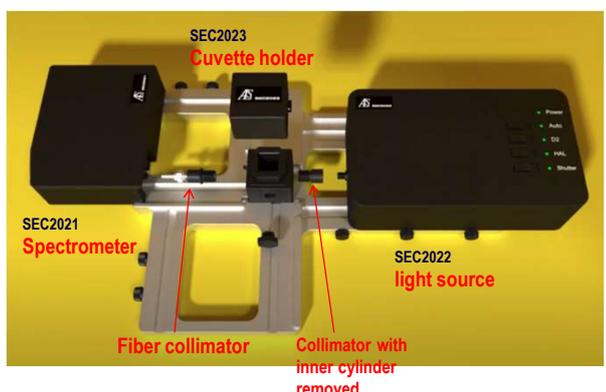
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5. Create an Absorbance measurement

5.1 Absorbance measurement setup

Setup of SEC2020 spectrometer system





SEC2020 system setup



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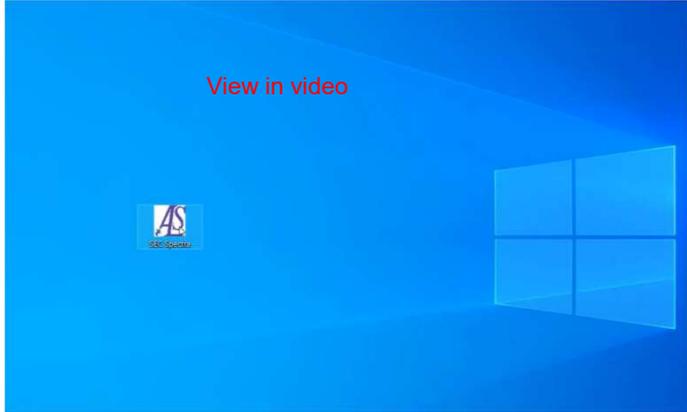


5. Create an Absorbance measurement

5.2 Preparation for absorbance measurement

Connect SEC2020 to PC, insert a cuvette including reference solution into the cuvette holder.
Start software SEC Spectra, **Spectrum_0** is created automatically.

[View in video](#)



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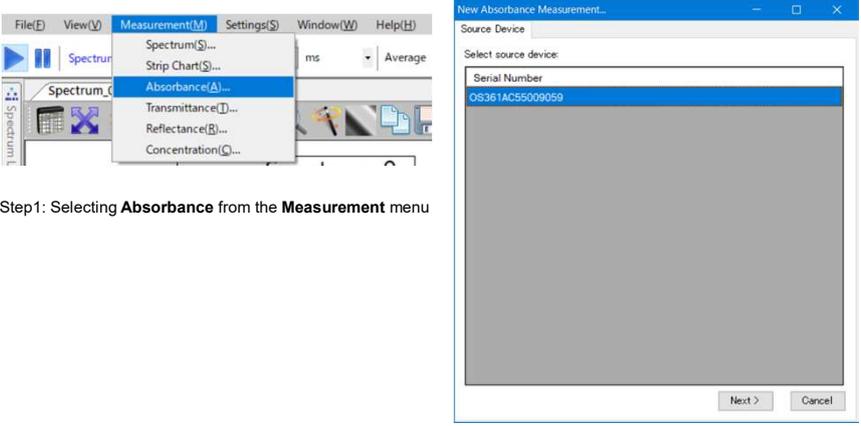
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5. Create an Absorbance measurement

5.2 Preparation for absorbance measurement

Set up of software SEC Spectra



Step1: Selecting **Absorbance** from the **Measurement** menu

Step2: On the **Source Device** window, click **Next**.

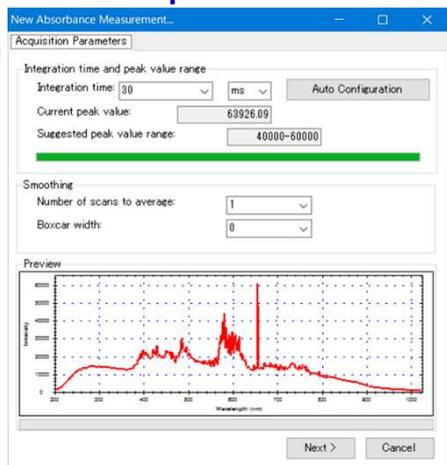
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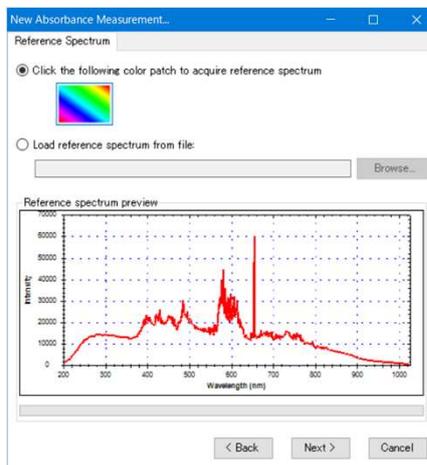
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5. Create an Absorbance measurement

5.2 Preparation for absorbance measurement



Step3: Specify the **integration time**, **average**, **Boxcar width**, click **Next**.

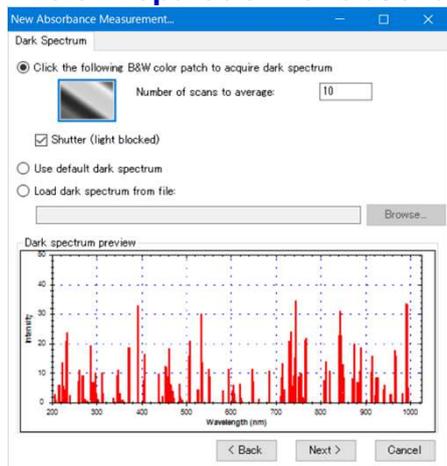


Step4: Click on the color patch to acquire the live spectrum as the **reference**, click **Next**.

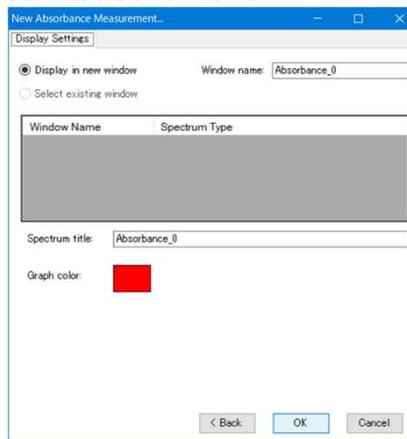


5. Create an Absorbance measurement

5.3 Preparation for absorbance measurement



Step5: Click on black-and-white patch to acquire the **dark** spectrum, click **Next**.

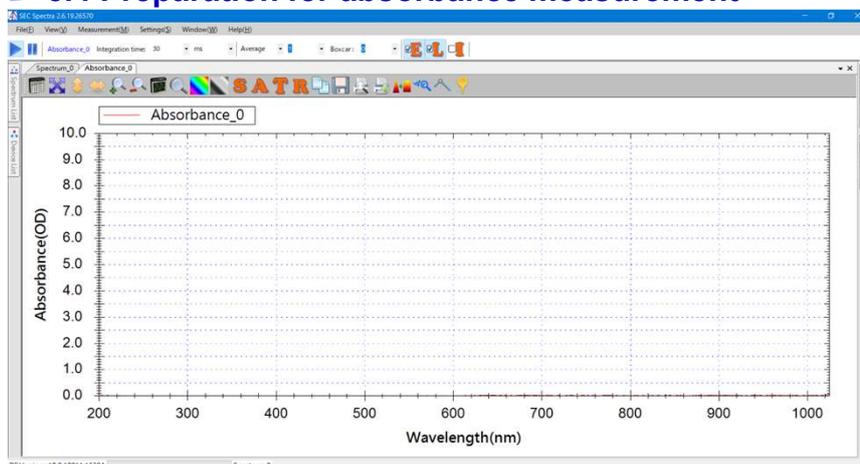


Step6: specify the name for a new absorbance window, spectrum title and color for the graph, click **OK**.



5. Create an Absorbance measurement

5.4 Preparation for absorbance measurement



Step7: Newly created absorbance graph.

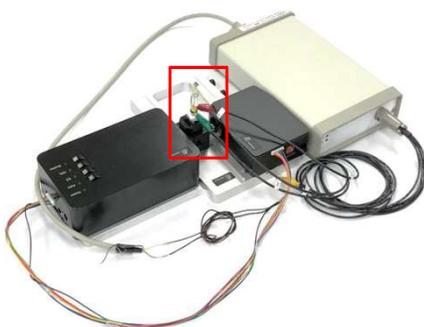


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5. Create an Absorbance measurement

5.5 Absorbance measurement



Normally, here you need to replace the reference solution by a sample solution.

In this case, we replace to a **potassium ferricyanide solution**.

Note:

If you set the **potassium ferricyanide solution as reference**, then, you can view the absorbance change (Δ Absorbance) during electrolysis.



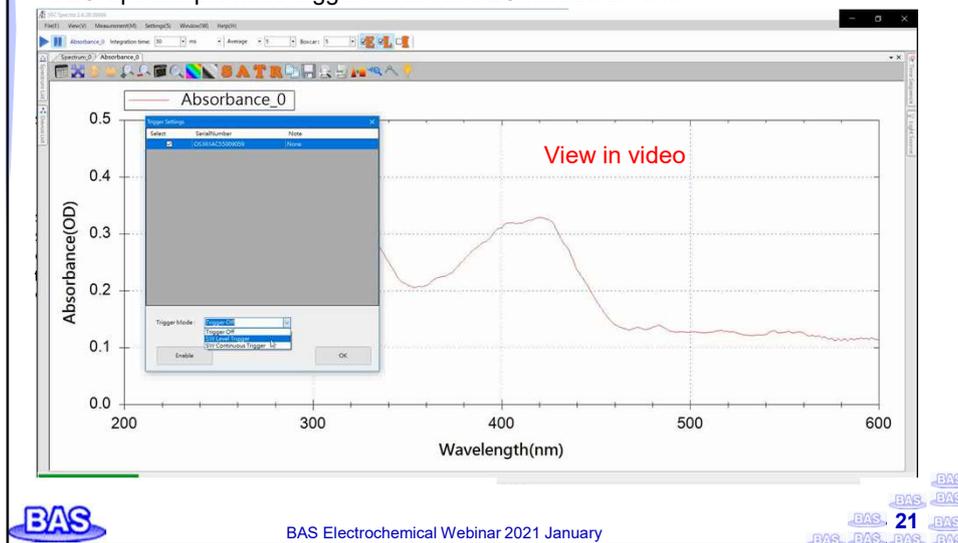
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6. Trigger mode setting

6.1 Preparation of trigger mode

SEC Spectra provides trigger mode for SEC measurement.



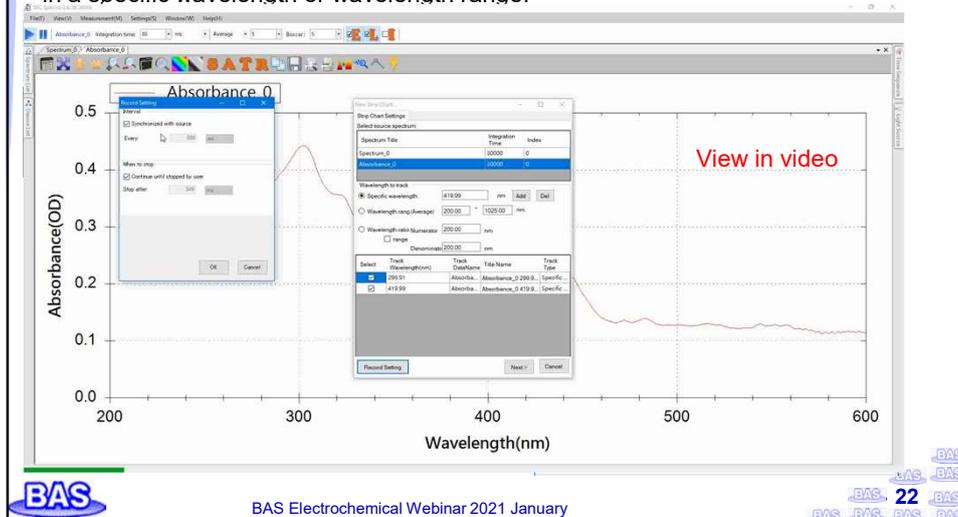
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7. Example of Strip Chart Record

7.1 Recording Specific Wavelengths

SEC Spectra provides a Strip Chart for continuous observation of numerical changes in a specific wavelength or wavelength range.



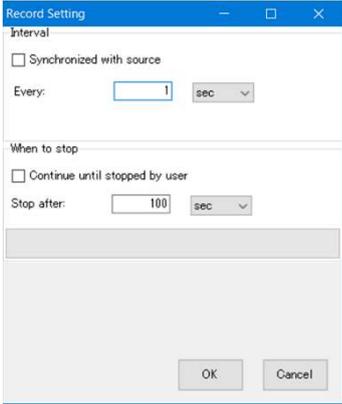
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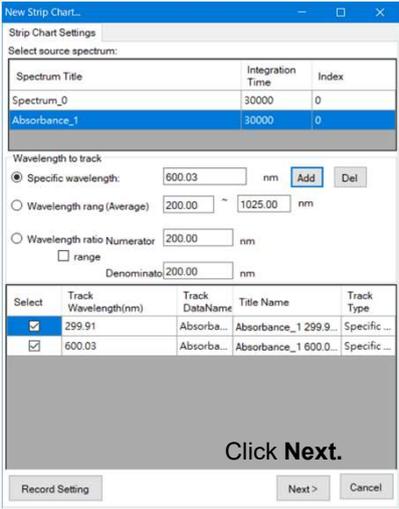
7. Example of Strip Chart Record

7.2 Recording Setting of Strip Chart

Step1: Set the data acquisition Interval (Every 1sec here).
 Step2: **When to stop.** (Stop after 100 sec)
 Click **OK**, return to previous window



The Record Setting dialog box has two sections. The first section, 'Interval', has a checkbox for 'Synchronized with source' which is unchecked. Below it, 'Every:' is set to '1' with a 'sec' dropdown. The second section, 'When to stop', has a checkbox for 'Continue until stopped by user' which is unchecked. Below it, 'Stop after:' is set to '100' with a 'sec' dropdown. 'OK' and 'Cancel' buttons are at the bottom.



The New Strip Chart dialog box has several sections. 'Strip Chart Settings' includes a table for 'Select source spectrum:' with columns 'Spectrum Title', 'Integration Time', and 'Index'. The table contains two rows: 'Spectrum_0' (30000, 0) and 'Absorbance_1' (30000, 0). Below this is 'Wavelength to track' with three radio buttons: 'Specific wavelength:' (selected, 600.03 nm), 'Wavelength rang (Average):' (200.00 ~ 1025.00 nm), and 'Wavelength ratio Numerator:' (200.00 nm). A 'Denominator:' field is set to 200.00 nm. At the bottom is a table with columns 'Select', 'Track Wavelength(nm)', 'Track DataName', 'Title Name', and 'Track Type'. Two rows are checked: 299.91 nm and 600.03 nm. A 'Click Next.' button is overlaid on the bottom half of the dialog.

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7. Example of Strip Chart Record

7.2 Recording Setting of Strip Chart

Step1: Set window name, click **OK** to create a new **Strip Chart** window.



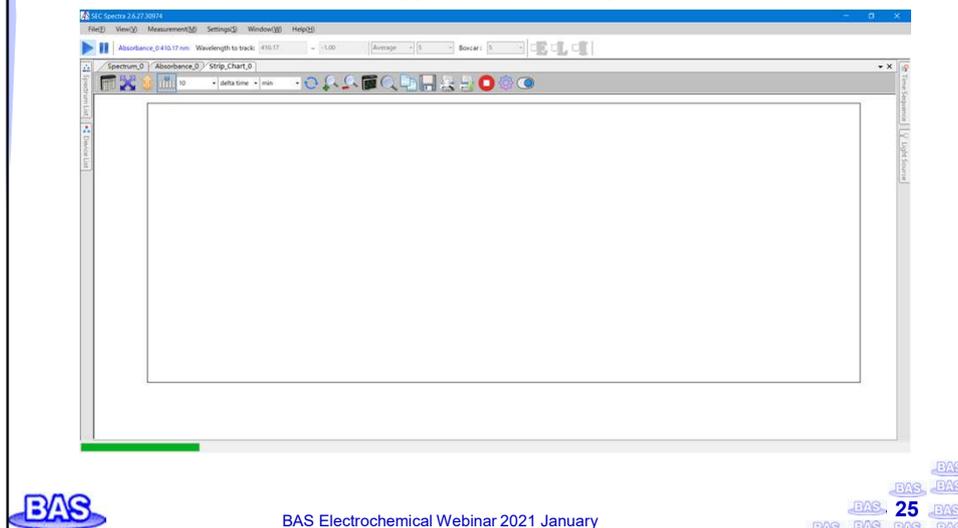
The New Strip Chart dialog box shows 'Display Settings'. The 'Display in new window' radio button is selected, and the 'Name:' field contains 'Strip_Chart_0'. The 'Select existing window' radio button is unselected. Below is a table with columns 'Window Name' and 'Spectrum Type', which is currently empty. '< Back', 'OK', and 'Cancel' buttons are at the bottom.

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7. Example of Strip Chart Record

7.3 Recording window of Strip Chart

As the spectrometer is set to trigger mode, the signal in the window is stopped.



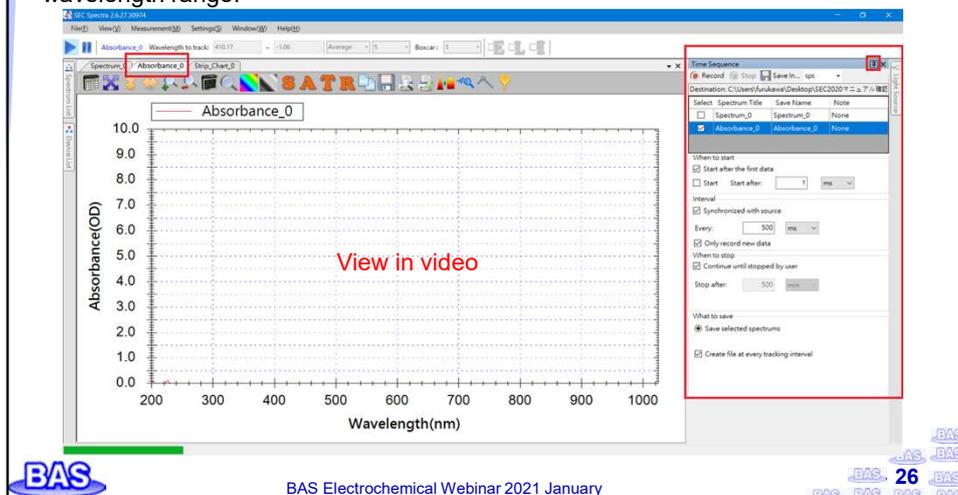
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8. Example of Time Sequence Record

8.1 Settings of Time Sequence Measurement

SEC Spectra provides a Time Sequence function that records changes in entire wavelength range.



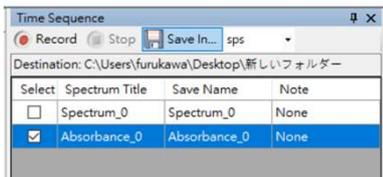
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8. Example of Time Sequence Record

8.2 Settings of Time Sequence Measurement

Step2: Specify the folder to **save** the recorded data.



Step3: Specify the **start time**. Here we use the default setting **Start after the first data**

Step4: Set interval (**Every 5 sec**) for the recording

Step5: Next, specify the stop time (**after 100 sec**).

Step6: Whether **Create file at every data tracking interval**

Step7: Press the **Record** button to start the record.



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9. Example of TTL signal output setting

9.1 External device TTL signal output setting

Set the Model 2325 to output TTL High level signal during the measurement.

Model 2325 SETUP screen



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9. Example of TTL signal output setting

9.2 Set Amperometry measurement conditions

Model 2325 Main screen
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	A	B	C
Potential (V)	0.094	4.00E-5	-4.38E-11
Current (A)	0.334	-6.38E-11	-4.90E-11

10. Start SEC measurement

10.1 Run SEC measurement

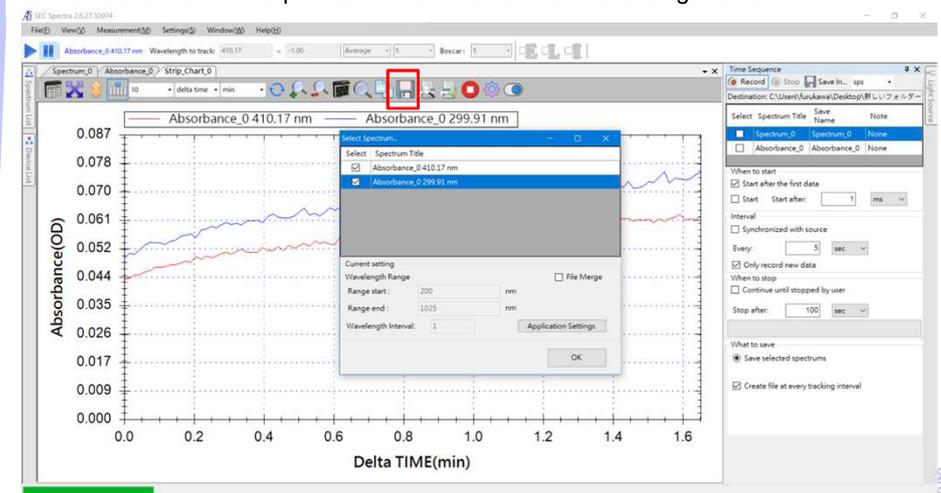
Click Run of Model 2325 to start Amperometric i-t, in the same time output TTL High level signal during the measurement.

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10. Start SEC measurement

10.2 Save Strip Chart record results

click on save icon in "Strip Chart" window to save the recording results.



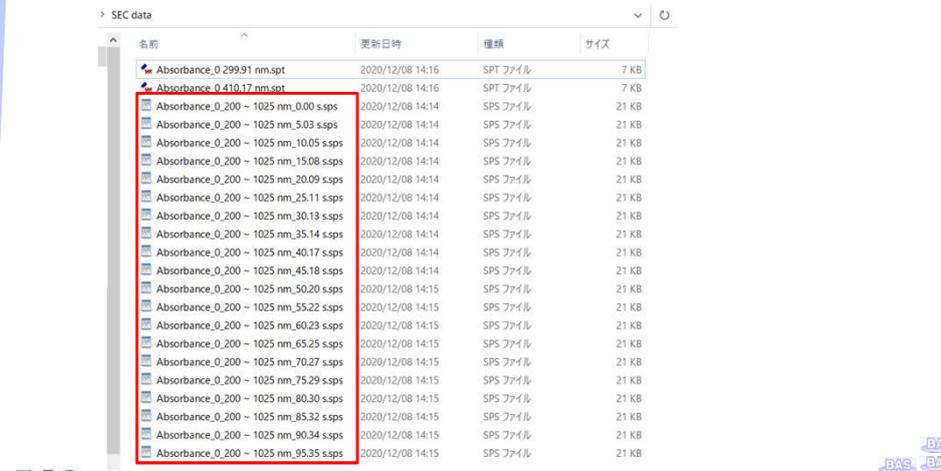
The screenshot shows the 'Strip Chart' window with a 'Save' icon highlighted by a red box. The main window displays a graph of Absorbance (OD) vs Delta TIME (min) for two wavelengths: 410.17 nm and 299.91 nm. A 'Select Spectra' dialog box is open, showing the selected spectra and current settings.

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10. Start SEC measurement

10.3 Saved TimeSequence record results

The recorded results are saved in a preset folder.



The screenshot shows a file explorer window displaying the contents of the 'SEC data' folder. The files listed include:

名前	更新日時	種類	サイズ
Absorbance_0_299.91 nm.spt	2020/12/08 14:16	SPT ファイル	7 KB
Absorbance_0_410.17 nm.spt	2020/12/08 14:16	SPT ファイル	7 KB
Absorbance_0_200 ~ 1025 nm_0.00 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_5.03 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_10.05 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_15.08 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_20.09 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_25.11 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_30.13 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_35.14 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_40.17 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_45.18 s.sps	2020/12/08 14:14	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_50.20 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_55.22 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_60.23 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_65.25 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_70.27 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_75.29 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_80.30 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_85.32 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_90.34 s.sps	2020/12/08 14:15	SPS ファイル	21 KB
Absorbance_0_200 ~ 1025 nm_95.35 s.sps	2020/12/08 14:15	SPS ファイル	21 KB

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10. Start SEC measurement

10.3 Saved TimeSequence record results

You can load the data to compare the results from **File>Open Spectrum File** menu.

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10. Start SEC measurement

10.3 Saved Time Sequence record results

If you un-check on Time Sequence Setting: Whether **Create file at every data tracking interval**
The spectrum should be record as a video, and you can open from **File>Open Time Sequence File** menu.

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11. Summary of SEC measurement

- Spectroelectrochemistry(SEC) is a measurement which combined the spectroscopy measuring method with the electrochemical measuring method simultaneously.
- Using the trigger mode of the SEC2020 spectrometer system, makes it easy to get the synchronized SEC measurements.
- BAS Inc. offers cuvette-type and flow-type SEC cells with ITO, GC, Pt and Au electrodes to met your electrochemical purpose.



End

- Thank you for your attention.

