

**ENERGY DISPERITIVE
X-RAY FLUORESCENCE SPECTROMETER**



USER MANUAL

Rev. 5

RIGAKU CORPORATION

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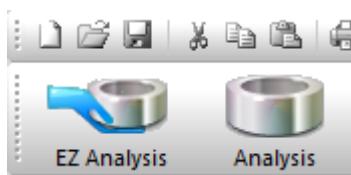
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1. ANALYSIS OPERATION

1.1 OUTLINE

An analysis can be made using [EZ Analysis] or [Analysis] on the tool bar. An FP application or an empirical application must be prepared beforehand.



The outline of each measurement is given below. Use the appropriate one according to your needs.

- Measurement Using [EZ Analysis]

By registering a prepared application, an analysis can be made with minimum operation.
(Up to five applications can be registered.)

- Measurement Using [Analysis]

Any of the prepared applications can be selected. A folder for saving can also be selected.



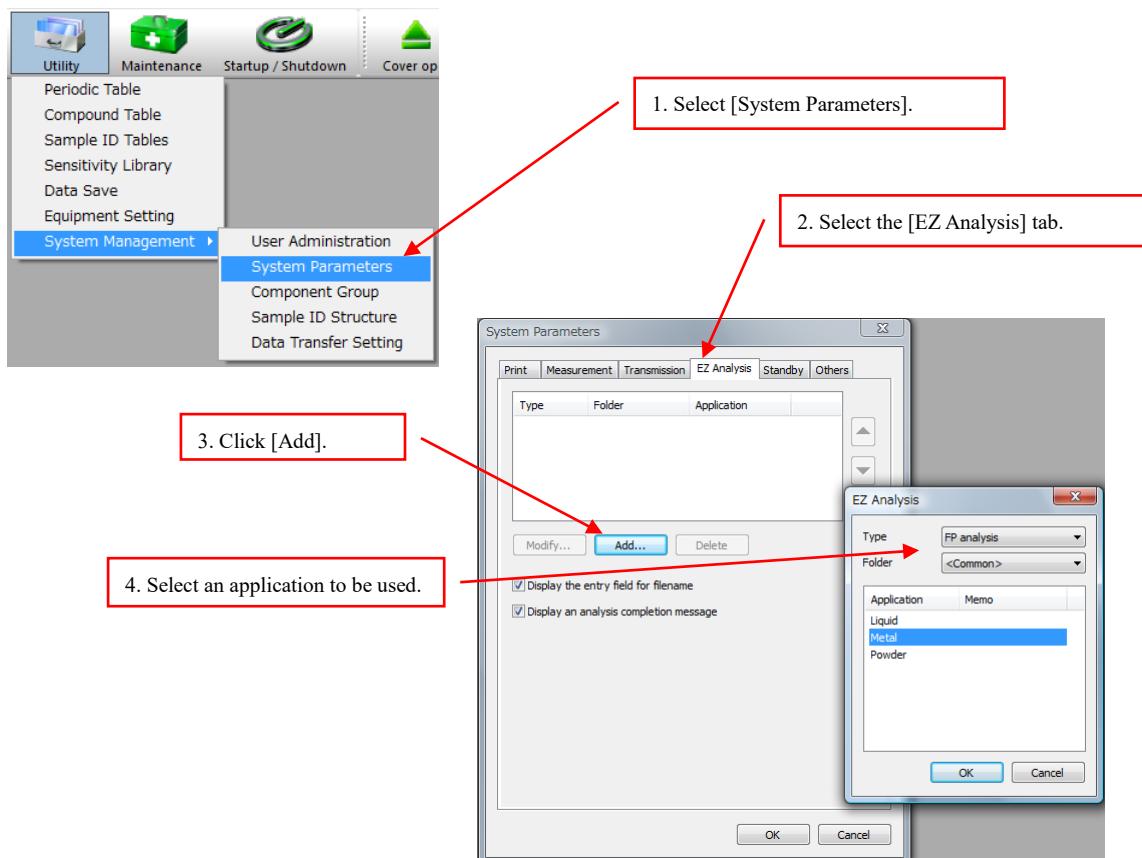
- Before setting operation for an analysis, make sure that the type of sample changer attached to the spectrometer is the same as the one that has been set using [Equipment Setting]. (See “1.4 i. Setting for Sample Changer”.)
- For safety, applications using different measurement atmospheres cannot be selected simultaneously in a series of analyses. When measurements are to be made using the 15-sample changer, only applications that have the same measurement atmosphere as that of the application that has been registered first can be registered for analyses.

1.2 OPERATIONAL PROCEDURE FOR “EZ ANALYSIS”

To make a measurement using [EZ Analysis], a prepared application must be registered beforehand. The procedure is described below in the paragraphs “Registration of Application” and “Analysis Using EZ Analysis”:

Registration of Application

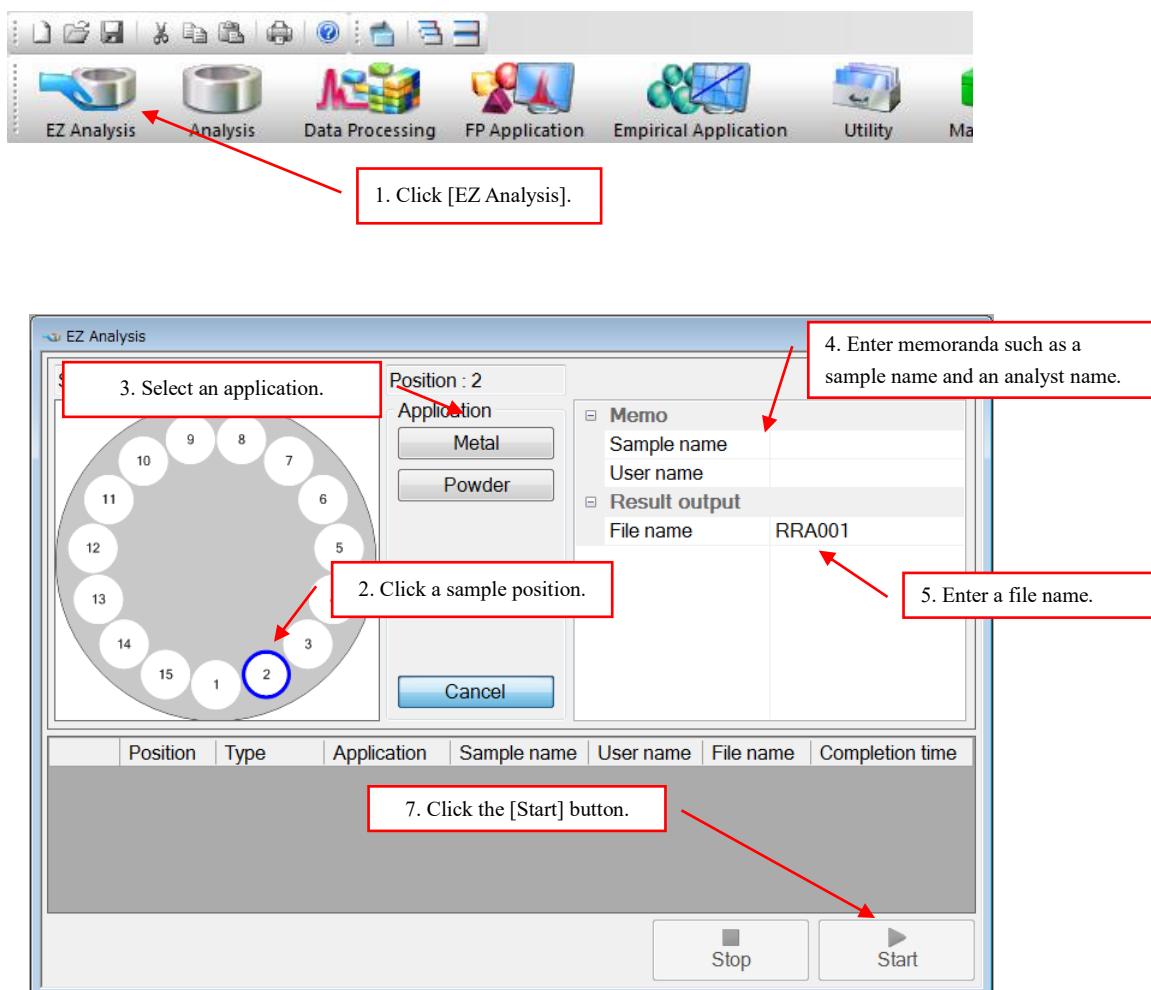
1. Click [Utility] → [System Management] → [System Parameters] on the tool bar.
2. Select the [EZ Analysis] tab on the [System Parameters] dialog.



3. Click [Add] and select an application to be registered for EZ Analysis.
4. Select an application type (FP analysis or empirical analysis) and a folder in which an application to be registered has been stored, and applications in the folder will be displayed. Select the application to be registered and click [OK], and it will be added to the list.
5. Check the added application and click [OK]. A message [When system parameters have been changed, please restart the NEX CG software] appears. Click [OK] and restart the NEX CG software.

Analysis Using EZ Analysis

1. Click [EZ Analysis] on the tool bar.
2. Click a sample position on the [EZ Analysis] dialog.
3. Select an application to be used.
4. Enter information such as a sample name and an analyst name, if necessary.
5. Enter a file name. This name is used as the data file name when saving data in the hard disk.
To cancel the analysis reservation, click the sample position and click [Cancel] in the [Application] group box.
6. Set samples using steps 2 to 5.
7. Click the [Start] button.

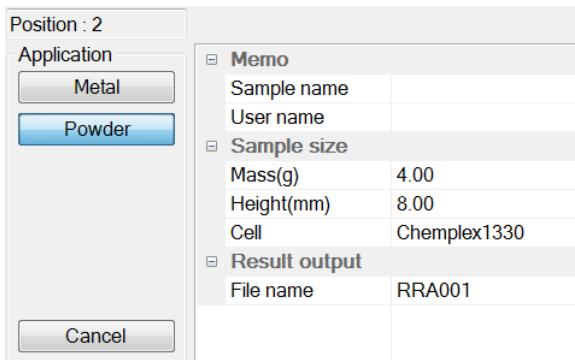


Note: The above screen appears when the 15-sample changer is used.

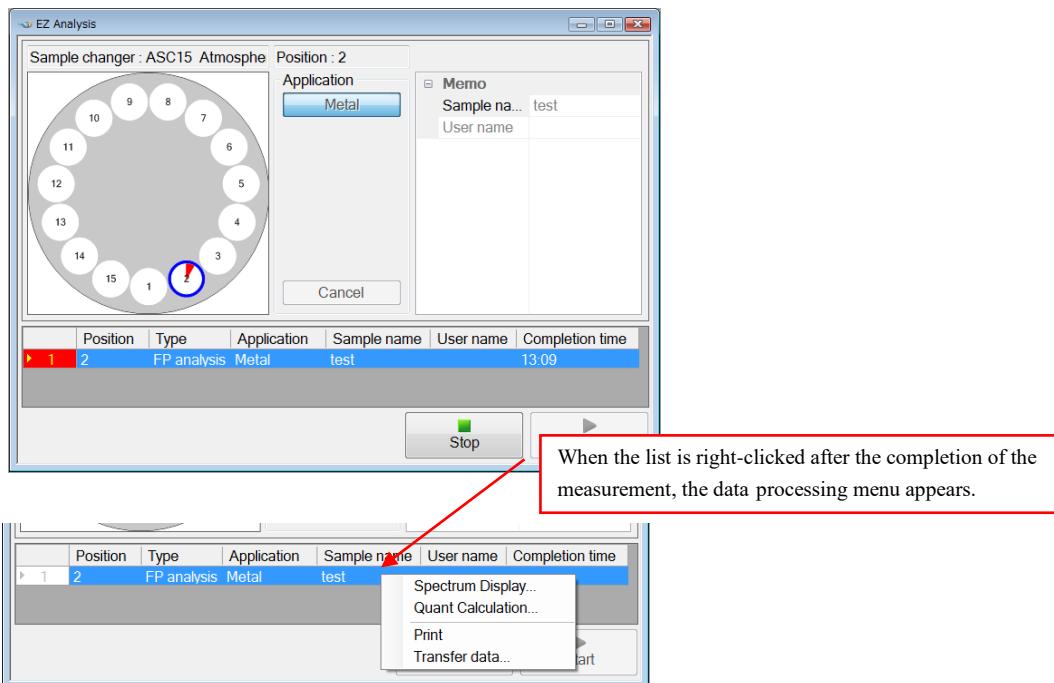
1. ANALYSIS OPERATION



- When a sample name is entered, it can be entered automatically also as the file name. See “ii. Simplification of Sample File Name Input” in “1.4 OTHER FUNCTIONS”.
- Input fields for the sample size etc. may appear depending on the application. Set data according to the sample.



- Analyses are made in order of sample position numbers. Information on the sample that is being measured is displayed in the list in the lower part. When the list is right-clicked after the completion of the measurement, the data processing menu appears.

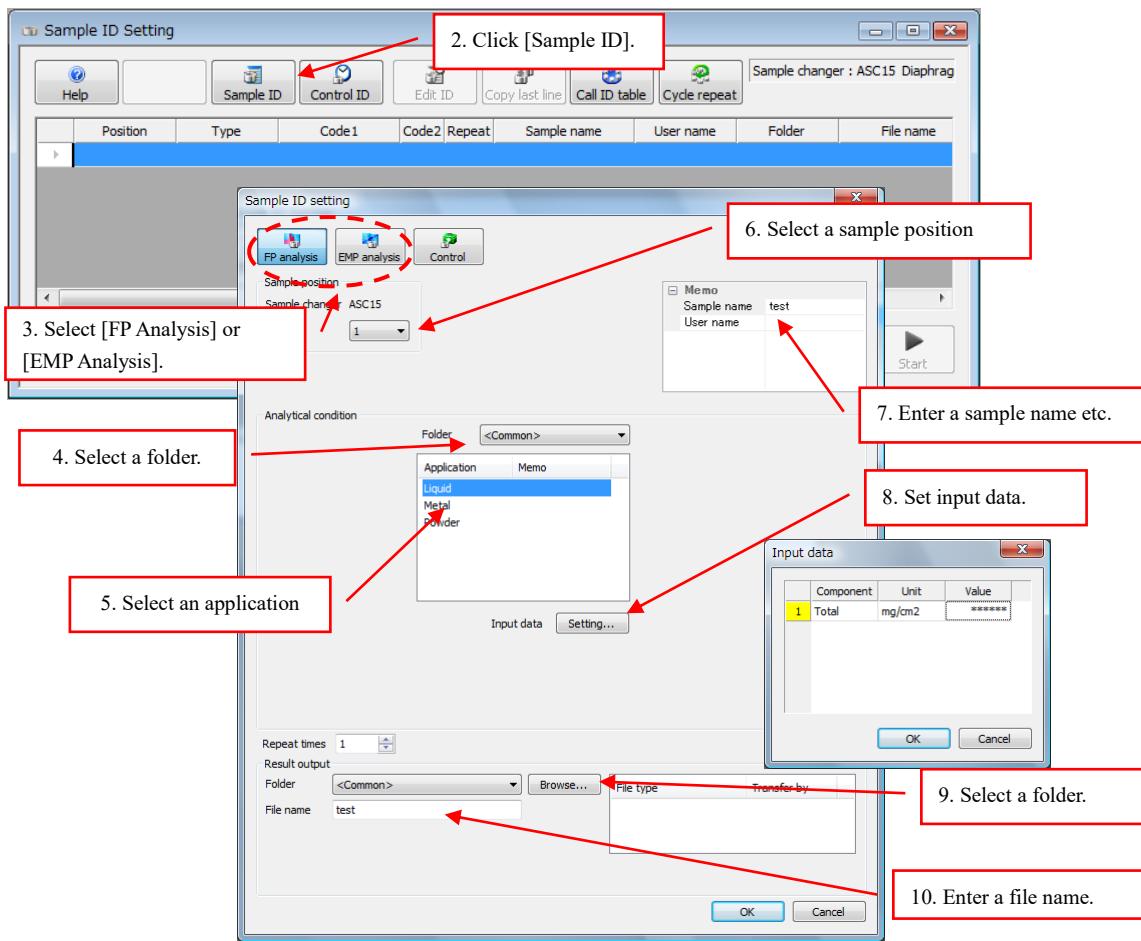


- When an analysis has been started, the addition to or the change of entered information is not possible.
- To stop an analysis, click the [Stop] button.

1.3 OPERATIONAL PROCEDURE FOR “ANALYSIS”

In the case of an analysis using [Analysis], any of the prepared applications can be selected. A folder for saving can also be selected.

1. Click [Analysis] on the tool bar.
2. Click [Sample ID] on the [Sample ID Setting] screen. Or double-click a line displayed in blue. A screen to set a sample ID appears.



Selection of Application

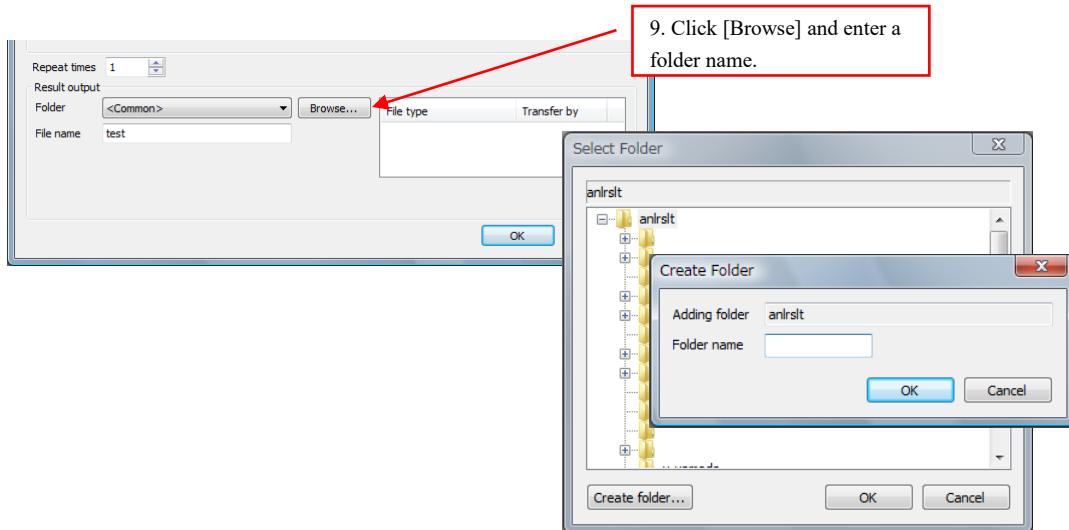
3. To use an FP application, click [FP Analysis]. To use an empirical application, click [EMP Analysis].
4. In the frame for analysis information, select a folder in which an application to be used has been stored.
5. Select an application to be used.

Setting of Samples to Be Measured

6. Select a sample position.

1. ANALYSIS OPERATION

7. Enter information such as a sample name and an analyst name, if necessary.
8. When there is an input component such as coating weight, click [Setting] and enter a value.
9. Select a folder for saving an analyzed result. To create a new folder, click [Browse], click [Create folder] on the [Select Folder] screen, and enter a name.



- A new folder is created just under the folder that has been selected on the dialog for folder selection. To create a folder just under the common folder, select [anrls1t] and then click [Create folder].
10. Enter a file name.
 11. Click [OK]. The analysis ID that has been set is reserved.
 12. When the system has a sample changer, repeat steps 2 to 11 for analysis information, if necessary.
 13. Click [Start], and a measurement will start.

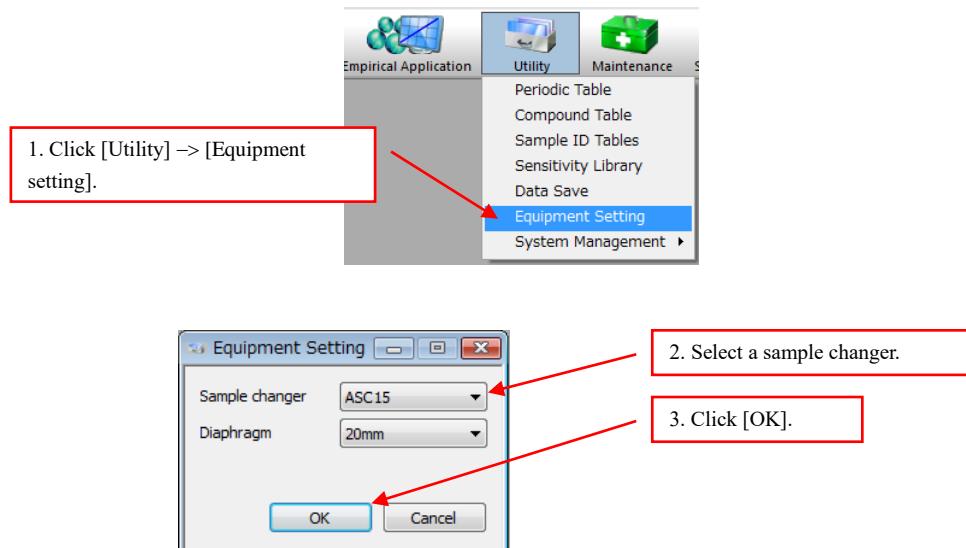
1.4 OTHER FUNCTIONS

Described below are functions used in common for [EZ Analysis] and [Analysis], namely, a setting for the sample changer and the simplification of sample file name input. The contents to be introduced are outlined below:

i. Setting for Sample Changer

Described below is a software setting for the sample changer attached to the sample chamber. The setting is reflected also on the [EZ Analysis] screen.

1. Click [Utility] → [Equipment Setting] on the tool bar.
2. Select a sample changer in the [Sample changer] combo box.
3. Click [OK].



One of the following can be selected for a sample changer. (Some of them may not be displayed depending on selection for options.)

- (1) None: For one sample
- (2) ASC15: 15-sample changer
- (3) ASC10: 10-sample changer
- (4) ASC9: 9-sample changer
- (5) ASC9S: 9-sample changer with spin function

1. ANALYSIS OPERATION

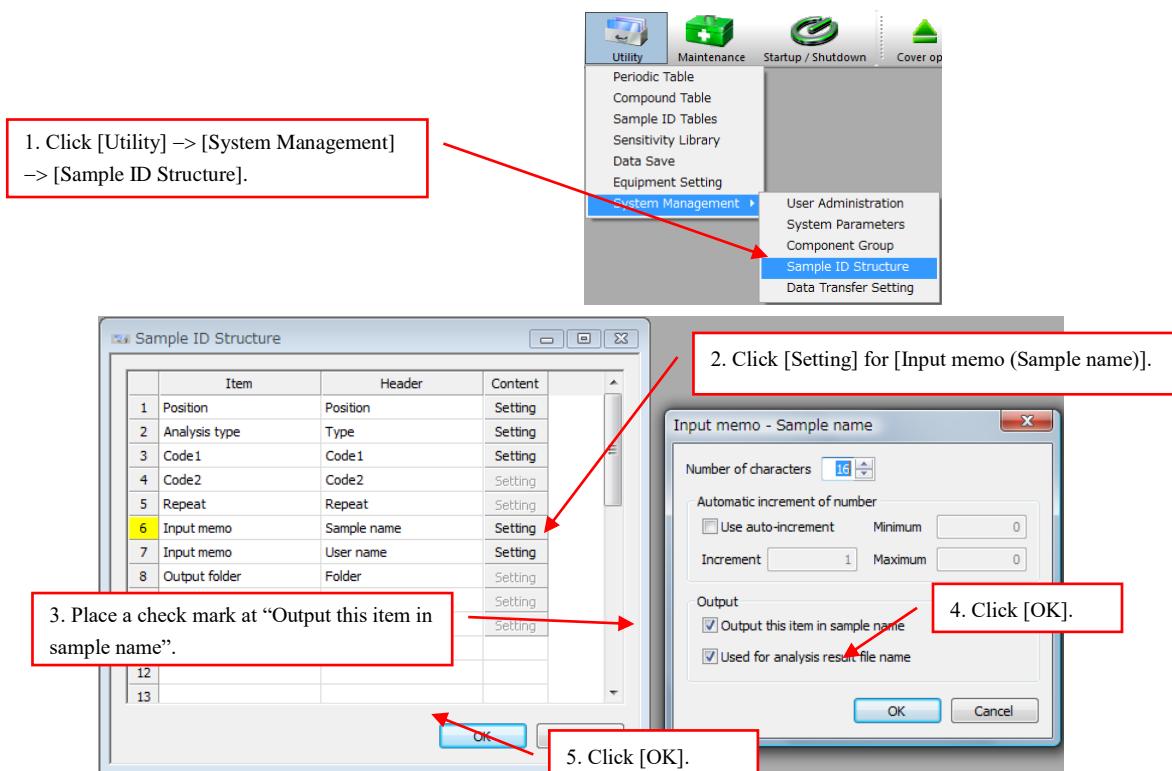
ii. Simplification of Sample File Name Input

A setting can be made such that a file name for saving in the hard disk is input automatically when a sample name is entered as a memorandum. Operation can be simplified.



Note: Although the screen in [EZ Analysis] is used, the operational procedure is the same in the case of [Analysis].

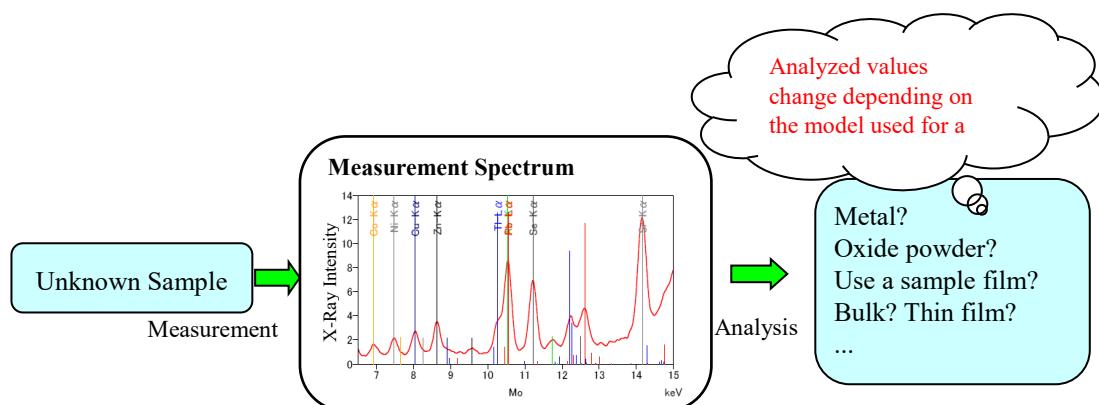
1. Click [Utility] → [System Management] → [Sample ID Structure] on the tool bar.
2. Click [Setting] for [Input memo (Sample name)].
3. Place a check mark at “Output this item in sample name”.
4. Click [OK].
5. Click [OK].
6. Restart the NEX CG software, and the setting will be reflected.



2. CREATION OF FP APPLICATION

2.1 OUTLINE

To make the FP quantitative analysis (RPF-SQX analysis) using the NEX CG, an FP application must be prepared according to the sample to be measured. Since the theoretical intensity calculation using the FP method is made, a sample model to be used for the calculation, such as the chemical combination type of sample components (metal, oxide or the like), flux, binder and presence of a sample protection film, must be set accurately. Because application templates created for various sample forms and measurement methods have been registered, prepare an FP application using or referring to those templates.



- List of FP Application Templates

Sample	Tab	Template
Metal Sample	Metal & Alloy	Metal
Powder Sample	Oxide Powder	Powder
		Pellet
		Powder_Oxide
		Pellet_Oxide
	Fusion Bead	Fusion Bead
Plastic Sample	Polymer	Powder
		Pellet
Liquid Sample	Liquid	Liquid
		Oil

2.2 INTRODUCTION OF APPLICATION TEMPLATES

The application templates shown below have been registered for various conditions after the sample processing (preparation for a measurement) of various sample forms such as the metal, powder and liquid. Select a template nearest to the condition of a sample to be measured, and change its contents if necessary.

	Example of Sample	Printout Setting	Template	Tab
Metal	Metal sample such as metal chip or screw 	Mn, Ni, Cr ...	Metal	Metal & Alloy
Powder	Sample cell and sample film 	Si, Fe ... O (balance)	Powder	Oxide Powder
		SiO ₂ , Fe ₂ O ₃ ...	Powder_Oxide	
	Pressed sample 	Si, Fe ... O (balance)	Pellet	
		SiO ₂ , Fe ₂ O ₃ ...	Pellet_Oxide	
	Glass fusion bead 	SiO ₂ , Fe ₂ O ₃ ...	Fusion Bead	Fusion Bead
Plastic	Sample cell and sample film 	Pb, Cd ... PP (balance)	Powder	Polymer
		Pb, Cd ... PP (balance)	Pellet	
Liquid	Sample cell and sample film 	Pb, Cd ... H ₂ O (balance)	Liquid	Liquid
		S,Cl ... oil (balance)	Oil	

- Analyzable Element Range

Yellow: Analyzable element

Orange: Analyzable element (oxide)

Pink: Unanalyzable element

Template: Metal

Template:Oxide powder (Powder, Pellet, Powder Oxide, Pellet Oxide)

Polymer (Powder, Pellet)

Liquid (Liquid, Oil)

* The Powder Oxide and the Pellet Oxide are set as the oxide.

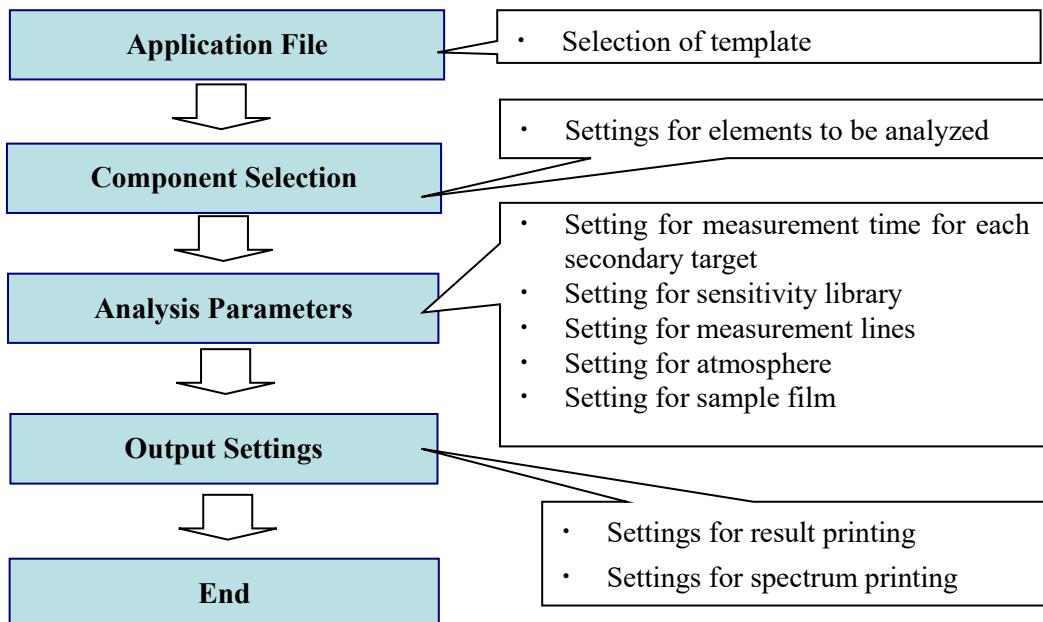
Template: Fusion Bead

2.3 CREATION OF FP APPLICATION

To create an FP application, start with [FP Application] on the tool bar.

The applications described in “2.2 INTRODUCTION OF APPLICATION TEMPLATES” have been registered beforehand. Use or refer to those templates when creating an FP application.

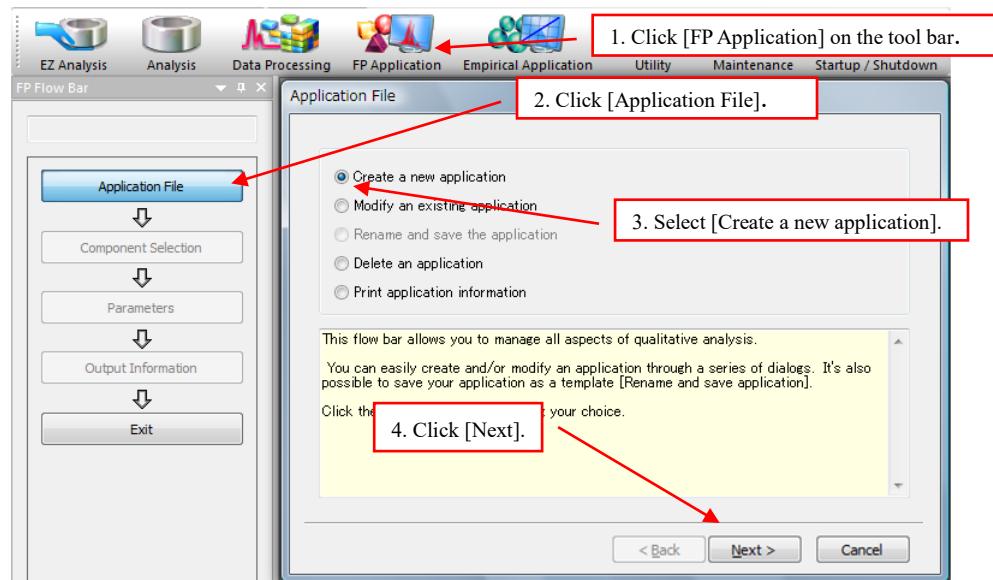
The overall flow is shown below:



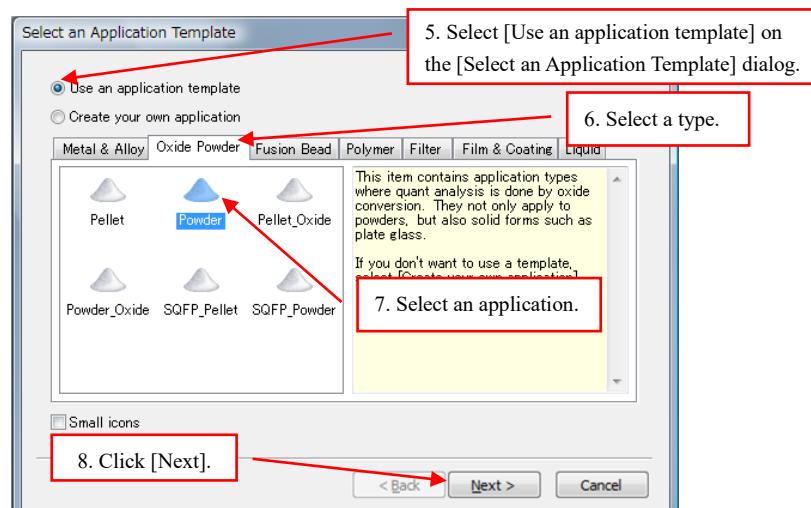
i. Creation of Application File

Set the name of an FP application to be created, the storage place for the application and template to be used.

1. Click [FP Application] on the tool bar.
2. Click [Application File].
3. Select [Create a new application] on the [Application File] dialog.
4. Click [Next].

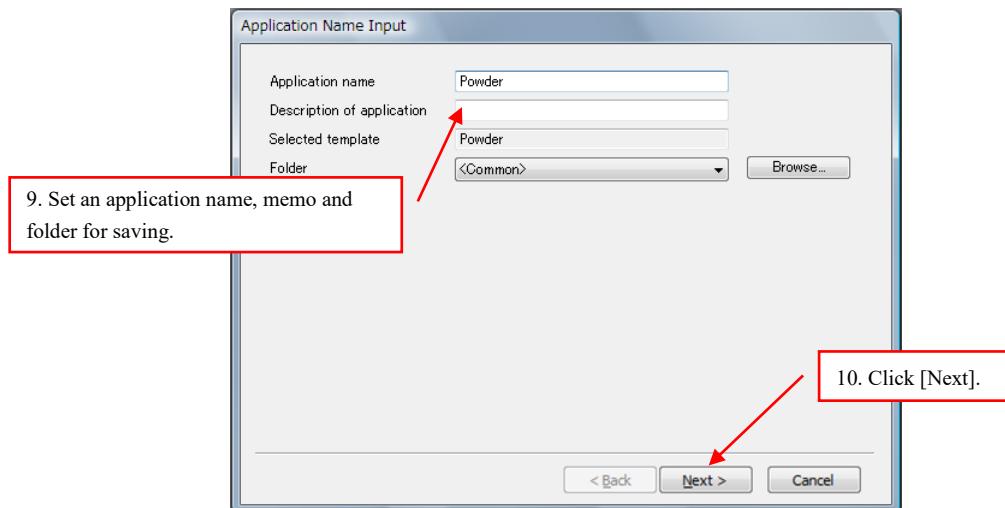


5. Select [Use an application template] on the [Select an Application Template] dialog.
6. Select a type from the tabs.
7. Select an application.
8. Click [Next].

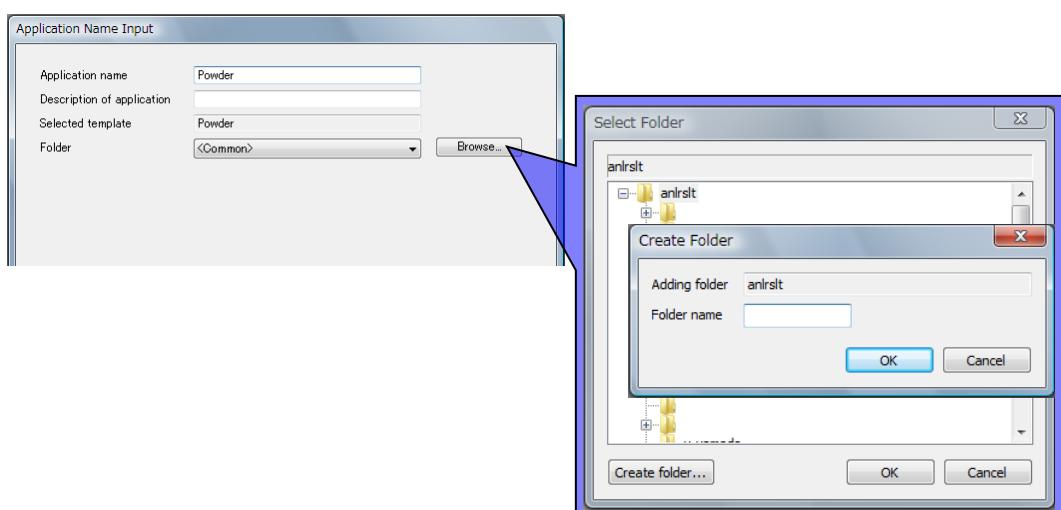


2. CREATION OF FP APPLICATION

9. Set an application name, memo and folder for saving on the [Application Name Input] dialog.
10. Click [Next].



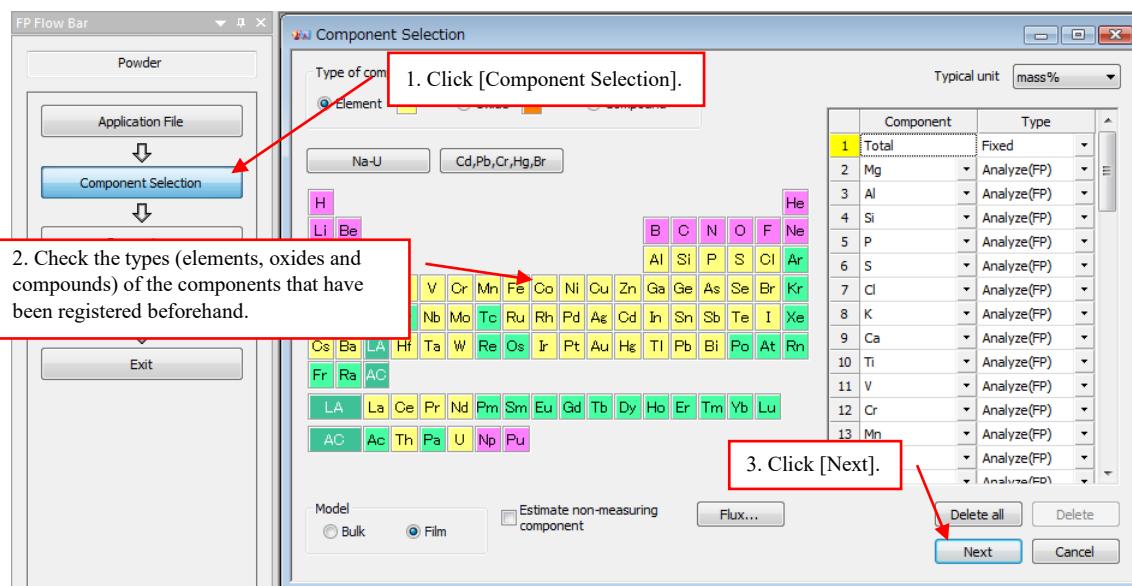
- To create a new folder in which to save an application file, click [Browse], click [Create Folder] on the [Select Folder] screen, and set a name.
- A new folder is created just under the folder that has been selected on the dialog for folder selection. To create a folder just under the common folder, select [anlcond] and then click [Create folder].



ii. Setting Procedure for Component Selection

Select the components to be used for a quantification calculation. Check the components that have been registered beforehand. Delete unnecessary elements, if any.

1. Click [Component Selection] on the flow bar.
2. Check the types (elements, oxides and compounds) of the components that have been registered beforehand in the [Type of component to add] group box, and carry out deletion or registration.
3. Click [Next].



- The constitution of the component selection varies slightly depending on an application template.
- All elements that can be measured are not included in a template as analyzable elements to be used for a quantification calculation; rare gases, elements that are not likely to exist in a sample, and similar elements are excluded. Add elements if necessary. However, since a fixed quantity of Zr is detected from the inside of the instrument, an impurity correction must be made. (For the registration of an impurity, see “7. DATA PROCESSING” in the User Manual.)
- When a sample is thin and its fluorescent X-ray intensities are affected by its thickness in an analysis using an FP application, an accurate correction becomes possible by using the “thin film” for the sample model and entering coating weight (sample weight per unit area in mg/cm²) as the thickness information. On the other hand, the “bulk” is used as the sample model for a sample when the fluorescent X-ray intensities are not affected by sample thickness. In the case of the “bulk” model, thickness information does not need to be set.

2. CREATION OF FP APPLICATION

- Set the “thin film” for the sample model of a sample that is extremely thin and consists of analysis elements with large atomic numbers.
- Although a template for bead samples has been registered beforehand, settings can be changed by clicking [Flux] on the screen when the dilution ratio must be changed or the flux used is not lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$).
- When binder is mixed with powder sample, click [Flux] on the screen and set data.
- Component Types

Comp.	Type	Description
Total	Analysis (FP method) Fixed Input	Displayed when the sample model is the “thin film”. When a polymer or oxide powder sample is analyzed using the thin film model, select “fixed” or “input”.
Element Name	Analysis (FP method)	A concentration is calculated using a measured intensity. In the template, this setting is used for all elements to be measured.
	Balance (Presettable for only 1 component)	This setting is necessary when a sample contains components that are not measured, such as C, H and O, as in the case of a polymer. <u>A quantification calculation is made such that the concentrations of detected components (and the fixed or input component) and the balance component becomes 100 mass%.</u>
	Fixed flux Input flux	Set this type when flux or binder is added to a sample.

- Default Settings (Component Selection) for Each Template

Template	Model	Comp.	Balance	Flux
Metal	Bulk	Element	—	—
Oxide Powder (Powder, Pellet)	Bulk	Element	O	—
Oxide Powder (Powder_Oxide, Pellet_Oxide)	Bulk	Oxide	—	—
Bead	Bulk	Oxide	—	$\text{Li}_2\text{B}_4\text{O}_7$
Polymer (Powder, Pellet)	Thin film	Element	PP	—
Liquid	Thin film	Element	H_2O	—
Oil	Thin film	Element	Oil	—

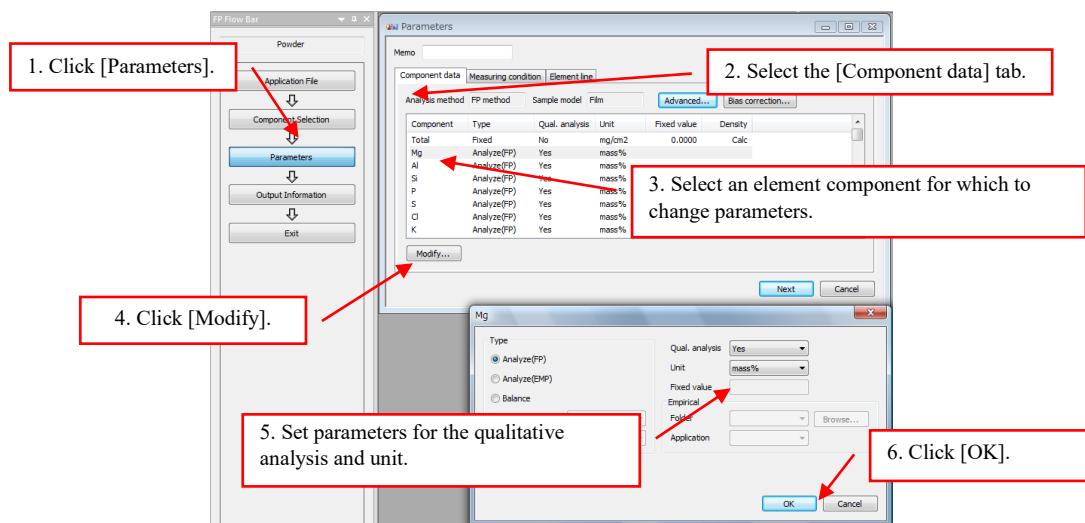
iii. Analysis Parameter Setting Procedure

Analysis parameters are set on the (A) Component data tab, (B) Measuring condition tab and (C) Analysis element tab. Change the conditions registered beforehand in a template, such as the usage of the sample film and the measurement atmosphere, according to the sample. The setting procedure on each tab is described below. Refer to necessary descriptions.

(A) Setting Procedure on Component Data Tab

The component type can be changed from “Analysis” to “Input” or “Fixed” as described in “ii. Setting Procedure for Component Selection”. When a fixed value is set, it can be entered. On [Advanced], the sample film, blank sample and impurity can be set.

1. Click [Parameters] on the flow bar.
2. Select the [Component data] tab on the [Parameters] dialog.
3. Select an element component for which to change parameters.
4. Click [Modify].
5. Set a component type (analysis using the FP method, balance component, known fixed value, known value input manually at the time of an analysis or flux), *quantification* calculation method ([Yes] or [No] for the qualitative analysis), unit and fixed value (when the type is the known value).
6. Click [OK].
7. Repeat steps 3 to 6 for relevant elements.



- [Yes] or [No] can be selected for the qualitative analysis. When [Yes] is selected, a peak is retrieved from a measured spectrum, and a calculation is made for an identified element. When [No] is selected, a calculation is made forcibly regardless of the detection of a peak.

2. CREATION OF FP APPLICATION

- Calculation Method for Coating Weight (Sample Weight (g) per Unit Area (1 cm²))

Example: Measurement of polymer sample (2.0g) in polyethylene sample cell (Chempex 1330 with ID of approximately 24mm)

$$\text{Sample Weight} / \text{Sample Area} = \text{Coating Weight}$$

$$(2000\text{mg}) / (1.2 \times 1.2 \times 3.14 \text{ cm}^2) = \underline{\underline{442 \text{ mg/cm}^2}}$$



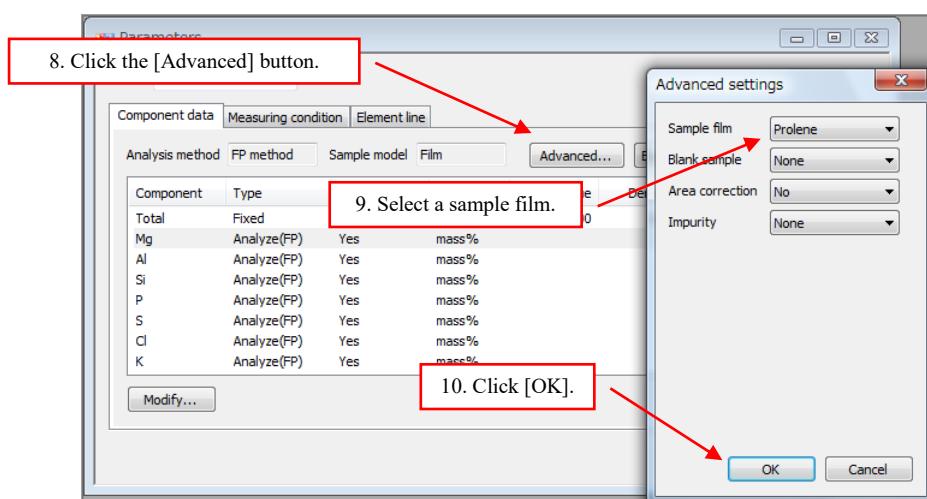
- Calculation Method for Dilution Ratio

Example: Measurement of powder sample (4.0g) mixed with boric acid (0.4g) after pressing

$$\text{Binder (Flux) Weight} / \text{Sample Weight} = \text{Dilution Ratio}$$

$$(0.4 \text{ g}) / (4.0 \text{ g}) = \underline{\underline{0.1}}$$

8. Click the [Advanced] button on the [Component data] tab.
9. Select a sample film used for a sample cup on the [Advanced settings] dialog. Set the blank sample correction and impurity correction, which must be registered beforehand, if necessary.
10. Click [OK].



- When a sample film is used for a measurement, be sure to set a film type. A correction is made for absorption by the film. A film can be selected from (1) Prolene: 4μm Prolene film (Cat. No.CH416), (2) Prolene 2: double 4μm (Cat. No.CH416) Prolene film, (3) Mylar: 3.6μm Mylar (Cat. No.CH150) and (4) Ultra-Polyester: 1.5μm ultra-polyester (Cat. No.CH090).
- [Blank sample] and [Impurity] are used for similar correction functions for impurities and impurity lines. **[Impurity]** is ordinarily used. For the procedure to register impurities, see "7. DATA PROCESSING" in the User Manual.

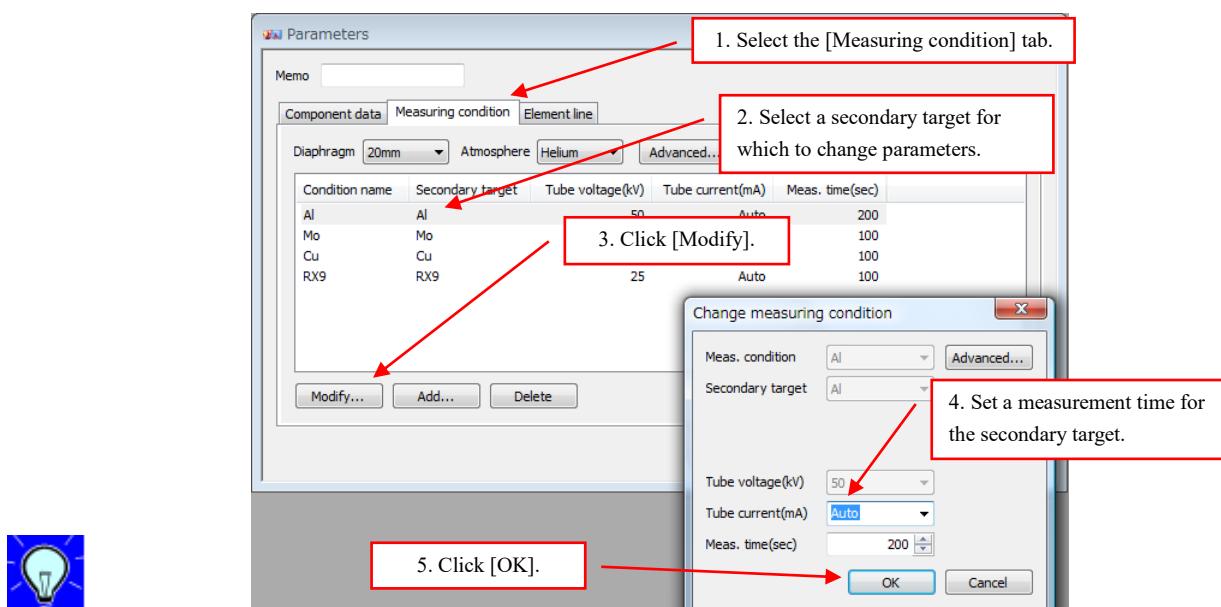
(B) Setting Procedure on Measuring Condition Tab

An excitation condition, measurement time and measurement atmosphere for each secondary target can be changed. The sample spin and measurement order optimization can be set using [Advanced]. (The sample spin function is an option.)

1. Select the [Measuring condition] tab.

Change of Excitation Condition and Measurement Time

2. Select a secondary target for which to change parameters. Set an optimum time for each of the secondary targets Al, Mo, Cu and RX9. (An optional Si target may also be installed, in addition to the four standard targets.)
3. Click [Modify].
4. Set a measurement time for the secondary target.
5. Click [OK].
6. Set a measurement time for each secondary target with steps 2 to 5.

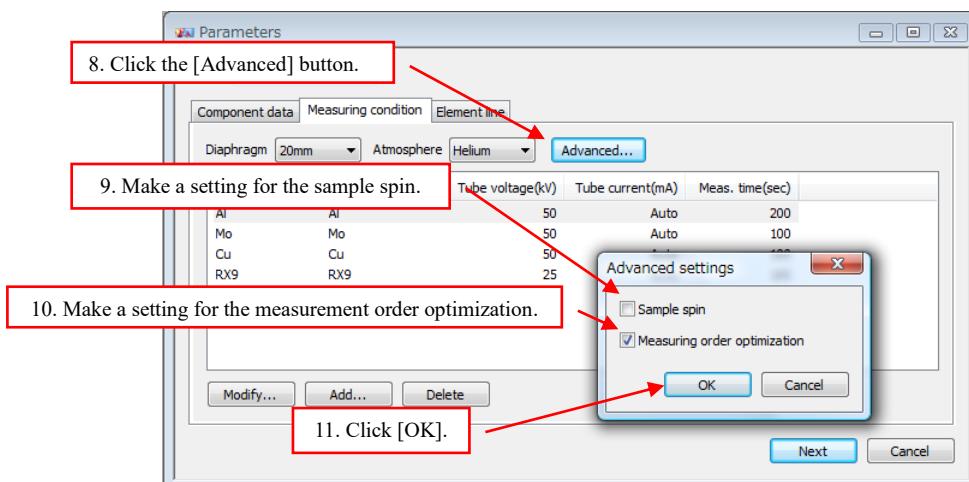
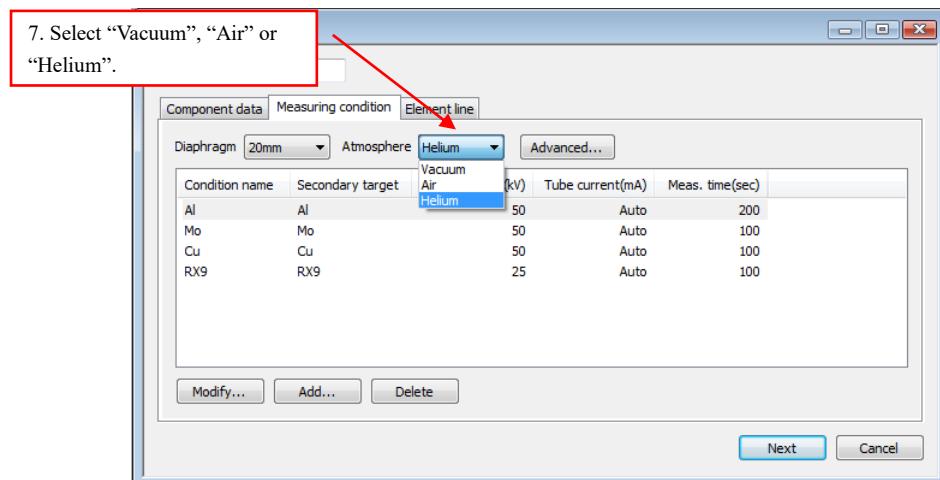


- Although the tube current can be changed, we generally recommend that it be set automatically.
- The smoothing function, threshold values for the peak search, etc. can be set using [Advanced] on the [Measuring condition] dialog. They need not be changed when default settings can be used.
- Template Standard Conditions (Measuring Conditions)

Secondary Target	RX9	Cu	Mo	Al
Tube Voltage (kV)	25	50	50	50
Tube Current (mA)	Automatic	Automatic	Automatic	Automatic
Measurement Time (sec.)	100	100	100	200

Change of Measurement Atmosphere

7. Select “Vacuum”, “Air” or “Helium” for the atmosphere on the [Measuring condition] tab.
8. Click the [Advanced] button.
9. Make a setting for the sample spin on the [Advanced setting] dialog. This item is valid when the sample spin mechanism has been attached.
10. Make a setting for the measurement order optimization on the [Advanced setting] dialog. Ordinarily, select the measurement order optimization.
11. Click [OK].



- The setting for the sample spin is valid only in the case where the sample chamber has the sample spin mechanism and the sample changer that can be used with the sample spin. In other cases, the spin is not carried out regardless of the setting.
- When the measurement order optimization is selected, measurements are made from the secondary target with the highest energy in order of energy (Al → Mo → Cu → RX9).

(C) Setting Procedure on Element Line Tab

A library can be changed (for example, from the standard library to the matching library), and an analysis line can be changed (for example, from automatic selection to K α).

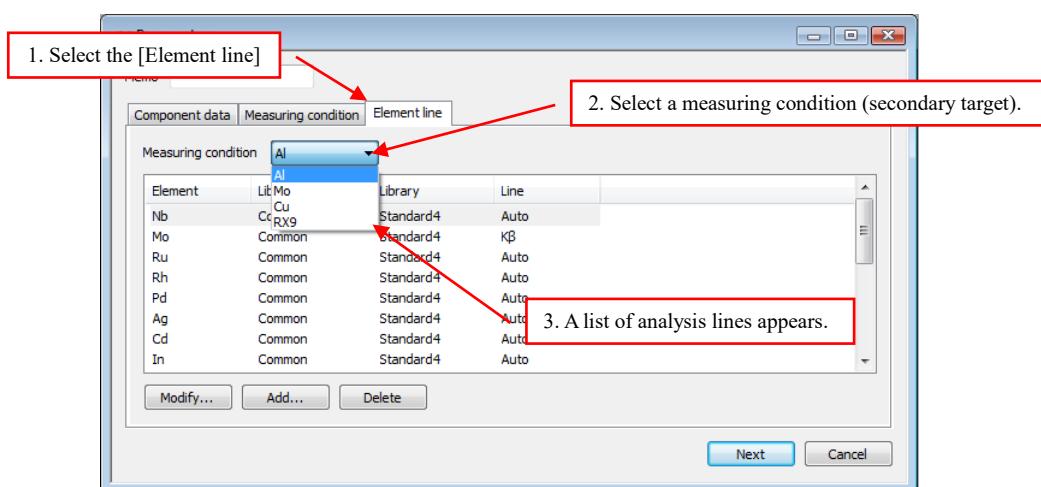
1. Select the [Element line] tab.
2. Select a measuring condition (secondary target) from the [Measuring condition] combo box.
3. A list appears of analysis lines to be analyzed using the secondary target selected above. (An analysis line is set automatically.)
4. Select an analyte for which to change parameters.
5. Click [Modify].

Change of Library

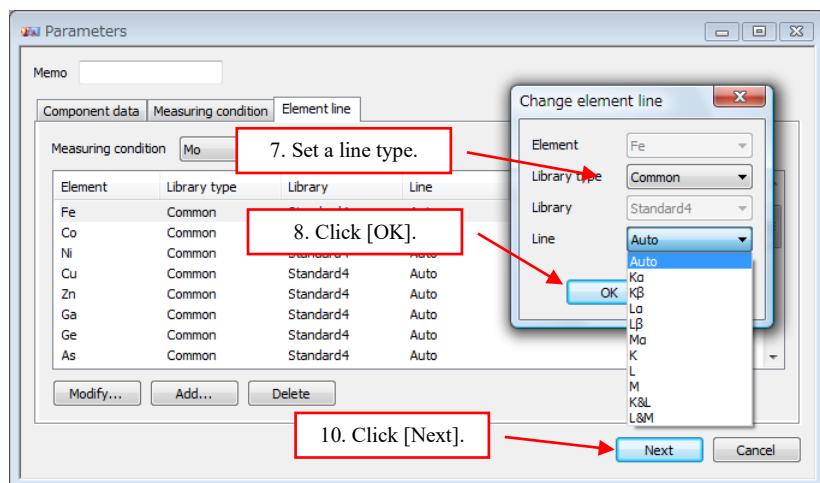
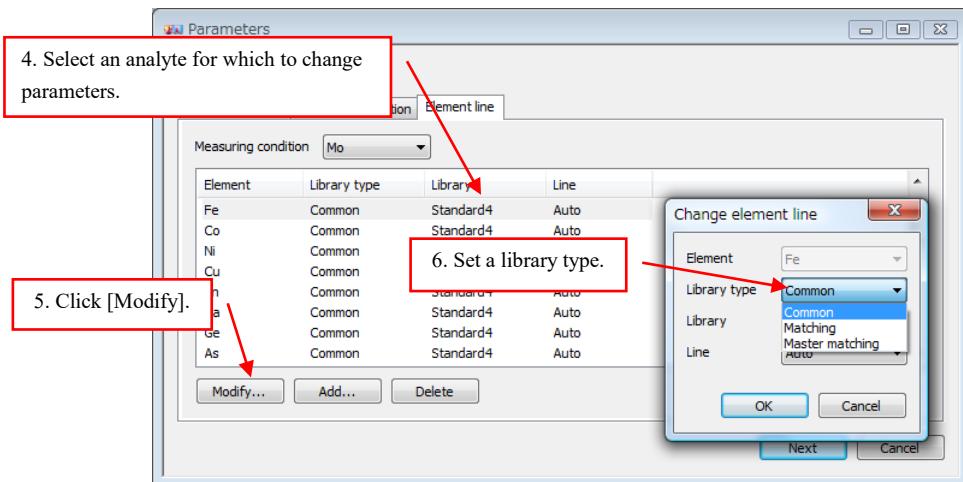
6. Set a library type on the [Change element line] dialog. The “common library” has sensitivity values registered in the instrument as default values, and those values are ordinarily used. A “matching library” can be used by registering beforehand standard samples that have sensitivity values close to those of unknown samples. Accuracy can be improved. For the procedure to create a matching library, see “3. SETTING PROCEDURE FOR MATCHING LIBRARY”.

Change of Analysis Line

7. Set a line type on the [Change element line] dialog. Ordinarily, select the automatic setting.
8. Click [OK].
9. Set parameters for each element with steps 2 to 9.
10. When all settings have been completed, click [Next].



2. CREATION OF FP APPLICATION



- When the matching library type is selected, a list of matching libraries registered for the selected element appears at [Library type], and a library can be selected from them.

- Template Standard Conditions (Manually Input Components and Other Parameters)

Template	Manually Input Component	Other Parameters
Metal	None	<ul style="list-style-type: none"> Sample film: None Measurement atmosphere: Vacuum
Oxide Powder (Powder, Powder_Oxide)	None	<ul style="list-style-type: none"> Sample film: Prolene Measurement atmosphere: Helium
Oxide powder (Pellet, Pellet_Oxide)	None	<ul style="list-style-type: none"> Sample film: None Measurement atmosphere: Vacuum
Fusion Bead	Dilution ratio ^{*1} (Fixed value: 10)	<ul style="list-style-type: none"> Flux: Li₂B₄O₇ Sample film: None Measurement atmosphere: Vacuum
Polymer (Powder)	Dilution ratio ^{*2} (Fixed value: 442 mg/cm ²)	<ul style="list-style-type: none"> Sample film: Prolene Measurement atmosphere: Helium
Polymer (Pellet)	Dilution ratio ^{*2} (Fixed value: 159 mg/cm ²)	<ul style="list-style-type: none"> Sample film: None Measurement atmosphere: Vacuum
Liquid	Dilution ratio ^{*2} (Fixed value: 885 mg/cm ²)	<ul style="list-style-type: none"> Sample film: Prolene Measurement atmosphere: Helium Measurement in vacuum impossible
Oil	Dilution ratio ^{*2} (Fixed value: 885 mg/cm ²)	<ul style="list-style-type: none"> Sample film: Prolene Measurement atmosphere: Helium Measurement in vacuum impossible

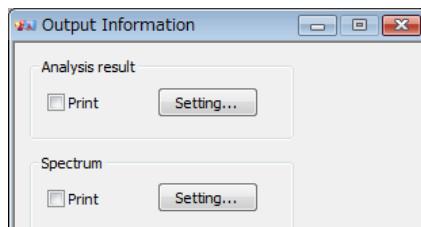
*1: Dilution Ratio = Flux Weight / Sample Weight

*2: Coating Weight = Sample Weight / Sample Area

- The coating weight fixed value (442 mg/cm²) of the polymer (loose) is calculated on the assumption that 2g of sample is put in the sample cell CH1530 with an inside diameter of approximately 24mm or CH1330 with an inside diameter of approximately 24.4mm (the inside diameter is assumed to be 24mm for the calculation). The coating weight fixed value (159 mg/cm²) of the polymer is calculated on the assumption that a sample is a plastic pellet with a thickness of approximately 2mm, a diameter of 40mm and weight of 2g.
- The coating weight fixed value (159 mg/cm²) of the polymer is calculated on the assumption that a sample is a plastic pellet with a thickness of approximately 2mm, a diameter of 40mm and weight of 2g.
- The coating weight fixed value (885 mg/cm²) of the liquid (aqueous solution or oil) is calculated on the assumption that 4g of sample is put in the liquid sample cell CH1530 with an inside diameter of approximately 24mm or CH1330 with an inside diameter of approximately 24.4mm (the inside diameter is assumed to be 24mm for the calculation).

iv. Output Setting Procedure

This item consists of [Analysis result] and [Spectrum]. Using [Analysis result], the calculated parameters can be set as well as the printing order and output the number of digits. Using [Spectrum], units and element markers to be used on a qualitative analysis chart can be set.

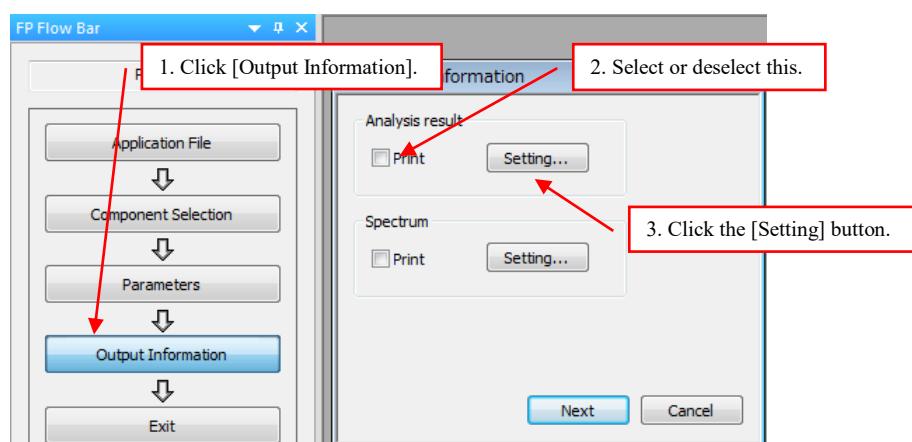


When a check mark is placed at each item, an analyzed result or a spectrum is printed according to the setting content after the completion of an analysis. The output setting items have been deselected in the templates.

1. Click [Output Information] on the application flow bar.

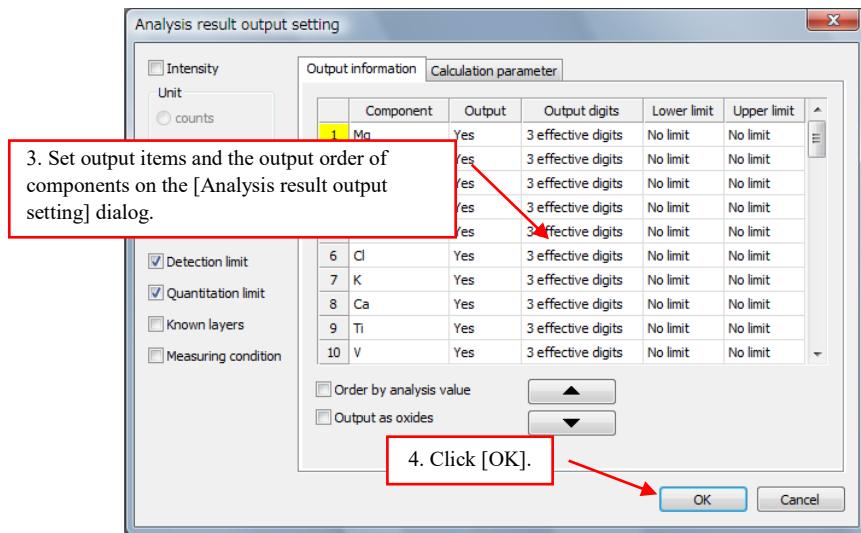
Output Setting Procedure for Analyzed Result

2. Select or deselect the [Print] check box in the [Analysis result] group box. When it is selected, an analyzed result is printed after the completion of an analysis.



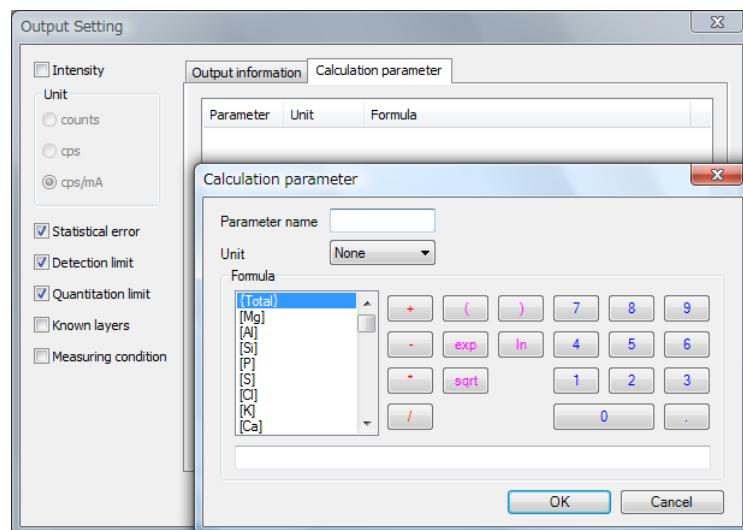
3. Click the [Setting] button in the [Analysis result] group box. Printing conditions can be set.
 - Select the check boxes of items to be printed.
 - For each component, settings can be made for whether it is output or not, the number of output digits, lower limit and upper limit.
 - By selecting a component and clicking [▲] or [▼], the printing order can be changed.
 - When the [Order by analysis value] check box is selected, components are output in order of analyzed value from the highest to the lowest.
 - When the [Output as oxides] check box is selected, the concentrations of all components are printed after converting them from those of elements (Si, Fe ...) into those of oxides (SiO₂, Fe₂O₃ ...).

4. Click [OK], and the [Output Information] screen will reappear.



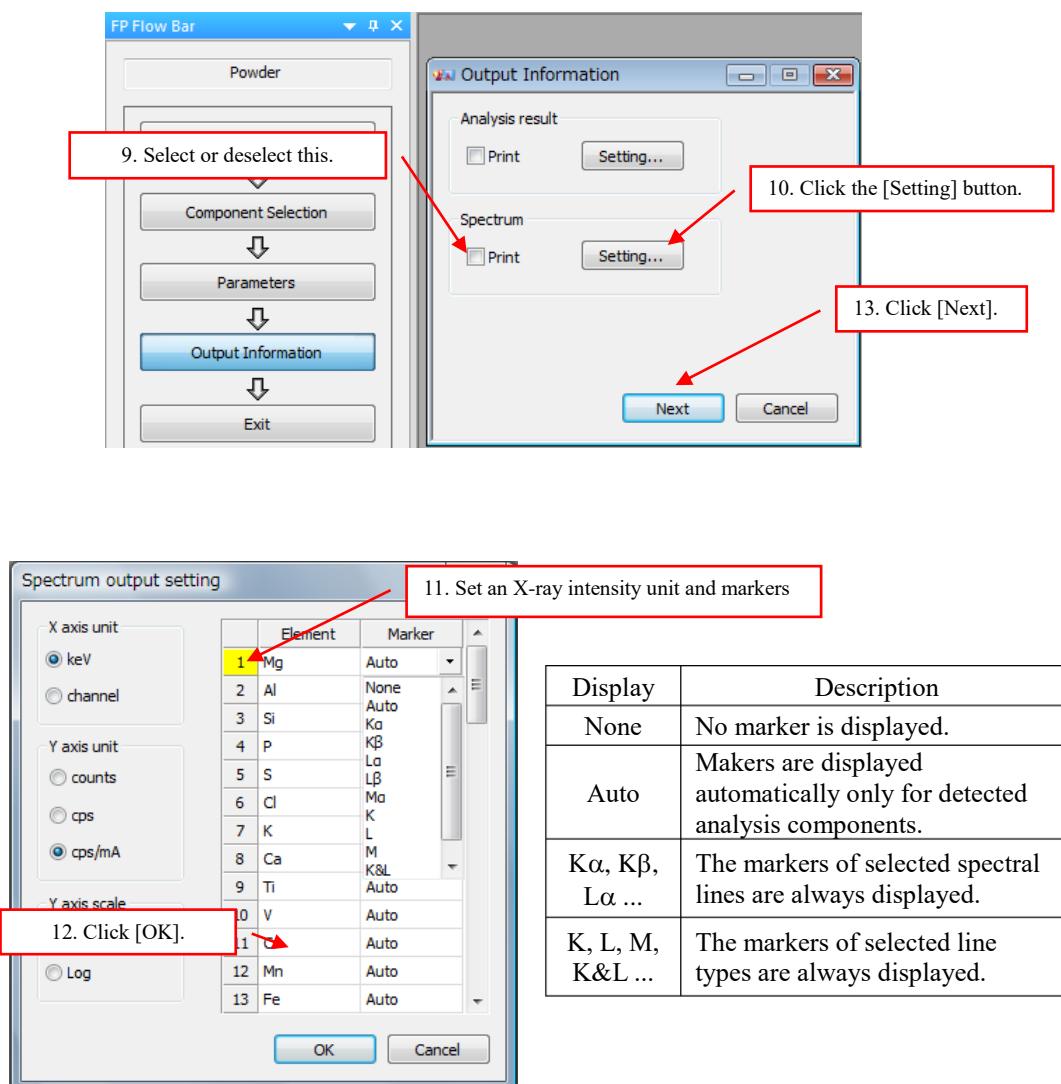
Setting Procedure for Calculated Parameter

5. Click the [Calculation parameter] tab.
6. Click [Add], and a dialog to input a formula will appear.
7. Set a parameter name and a unit.
8. Create a formula by double-clicking components and single-clicking calculation symbols and numerals.
9. After the settings are completed, click [OK].



Output Setting Procedure for Spectrum

10. Select or deselect the [Print] check box in the [Spectrum] group box. When it is selected, a spectrum is printed after the completion of a measurement.
11. Click the [Setting] button in the [Spectrum] group box.
12. Set an X-ray intensity unit and markers on the [Spectrum output setting] dialog. Select the automatic setting for markers.
13. Click [OK].
14. Click [Next], and the setting screen will close.

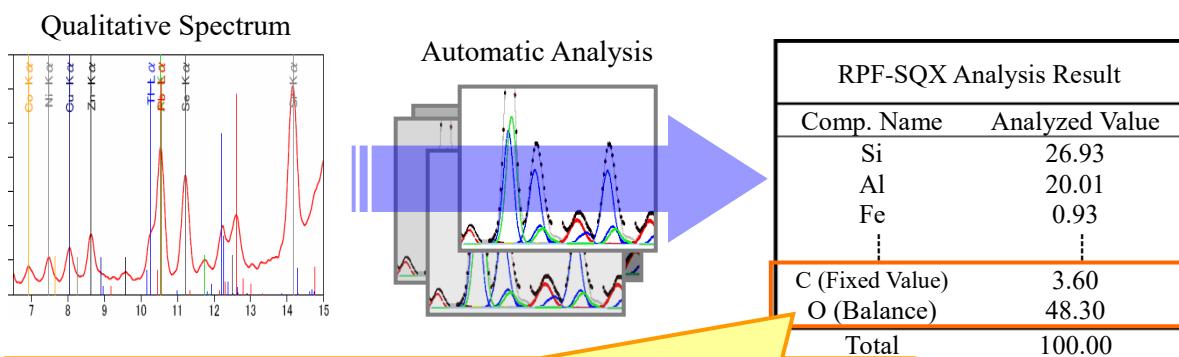


2.4 RPF-SQX SCATTERED RAY FP METHOD – SCATTERING FP

Scattered Ray FP, also called scattering FP, uses Compton and Thomson (Rayleigh) scatter peaks to estimate the average atomic number and percentage of the non-measuring components (variations in combinations of the elements H-F) in a sample.

The RPF-SQX (Rigaku Profiling Fitting-Spectra Quant X) analysis program analyzes a qualitative chart using the fundamental parameter (FP) method and waveform processing and calculates semi-quantitative analysis values. Analyzed values are calculated such that the total of the concentrations of detected components (and fixed-value and manually input components) becomes 100 mass%. Therefore, when a sample contains non-measurement components that have atomic numbers equal to or smaller than that of F, such as organic matter, O and C, those non-measurement components must be set as manually input or balance components.

Concept of RPF-SQX Analysis



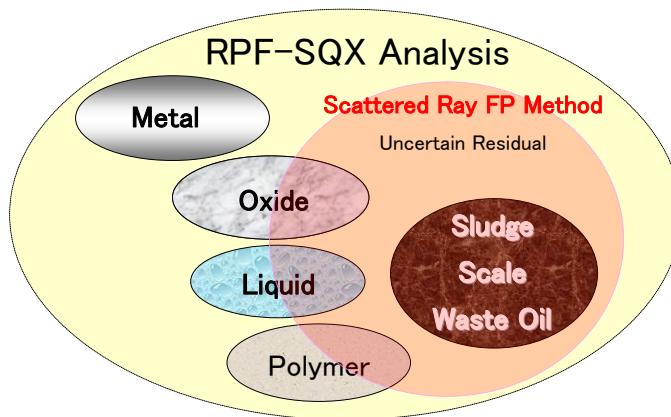
This setting is unnecessary when using the scattered ray FP method.

Non-measurement component values are estimated using rays scattered in a sample (Mo-K α Thomson scattering rays and Mo-K α Compton scattering rays).

In the scattered ray FP method (option), non-measurement components that have atomic numbers equal to or smaller than that of F are treated as one component that has an average atomic number, using scattered rays (Mo-K α Thomson scattering rays and Mo-K α Compton scattering rays) generated when characteristic X-rays (Mo-K α) generated from the Mo target are scattered in a sample. This component is added as a balance component to make quantitative calculations for detected elements that have atomic numbers equal to or greater than that of Na.

i. Samples to be Analyzed

The RPF-SQX scattered ray FP method is suitable for the analyses of samples that have the unknown concentrations of non-measurement components such as O and C. Samples to be analyzed are powder such as soil and cement, plastic and liquid containing non-measurement components.

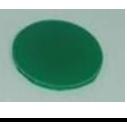


- The scattered ray FP method should be used for samples containing non-measurement components, such as soil, plastic and liquid. Do not use it for the analysis of samples that do not contain non-measurement components, such as metal chips. If it is used, a quantitative calculation result may not converge.

ii. Sample Processing

A template has been registered for each sample processing method. By carrying out sample processing according to a sample type, a template can be utilized effectively when creating an FP application.

- List (1) of Templates to Be Used for Scattered Ray FP Method

	Example of Sample	Template
Powder	Sample cell and sample film 	SQFP_Powder
	Pressed sample 	SQFP_Pellet
Plastic	Sample cell and sample film 	SQFP_Powder
	Hot press 	SQFP_Pellet
Liquid	Sample cell and sample film 	SQFP_Liquid

Recommended Sample Cells: Chempex 1330 and Chempex 1530



- A sample size must be the measurement diameter (20mm) or more. If it is less than the measurement diameter, carry out crushing, dilution or similar processing to fill the inside of the measurement diameter with the sample before a measurement.
- Although a slightly rough surface is allowable, a considerably rough one causes variations in scattered ray intensities and large analysis errors.
- If necessary, carry out processing such as drying and crushing.

iii. Measurement of Sample Size

In the RPF-SQX scattered ray FP method, it is necessary to measure the following items of a sample. These items are used to calculate the coating weight and density of a sample for an analysis using the scattered ray FP method.

Item	Unit	Remark
Sample Mass (Weight)	g	Measure the weight of the whole sample. In the case of a sample pressed using the ring method, measure the sample weight excluding the pressing ring.
Sample Height	mm	Measure the height (thickness) of a sample. The error of a sample height does not affect analysis values so much as those of a sample mass and base size. Make a measurement with precision of 1mm or so.
Sample Diameter	mm	In the case of a round sample, enter its diameter.
Sample Area	mm	Use this item in the case of a sample that is not round.
Sample Cell	–	Select this item when the Chemplex 1330 is used. (The Chemplex 1530 can also be used in common.)

- Procedure for Measurement of Sample Height When Using Sample Cell Chemplex 1330/1530

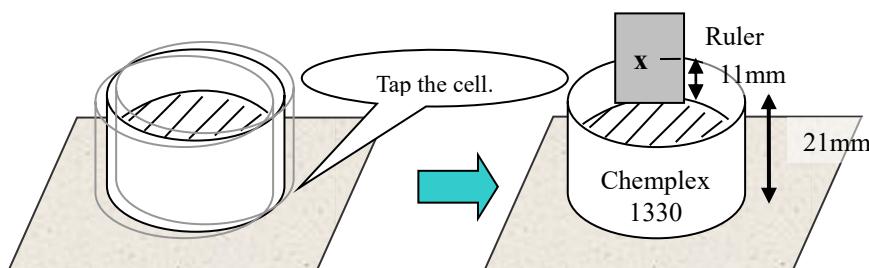
A sample height can be obtained by subtracting the distance between the top of the sample cell and that of the sample from the height of the sample cell. (The height of the Chemplex 1330 is 21mm, and that of the Chemplex 1530 is 23mm.)

1. Put weighed sample in a sample cell, and then tap it on clean paper several times to level the sample surface in it.
2. Insert a ruler into the sample cell, put its end on the sample surface, and read the scale of the ruler at the top of the sample cell in millimeters.
3. Calculate the sample height.

$$\text{Sample Height (mm)} = \text{Height of Sample Cell (mm)} - \text{Reading on Ruler (mm)}$$

- For example, when the Chemplex 1330 is used and the reading on the ruler is 11mm, the sample height is calculated as follows:

$$\text{Sample Height} = 21\text{mm} - 11\text{mm} = \underline{\underline{10\text{mm}}}$$



An FP application must be created before making an analysis. For the detailed procedure for creating an application, see “2. CREATION OF FP APPLICATION” in NEX CG User’s Manual. Select a template according to sample processing, and make necessary changes.

iv. List of Templates to be Used for Scattered Ray FP Method

The templates shown below have already been registered. Click [FP Application] on the tool bar, select [Create a new application] and click [Next], and a screen to select a template will appear. Select [Use an application template] and select an appropriate tab, and templates will be displayed.

- List (2) of Templates to Be Used for Scattered Ray FP Method

	Example of Sample	Printout Setting	Template	Tab
Powder	Sample cell and sample film 	Si, Fe ...	SQFP_Powder	Oxide Powder
	Pressed sample 	Si, Fe ...	SQFP_Pellet	
Plastic	Sample cell and sample film 	Pb, Cd ...	SQFP_Powder	Polymer
	Hot press 	Pb, Cd ...	SQFP_Pellet	
Liquid	Sample cell and sample film 	Pb, Cd ...	SQFP_Liquid	Liquid

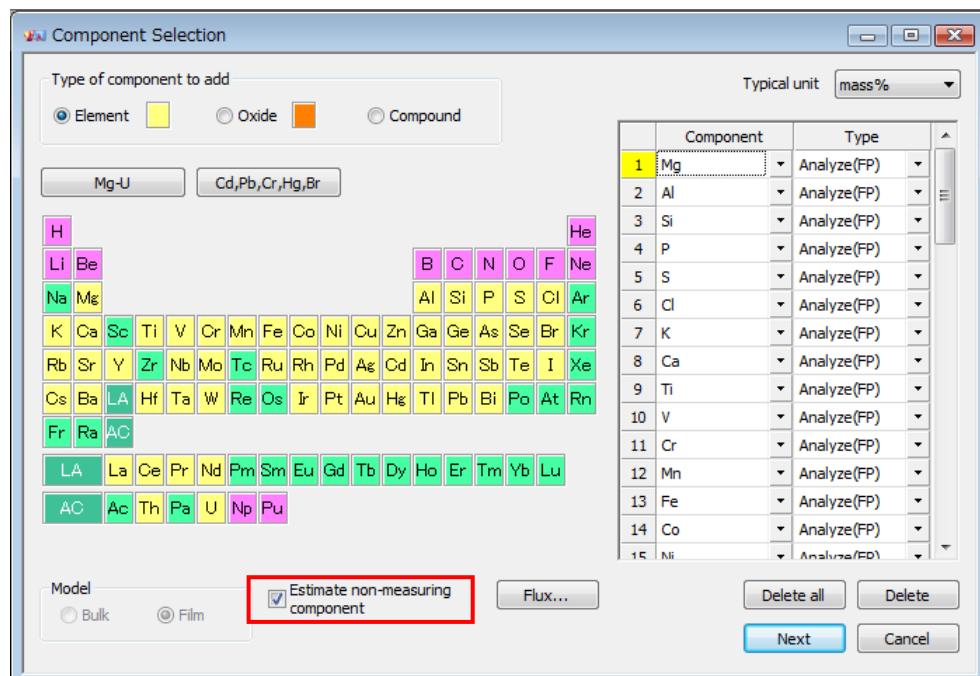
Recommended Sample Cells: Chemplex 1330 and Chemplex 1530

v. Setting for Component Selection

Analysis components can be added, deleted and changed. Because analysis components have already been selected in a template, use them intact when they need not be changed.



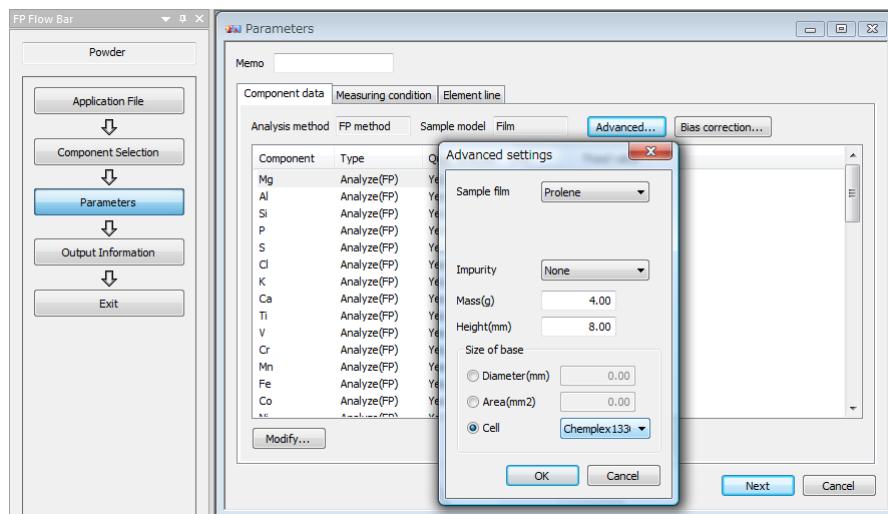
- The [Estimate non-measuring component] check box has been selected in all templates to be used for the scattered ray FP method. Do not change the setting unless the change is absolutely necessary because analyses using the scattered ray FP method will be impossible if the check box is deselected.
- If the [Estimate non-measuring component] check box is deselected and the setting is saved (the control proceeds to the next step), the relevant setting must be made again for [Element line] in [Parameters]. Take care because the setting must also be made again when the check box is selected immediately after it is deselected.



vi. Analysis Parameters

A. Setting Procedure on Component Information Tab

Enter the sample size measured in “2.2 MEASUREMENT OF SAMPLE SIZE”. It can also be changed when registering a sample ID (making an analysis reservation).



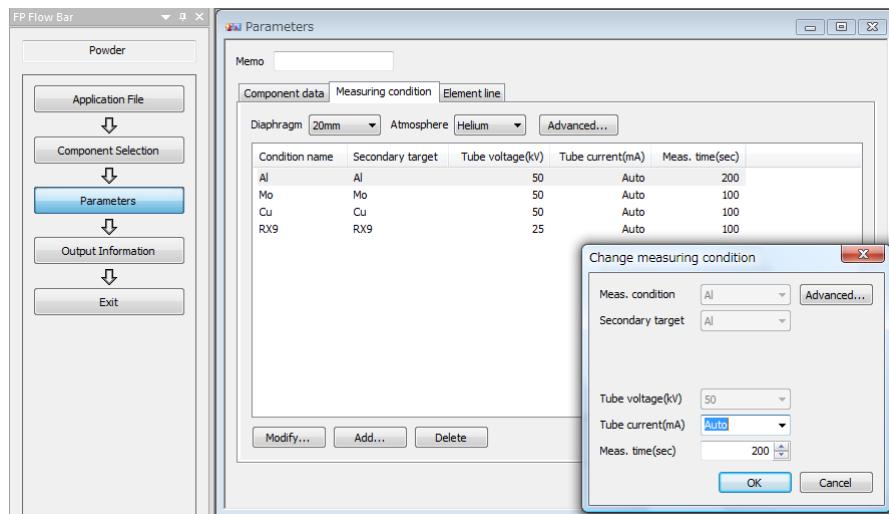
When samples of the same kind are to be analyzed using the same sample processing, entering values suitable to the samples in an application saves entering them during sample ID registration.

- Default Settings on [Advanced setting] on [Component data] Tab

Template	Manually Input Component	Other Settings
SQFP_Powder	<ul style="list-style-type: none"> Mass (sample weight): 4.0g Height: 8mm Cell: Chempex 1330 	<ul style="list-style-type: none"> Sample film: Prolene
SQFP_Pellet	<ul style="list-style-type: none"> Mass (sample weight): 2.0g Height: 3 mm Diameter: 25mm 	<ul style="list-style-type: none"> Sample film: None
SQFP_Powder	<ul style="list-style-type: none"> Mass (sample weight): 2.0g Height: 10mm Cell: Chempex 1330 	<ul style="list-style-type: none"> Sample film: Prolene
SQFP_Pellet	<ul style="list-style-type: none"> Mass (sample weight): 2.0g Height: 2mm Diameter: 40mm 	<ul style="list-style-type: none"> Sample film: None
SQFP_Liquid	<ul style="list-style-type: none"> Mass (sample weight): 4.0g Height: 8mm Cell: Chempex 1330 	<ul style="list-style-type: none"> Sample film: Prolene

B. Setting Procedure on Measuring Condition Tab

An excitation condition, measurement time and measurement atmosphere for each secondary target can be changed.



- Default Settings on [Measuring condition] Tab

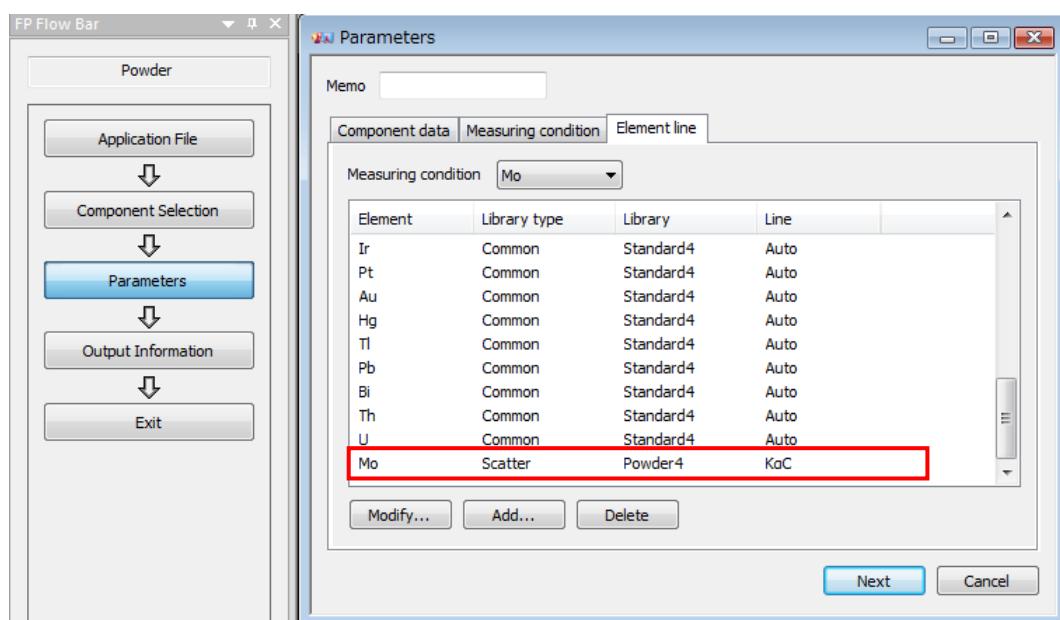
Secondary Target	RX9	Cu	Mo	Al
Tube Voltage (kV)	25	50	50	50
Tube Current (mA)	Auto	Auto	Auto	Auto
Measurement Time (sec.)	100	100	100	200
Measurement Atmosphere	Powder (Loose) Plastic (Loose) Liquid	Helium		
	Powder (Pellet) Plastic (Pellet)	Vacuum		

C. Setting Procedure on Analysis Line Tab

A library can be changed (for example, from the standard library to the matching library), and an analysis line can be changed (for example, from automatic selection to K α).



- In the templates to be used for the scattered ray FP method, Mo-K α scattered rays (K α C and K α T) have been added to the analysis lines. Do not delete any of them, and do not change any line type.

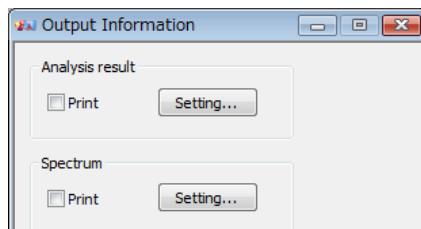


- Default Settings for Analysis Lines (Mo-K α Scattered Rays)

Tab Type	Element	Library	Line Type
Powder	Mo	Powder	K α C
Polymer	Mo	Plastic	K α C, K α T
Liquid	Mo	Liquid	K α C, K α T

vii. Output Setting Procedure

This item consists of [Analysis result] and [Spectrum]. Using [Analysis result], calculated parameters can be set as well as the printing order and output numbers of digits. Using [Spectrum], units and element markers to be used on a qualitative analysis chart can be set.

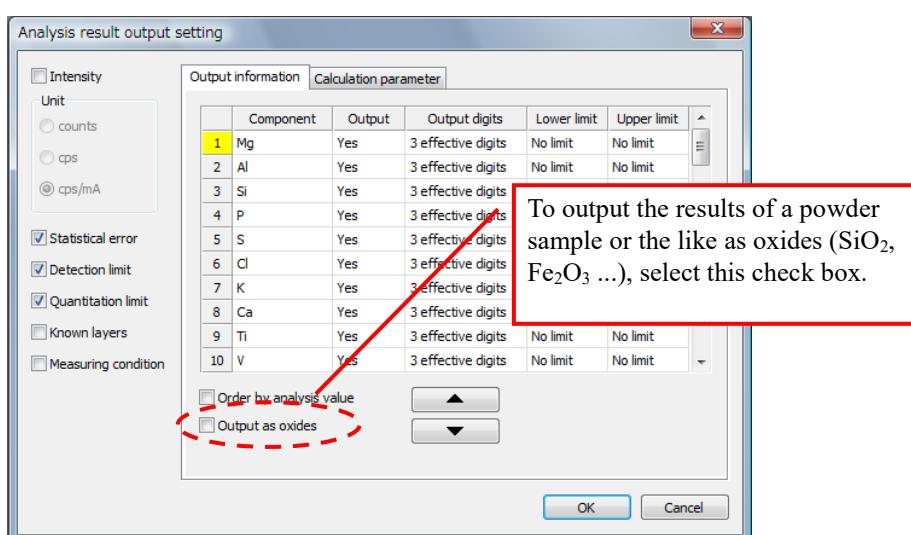


When a check mark is placed at each item, an analyzed result or a spectrum is printed according to the setting content after the completion of an analysis. The output setting items have been deselected in the templates.

A. Output Setting Procedure for Analyzed Result

Click [Setting] in the [Analysis result] group box. Printing conditions can be set.

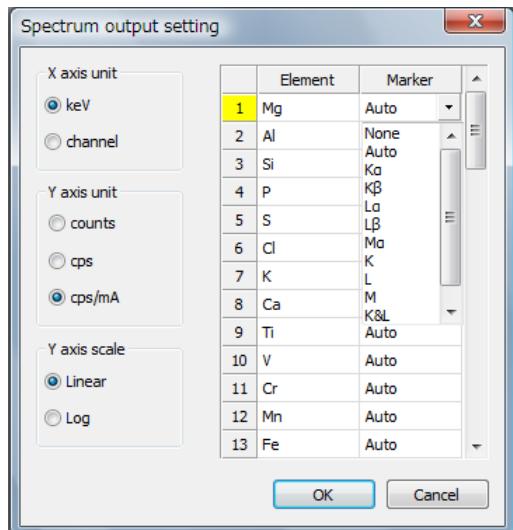
- Select the check boxes of items to be printed.
- For each component, settings can be made for whether it is output or not, the number of output digits, lower limit and upper limit.
- By selecting a component and clicking [\blacktriangle] or [\blacktriangledown], the printing order can be changed.
- When the [Order by analysis value] check box is selected, components are output in order of analyzed value from the highest to the lowest.
- When the [Output as oxides] check box is selected, the concentrations of all components are printed after converting them from those of elements (Si, Fe ...) into those of oxides (SiO_2 , Fe_2O_3 ...).



Output Setting Procedure for Spectrum

Click [Setting] in the [Spectrum] group box.

- The units of the X- and Y-axes of a qualitative chart can be set.
- Markers can be set. The details of the setting are shown in the table below:



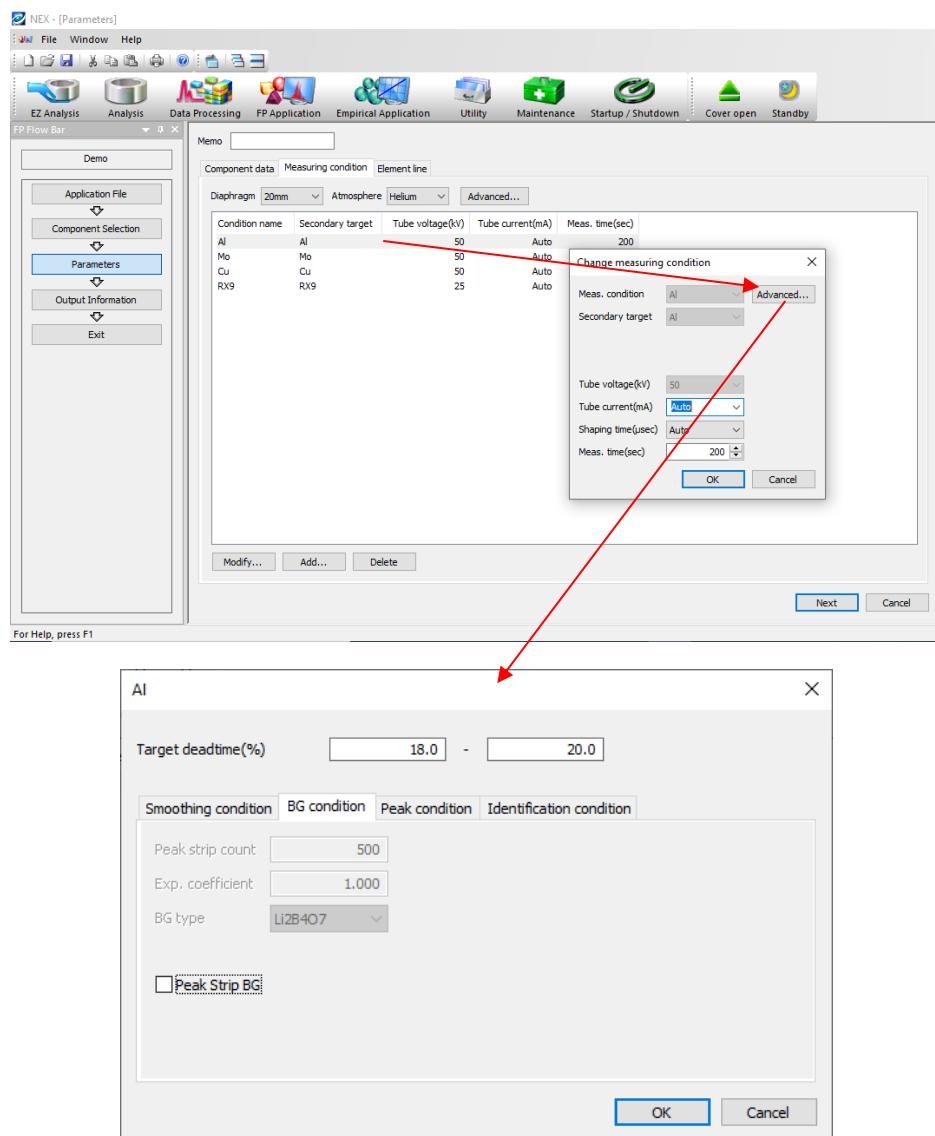
● Marker Setting

Display	Description
None	No marker is displayed.
Auto	Makers are displayed automatically only for detected analysis components.
K α , K β , L α ...	The markers of selected spectral lines are always displayed.
K, L, M, K&L ...	The markers of selected line types are always displayed.

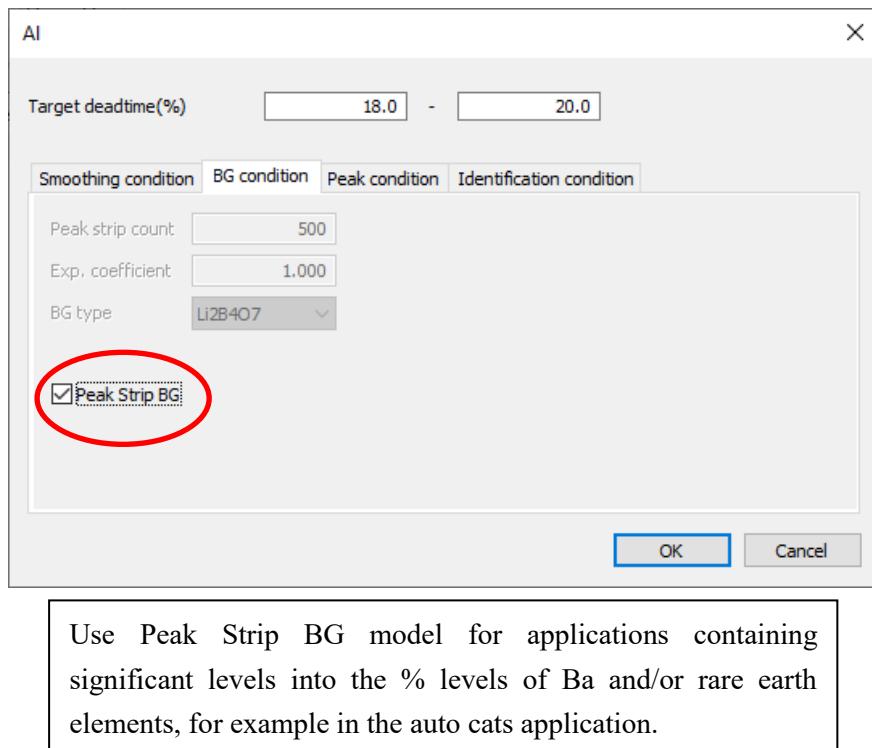
2.5 ADVANCED FP BACKGROUND MODELLING OPTION

The default Rigaku RPF-SQX FP background correction models are excellent for most applications. Beginning with software version NEX 6.16 the user has access to the FP BG modelling schemes.

In the FP application Parameters screen Measuring Condition tab, double-click a condition and click Advanced to view the BG correction model.



Applications that have significant levels of heavy elements in the high energy scatter region of the Al target can make use of the advanced Peak Strip modelling to best model the background when there are percent levels of Ba, La, Ce, Pr, Nd, Pm, etc. in the sample material. Simply select the Peak Strip option to override the default background model.



3. SETTING PROCEDURE FOR MATCHING LIBRARY

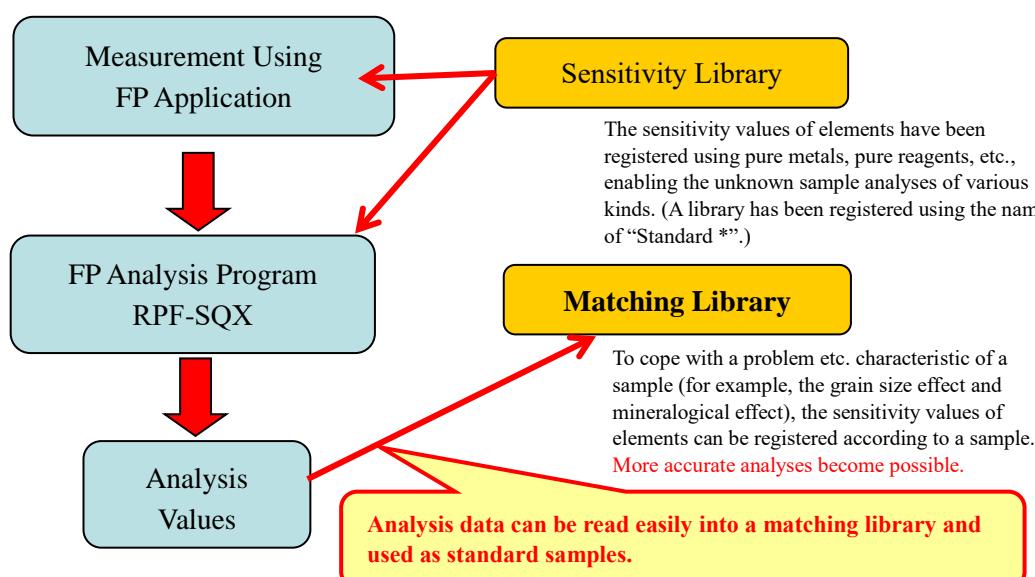
3.1 OUTLINE

The matching library has a function to improve the accuracy of analyses using FP applications, and its concept is shown in the figure below.

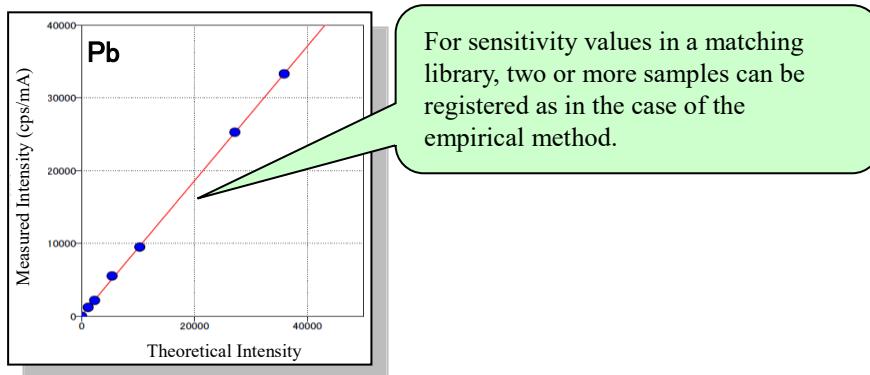
Spectrum data items obtained from a measurement using an FP application are analyzed by the FP analysis program RPF-SQX (Rigaku Profile Fitting – Spectra Quant X), and the analyzed values are calculated. Since spectrometer sensitivity values are necessary for the analysis calculation, the instrument has as standard a “sensitivity library” in which the spectrometer sensitivity values of elements have been stored. In this sensitivity library, the sensitivity values of all elements that can be measured have been registered using pure metals, pure reagents, etc. (It has been registered using the name of “Standard *”.). It enables the unknown sample analyses of various kinds.

The matching library to be introduced here has a function to register the sensitivity values of elements in standard samples resembling an unknown sample and enable more accurate analyses using those sensitivity values, as measures against errors caused by a problem etc., and characteristic of the sample (for example, the grain size effect and mineralogical effect).

The instrument also has a function to make obtained analyzed result data reflected easily in a matching library. For example, when an analysis is made using standard samples and an FP application and the result has some error, the accuracy of the analysis can be improved by registering the data in a matching library as standard samples.



Two or more standard samples can be registered for each element in a matching library as shown below, and it can be used as in the case of the empirical method:

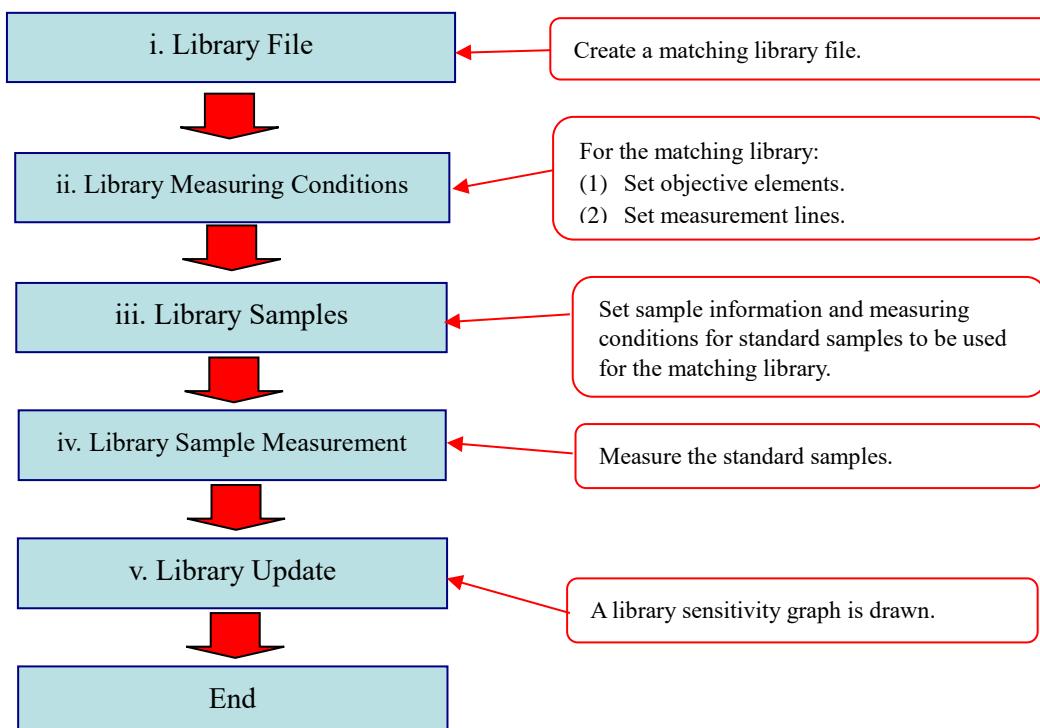


Two examples are given below. The first describes the procedure to create a new library for sulfur in oil. The second describes the procedure to register sensitivity values for a matching library using standard sample data measured with an FP application.

General flowcharts are shown below:

i. Outline of Procedure for Creating New Matching Library

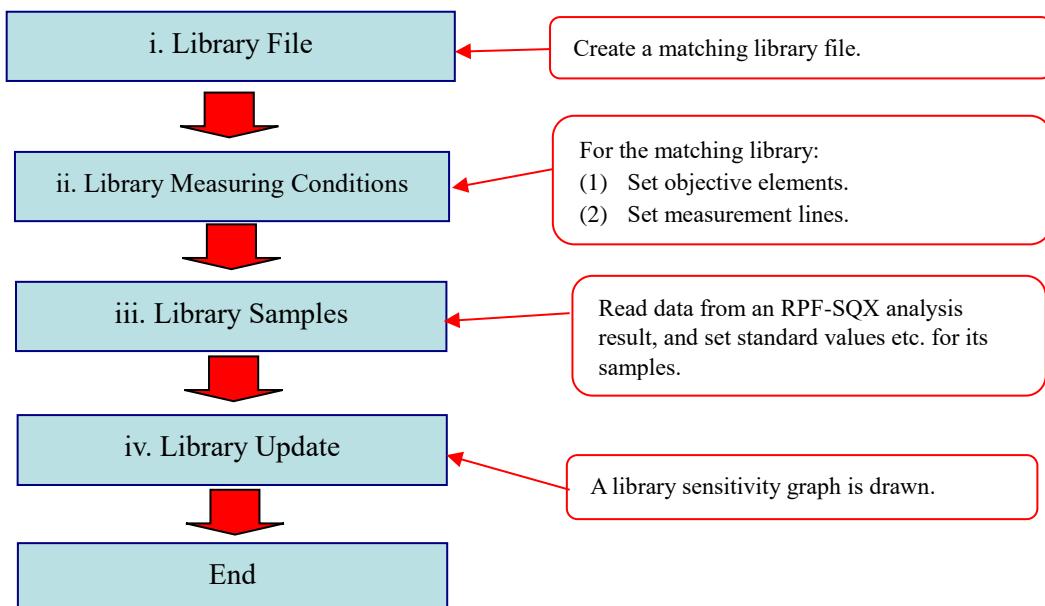
Determine a matching library name, elements to be used for matching and their measurement lines. Register the sensitivity values of the determined measurement lines of the elements using standard samples.



ii. Procedure for Creating Matching Library (by Copying RPF-SQX Result)

The procedure is the same until determining a matching library name, elements to be used for matching and their measurement lines, but sensitivity values are determined referring to X-ray intensities in an RPF-SQX result that has already been obtained.

In the case of a sample that has a complex matrix, more accurate library sensitivity values can be obtained by referring to an RPF-SQX result, in which X-ray intensities have been calculated taking account of the matrix and overlap effects.

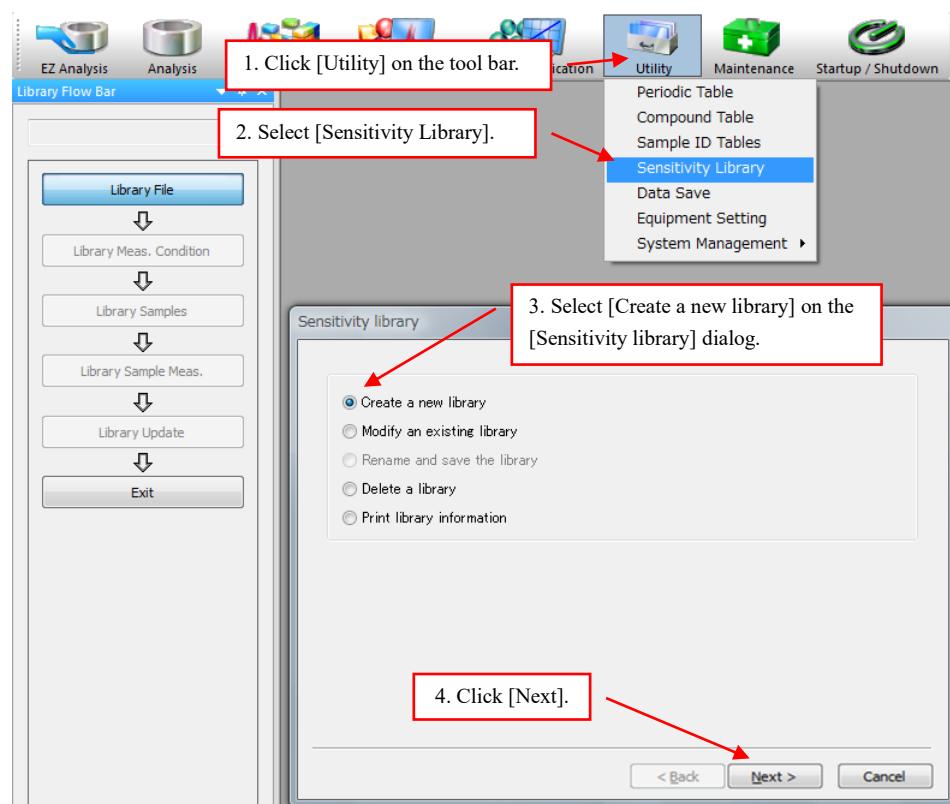


3.2 PROCEDURE FOR CREATING NEW MATCHING LIBRARY

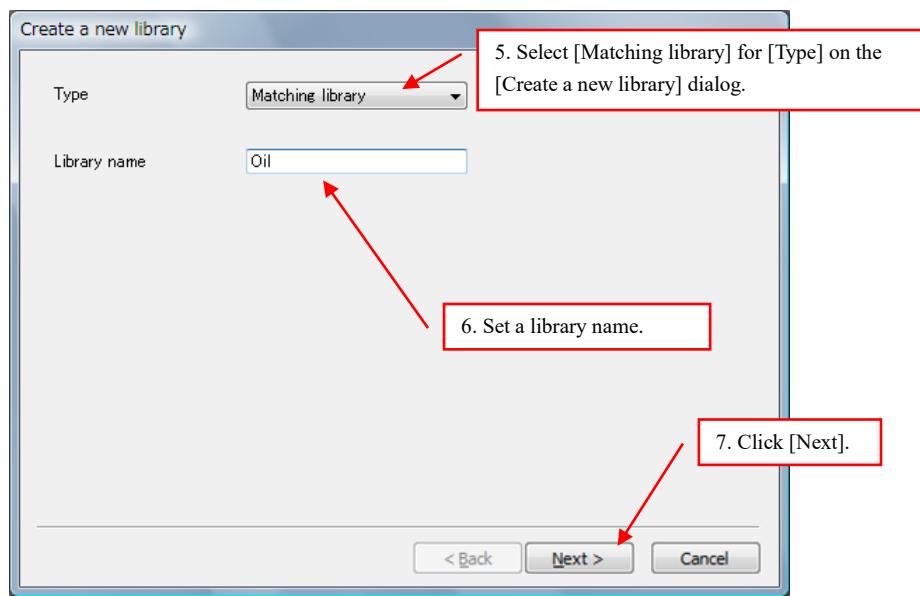
Purpose: The procedure to create a matching library for oil analyses is described below as an example. Buttons are explained in order of setting.

i. Creation of Library File

1. Click [Utility] on the tool bar.
2. Select [Sensitivity Library].
3. Select [Create a new library] on the [Sensitivity library] dialog.
4. Click [Next].



3. SETTING PROCEDURE FOR MATCHING LIBRARY

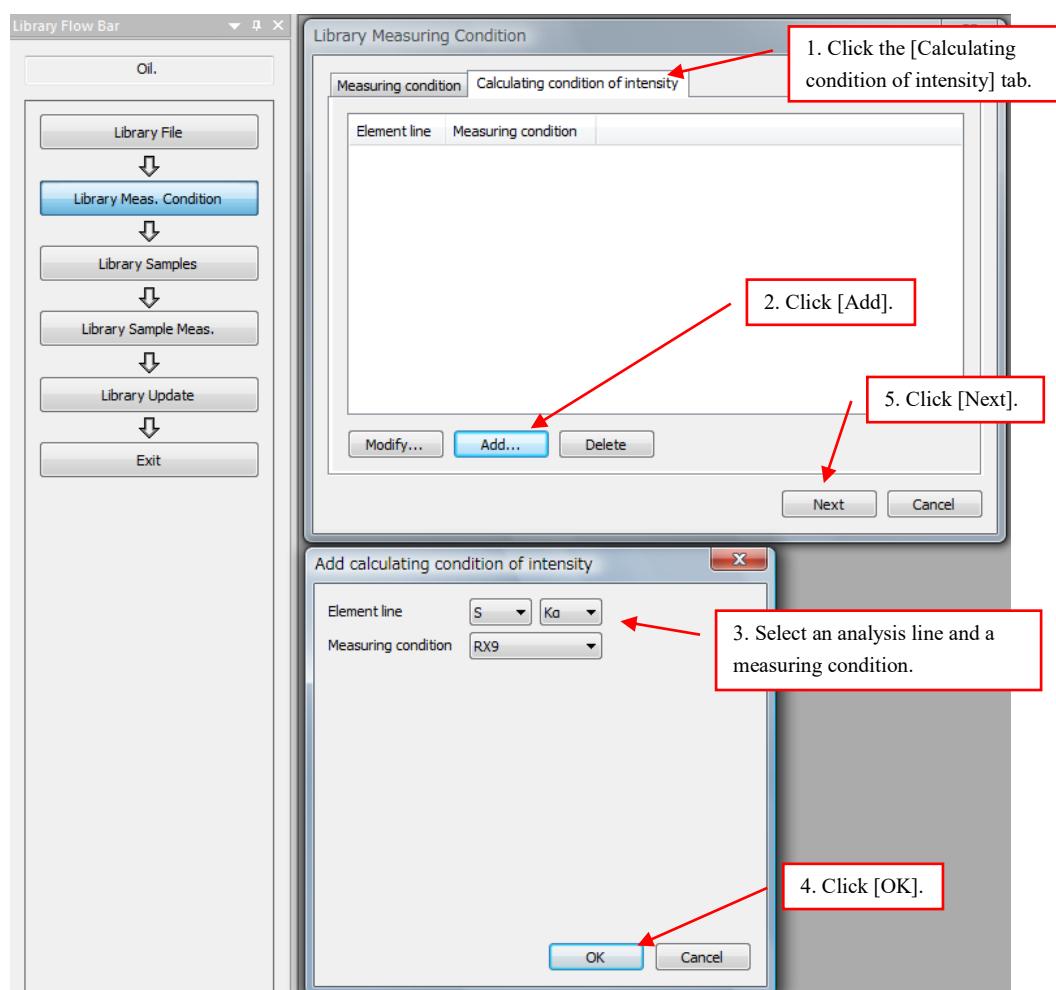


5. Select [Matching library] for [Type] on the [Create a new library] dialog.
6. Set a library name. “Oil” has been set as the library name in the above example.
7. Click [Next].

ii. Library Measuring Conditions

Select here the analysis lines and measuring conditions (secondary targets) to be registered in the library.

1. Click the [Calculating condition of intensity] tab.
2. Click [Add].
3. The [Add calculating condition of intensity] dialog appears. Select here an analysis line and a measuring condition for which to register a sensitivity value. In the example below, S-K α and RX9 have been selected as the analysis line and measuring condition respectively.
4. Click [OK].
5. Click [Next].



3. SETTING PROCEDURE FOR MATCHING LIBRARY



- The relationship between measurement lines and targets used are shown below. In general, the intensities of the K-, L- and M-lines decrease in that order. The intensities of the K α line are higher than those of the K $\beta 1$ line. Therefore, to analyze sulfur, it is recommended that the S-K α line be measured using the RX9 target.

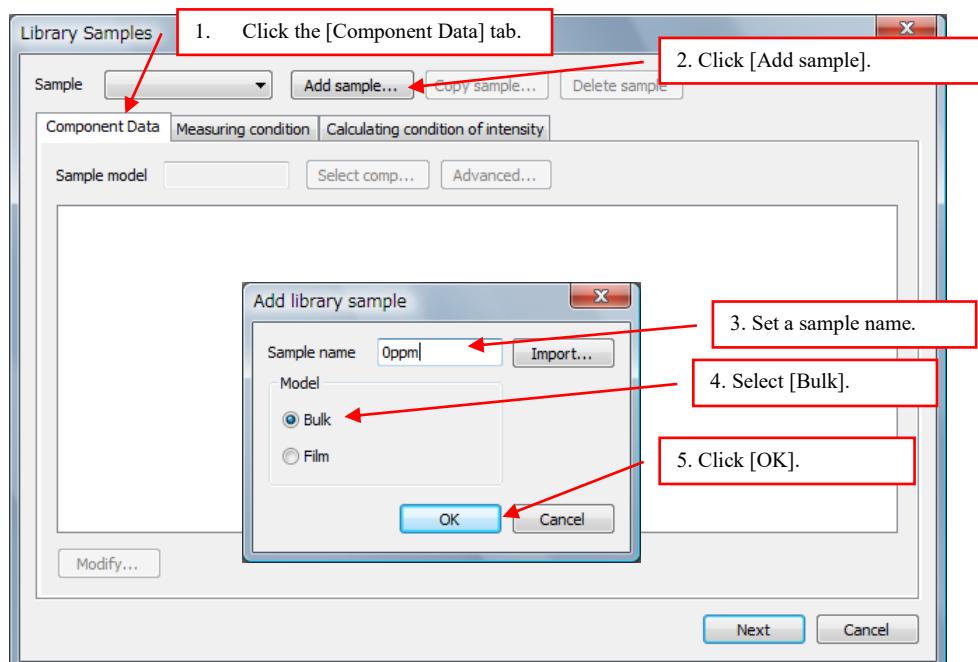
	RX9 Target												Cu Target																			
K-Line	11 Na	12 Mg	13 Al	14 Si	15 P	16 S	17 Cl	18 Ar	19 K	20 Ca	21 Sc	22 Tl	23 V	24 Cr																		
L-Line	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr	37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe	55 Cs	56 Ba	57 La	58 Ce	59 Pr	60 Nd	61 Pm	
M-Line						74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi																	
Energy	1					2										3			4			5						keV				
	Mo Target																															
K-Line	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr	37 Rb	38 Sr	39 Y																	
L-Line	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn	87 Fr	88 Ra	89 Ac	90 Th	91 Pa	92 U	
Energy	6		7		8		9		10		11		12		13		14		15	keV												
	Al Target																															
K-Line	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe	55 Cs	56 Ba	57 La	58 Ce	59 Pr	60 Nd											
Energy	16		20		25		30									40	keV															

iii. Library Samples

Set standard samples to be used for the matching library measurement. The procedure to set standard samples with sulfur concentrations of 0, 2, 5, 10, 20 and 50ppm in oil is described below as an example:

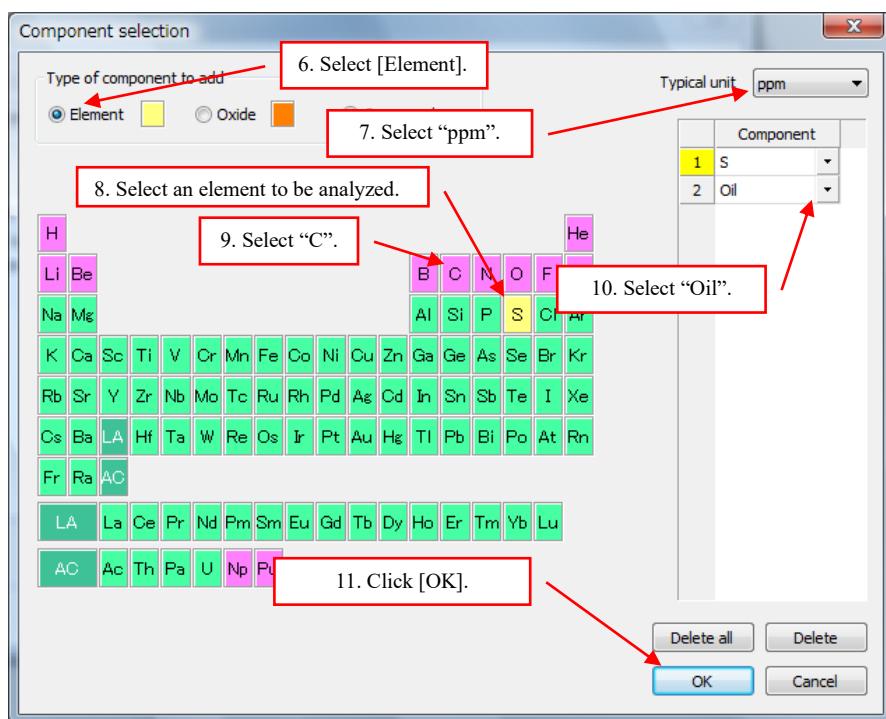
(A) Setting Procedure for Component Information

1. Click the [Component Data] tab.
2. Click [Add sample].
3. Set a sample name on the [Add library] dialog. Set “0ppm” here.
4. Select [Bulk] or [Film] for the model. Select [Bulk] here.
5. Click [OK].



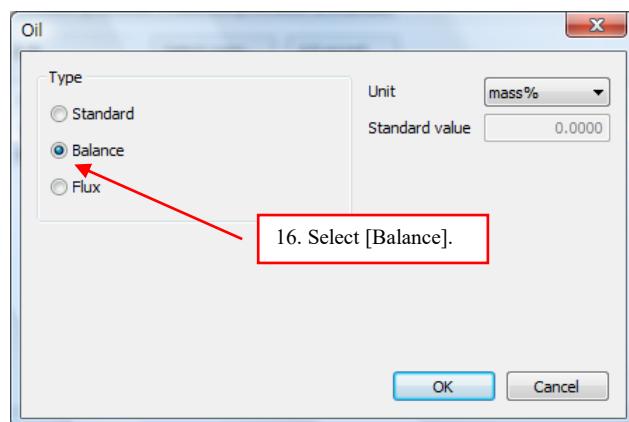
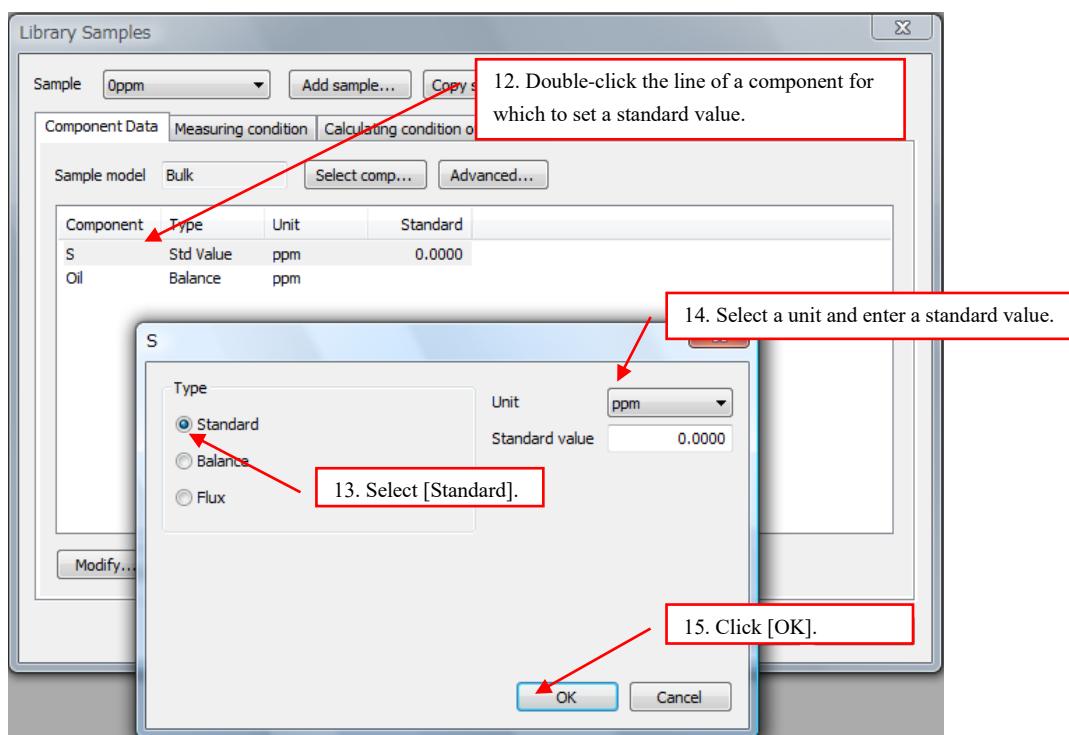
3. SETTING PROCEDURE FOR MATCHING LIBRARY

6. The [Component selection] dialog appears. Select the type (element, oxide or compound) of a component to be added in the [Type of component to add] group box. Select [Element] because sulfur in light oil is to be analyzed.
7. Set a unit to be used mainly. Set “ppm” here.
8. Click an objective element on the periodic table. Set sulfur here as the element to be measured. The selected component is added to the component list on the right of the window.
9. Select Oil (CH_2) as the balance component. First, select “C”.
10. Then select “Oil” on the pull-down menu of the component “C”.
11. Click [OK].



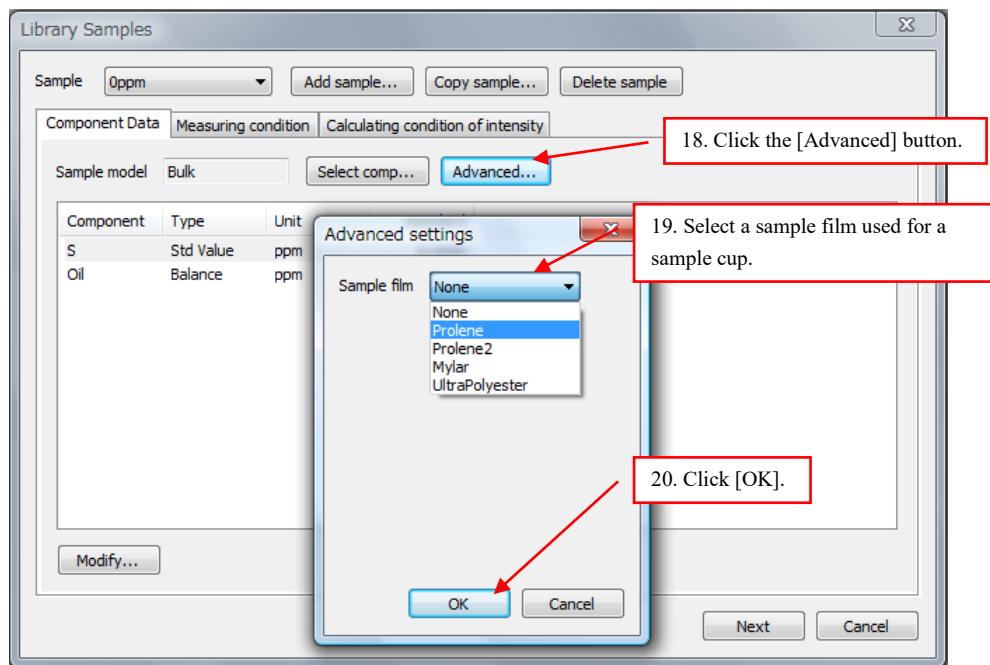
- Because the FP method is used, make a setting such that the total of the concentrations of elements falls within the range of 99 to 101%. For example, when there are components that cannot be measured, such as H_2O and CH_2 , the base component must be set for the FP method calculation. The base component is set as the balance in the software. In the case of sulfur analyses in light oil, sulfur is the element to be analyzed, and Oil (CH_2) is the balance component.

12. Double-click the line of a component for which to set a standard value on the [Component Data] tab. Double-click "S" here.
13. Select [Standard], [Balance] or [Flux] in the [Type] group box on the component dialog. Select [Standard] here.
14. When [Standard] is selected, select a unit and enter a standard value.
15. Click [OK].
16. Select [Balance] for "Oil (CH₂)".
17. When there are other elements, set parameters in the same procedure.



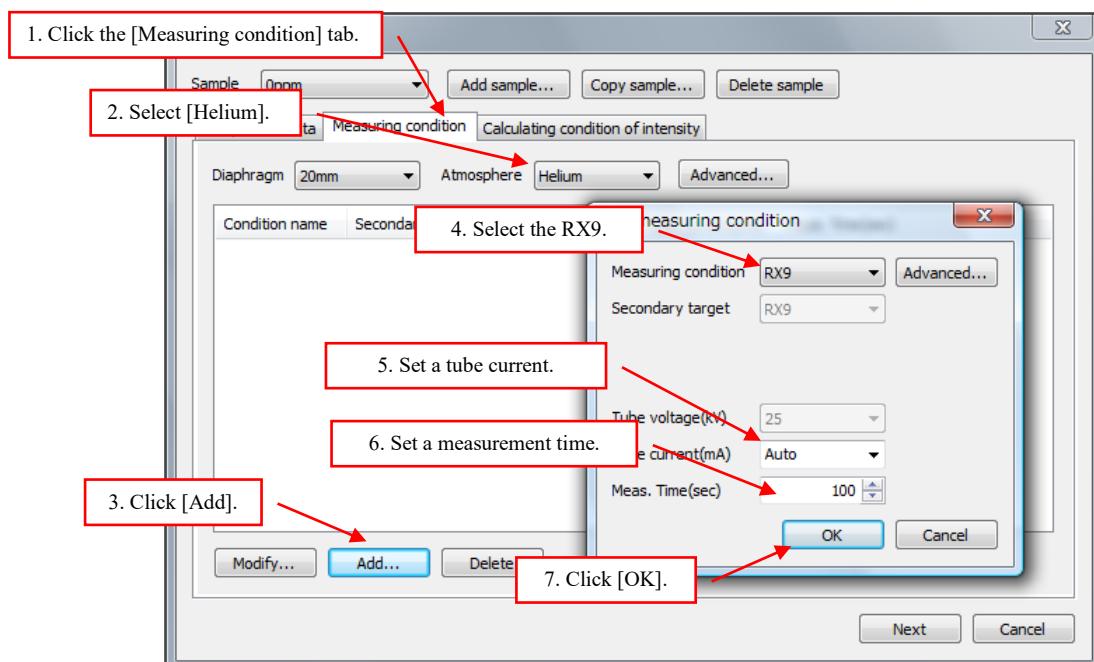
3. SETTING PROCEDURE FOR MATCHING LIBRARY

18. Click the [Advanced] button on the [Component Data] tab.
19. On the [Advanced setting] dialog, select a sample film used for a sample cup from (1) Prolene: 4 μ m Prolene film, (2) Prolene 2: double Prolene film, (3) Mylar: 3.6 μ m Mylar and (4) Ultra-Polyester: 1.5 μ m ultra-polyester. A correction is made for absorption by the film.
20. Click [OK].



(B) Setting Procedure for Measuring Conditions

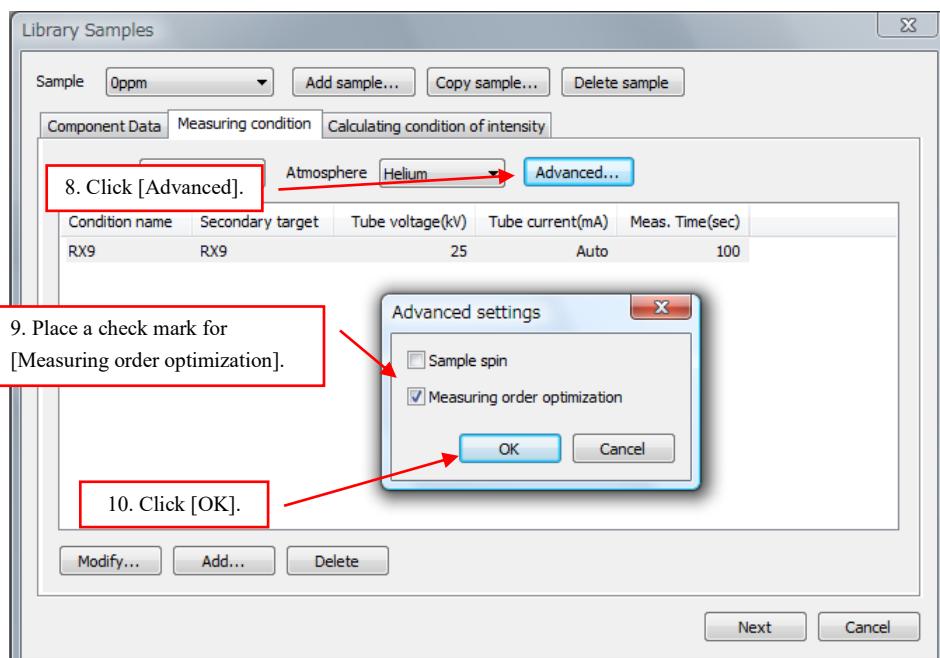
1. Click the [Measuring condition] tab.
2. Select the measurement atmosphere from the vacuum, air and helium. Select the helium here because the sample is a liquid solution.
3. To set a measuring condition, click [Add].
4. Select a secondary target to be used for a measurement from the Mo, Al, RX9 and Cu on the [Measuring condition] dialog. Select the RX9 here.
5. Set a tube current.
6. Set a measurement time.
7. Click [OK].



- The tube voltage has been set to 25kV only for the RX9 target and to 50kV for other targets. When [Auto] is selected for the tube current, it is controlled such that the total counting rate becomes a value at which the dead time of the detector is 25% or less.
- Rigaku recommends that the current be set to 1mA for the RX9 target and to 0.5mA for other targets so that the output of the X-ray tube becomes 25W.

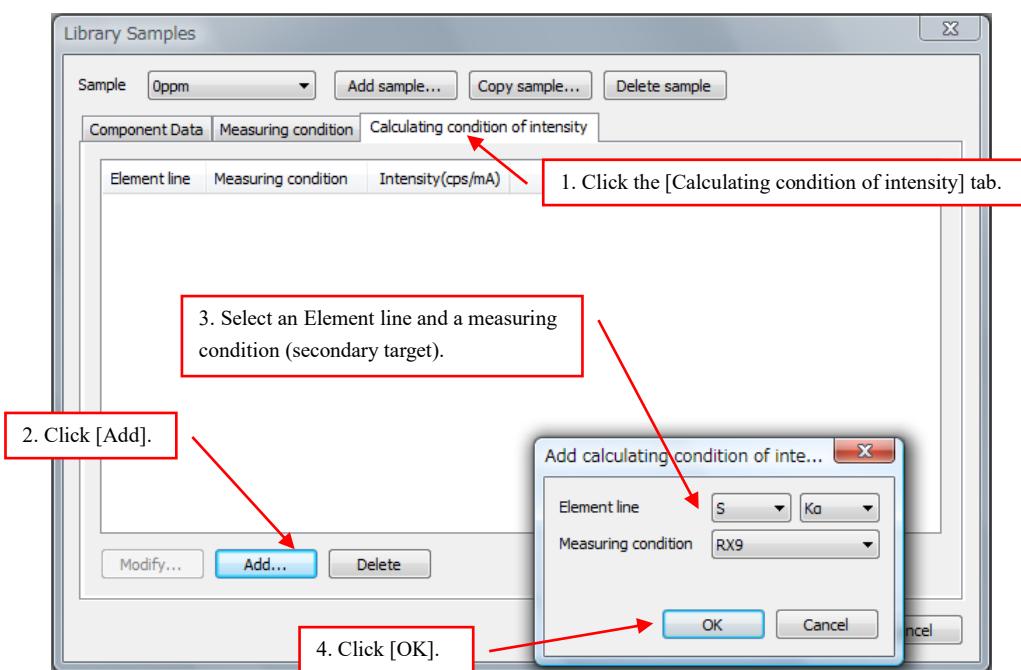
3. SETTING PROCEDURE FOR MATCHING LIBRARY

8. Click [Advanced] on the [Measuring condition] tab.
9. Place a check mark for [Measuring order optimization] on the [Advanced setting] dialog.
To make a measurement using the sample spin, place a check mark for [Sample spin].
10. Click [OK].



(C) Setting Procedure for Intensity Calculation Condition

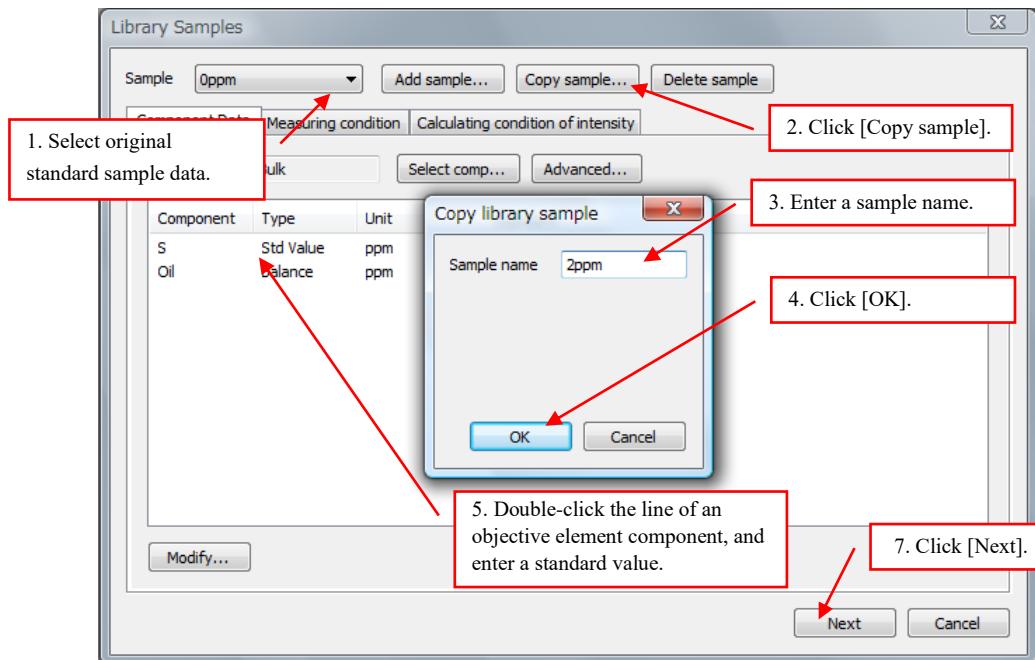
1. Click the [Calculating condition of intensity] tab.
2. Click [Add].
3. Select an analysis line and a measuring condition (secondary target) on the [Add calculating condition of intensity] dialog. The setting shown below is to analyze S-K α using the RX9.
4. Click [OK].



(D) Multiple Copying of Standard Sample

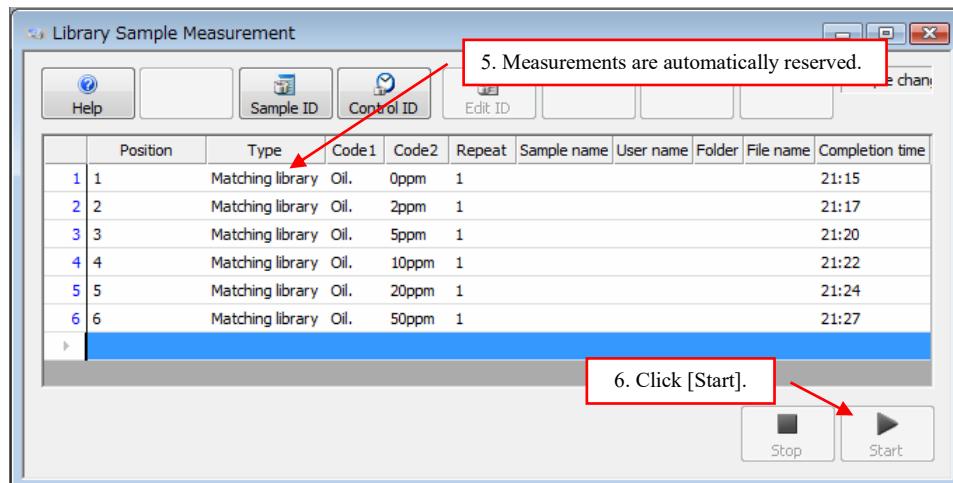
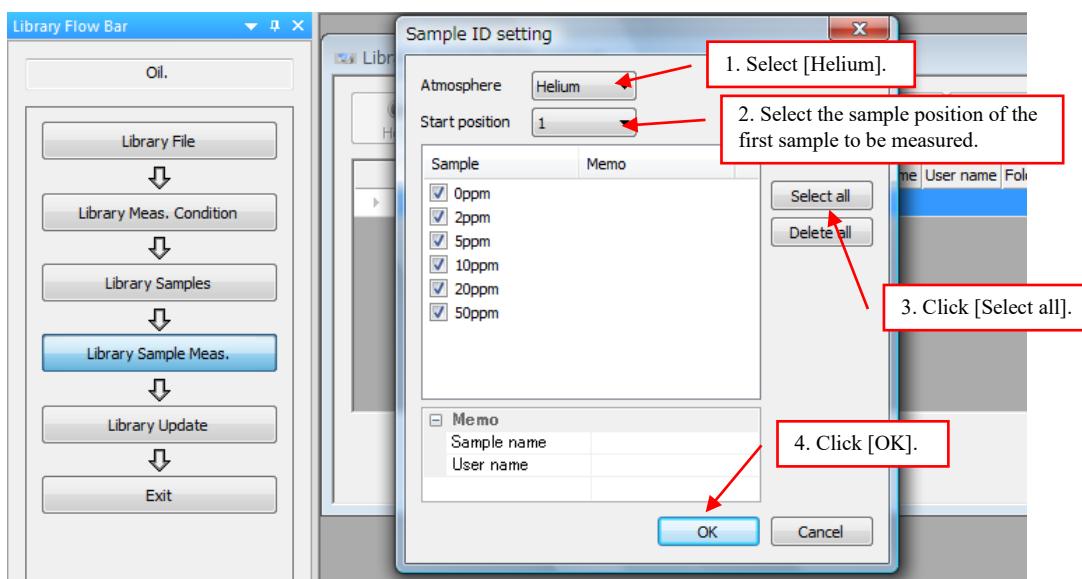
When at least one standard sample has been set, two or more standard samples of the same kind can be set easily by copying with different names and changing standard values.

1. Select original standard sample data.
2. Click [Copy sample] on the [Library Sample] dialog.
3. Enter a sample name on the [Copy library sample] dialog. Enter “2ppm” here.
4. Click [OK].
5. Make sure that the sample name has been changed to “2ppm”, double-click the line of an objective element component, and enter a standard value.
6. Repeat steps 1 to 5 for other standard samples.
7. Click [Next].



iv. Library Sample Measurement

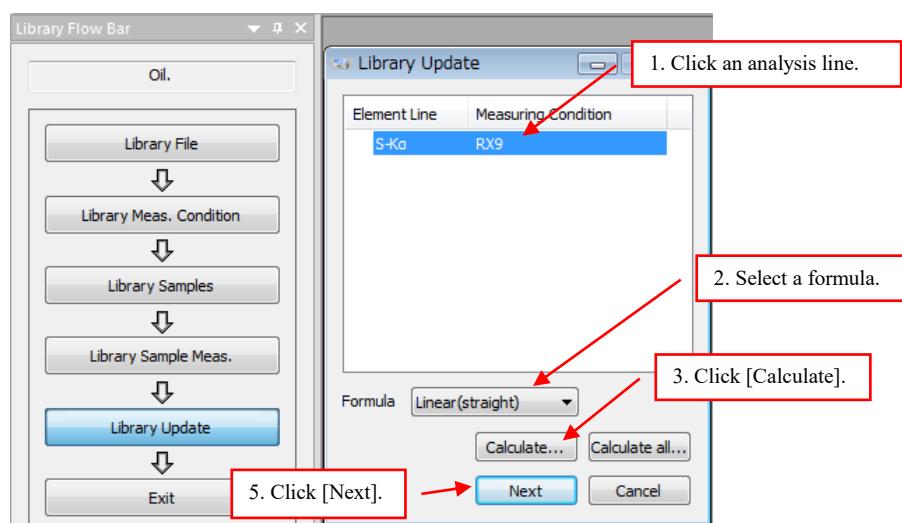
1. On the [Sample ID setting] dialog, select the measurement atmosphere that has been set in the measuring condition. Select the helium here.
2. Select the sample position of the first sample to be measured. (The sample positions of other samples are automatically determined.)
3. Select standard samples to be measured. Here, set “1” for the first sample position, and make a setting (click [Select all]) such that all standard samples are measured.
4. Click [OK].
5. Measurements are automatically reserved on the [Library Sample Measurement] dialog.
6. Put samples on the sample changer, and click [Start].



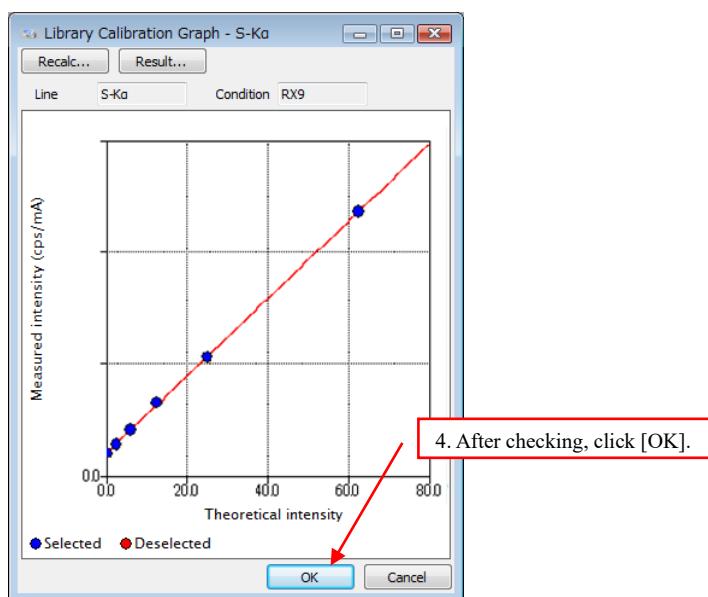
v. Library Update

After the measurement of library samples, the library update screen appears automatically.

1. Click an analysis line for which to draw a sensitivity graph.
2. Select the ratio (one-point method), straight line (linear equation) or curve (quadratic equation) for the formula. Select the linear equation here. When one sample is used, select the ratio (one-point method).
3. Click [Calculate].

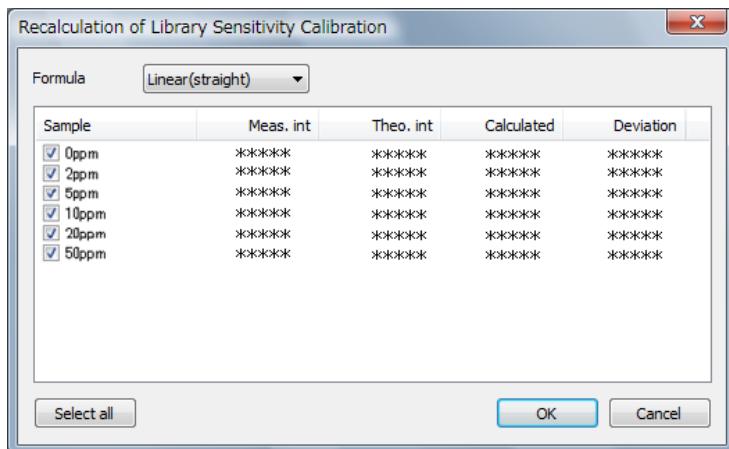


4. The [Library Calibration Graph] dialog appears. After checking, click [OK].
5. After entering all sensitivity values, click [Next] to complete the procedure.

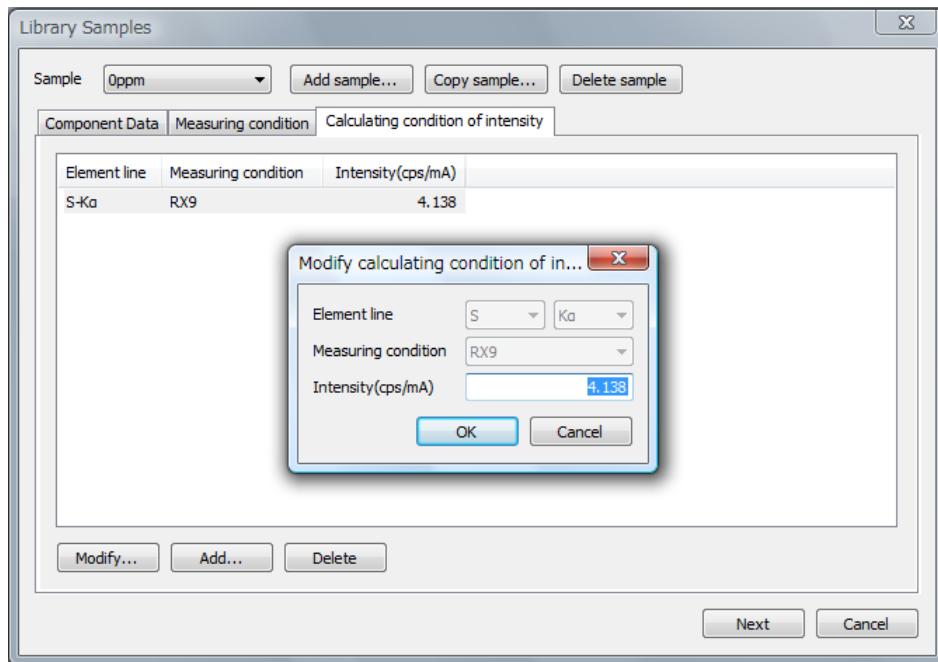




- By clicking [Recalc] on the [Library Calibration Graph] dialog, a formula can be changed and a standard sample with a distant correlation can be excluded.



- By selecting the [Calculating condition of intensity] tab on the [Library Sample] dialog, the X-ray intensity of each element can be edited.



- A negative intercept is not recommended in the sensitivity registration. When it becomes negative, the ratio (one-point method) is recommended.

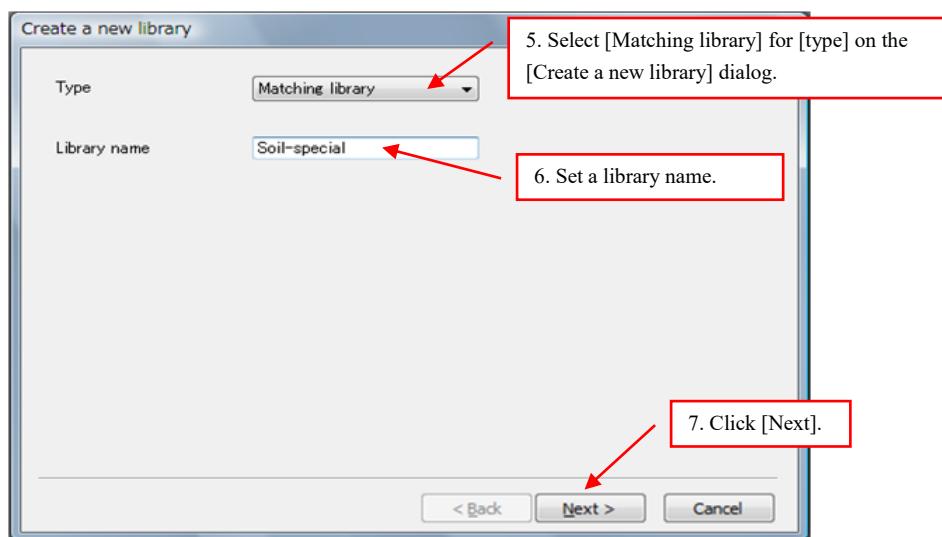
3.3 PROCEDURE FOR CREATING MATCHING LIBRARY (BY COPYING RPF-SQX RESULT)

Purpose: The procedure to create a matching library for soil analyses is described below as an example. Described below is the procedure to measure the soil standard sample NIST2709 using the scattered ray RPF-SQX and create a sensitivity library using the measurement result.

i. Creation of Library File

The procedure is basically the same as the setting procedure described in Section 3.2.

1. Click [Utility] on the tool bar.
2. Select [Sensitivity Library].
3. Select [Create a new library] on the [Sensitivity library] dialog.
4. Click [Next].

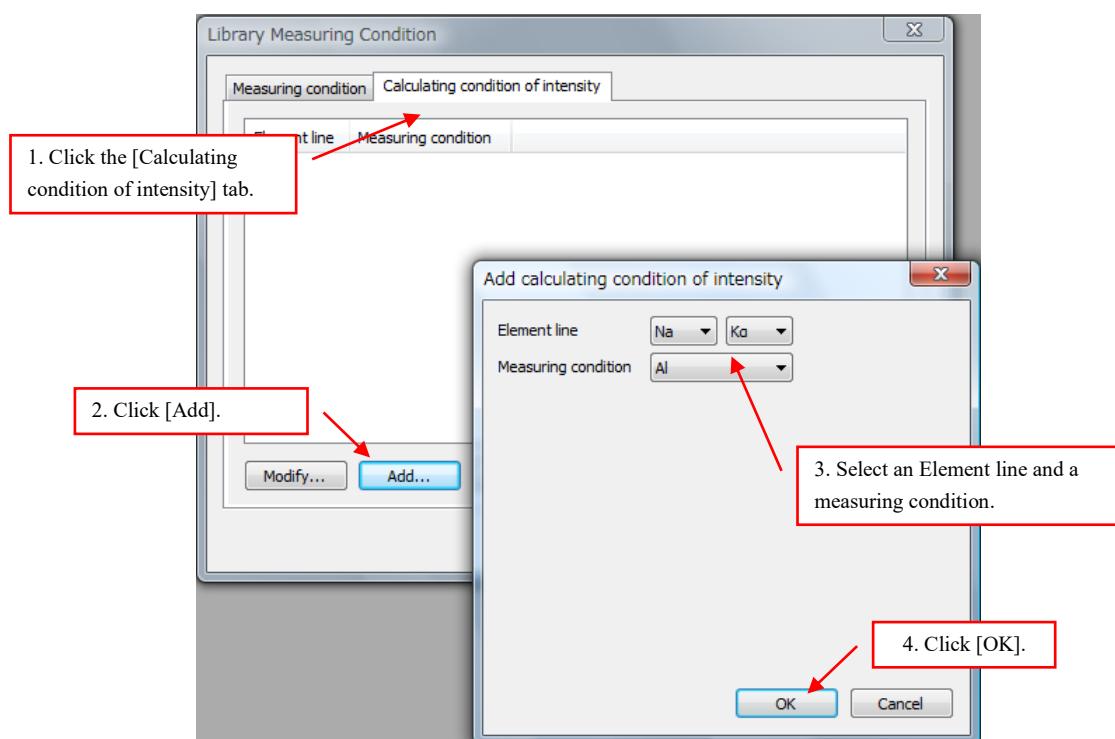


5. Select [Matching library] for [Type] on the [Create a new library] dialog.
6. Set a library name. “Soil-special” has been set as the library name in the above example.
7. Click [Next].

ii. Library Measuring Conditions

Select here the analysis lines and measuring conditions (secondary targets) of objective elements to be registered in the library. The objective elements are Na, Mg, Al, Si, P, S, Cl, K, Ca and Fe.

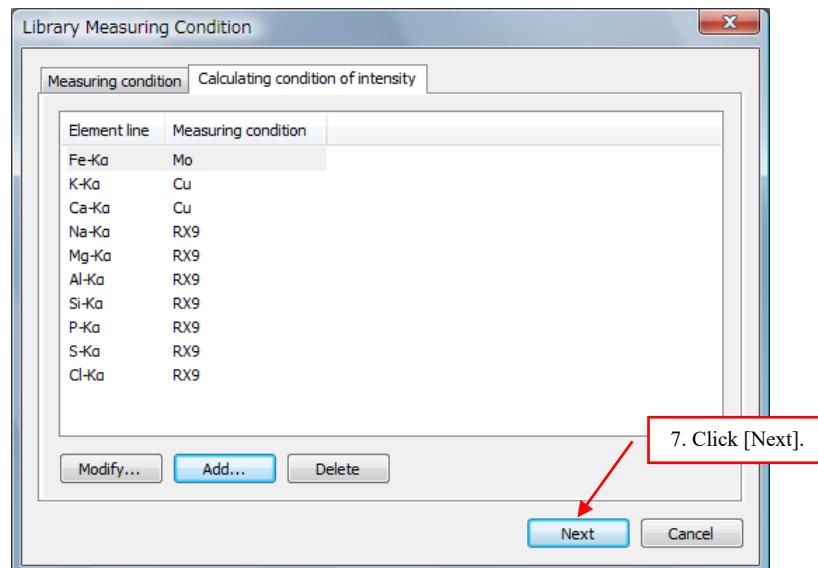
1. Click the [Calculating condition of intensity] tab.
2. Click [Add].
3. The [Add calculating condition of intensity] dialog appears. Select here an Element line and a measuring condition for which to register a sensitivity value. In the example below, Na-K α and RX9 have been selected as the Element line and measuring condition respectively.
4. Click [OK].
5. Enter data for the objective elements.



3. SETTING PROCEDURE FOR MATCHING LIBRARY

6. The Element lines and measuring conditions that have been set are shown below.
7. Click [Next].

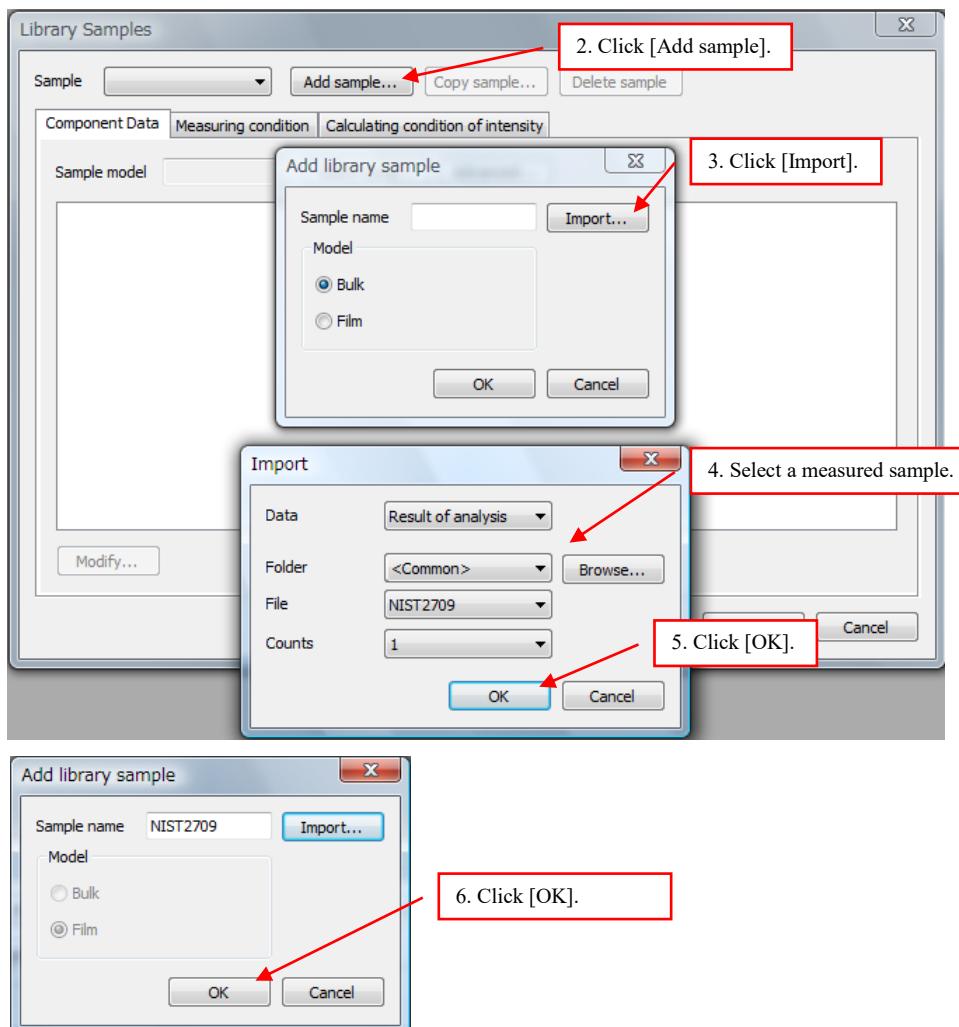
Element	Element Line	Measuring Condition (Secondary Target)
Na	Na-K α	RX9
Mg	Mg-K α	RX9
Al	Al-K α	RX9
Si	Si-K α	RX9
P	P-K α	RX9
S	S-K α	RX9
Cl	Cl-K α	RX9
K	K-K α	Cu
Ca	Ca-K α	Cu
Fe	Fe-K α	Mo



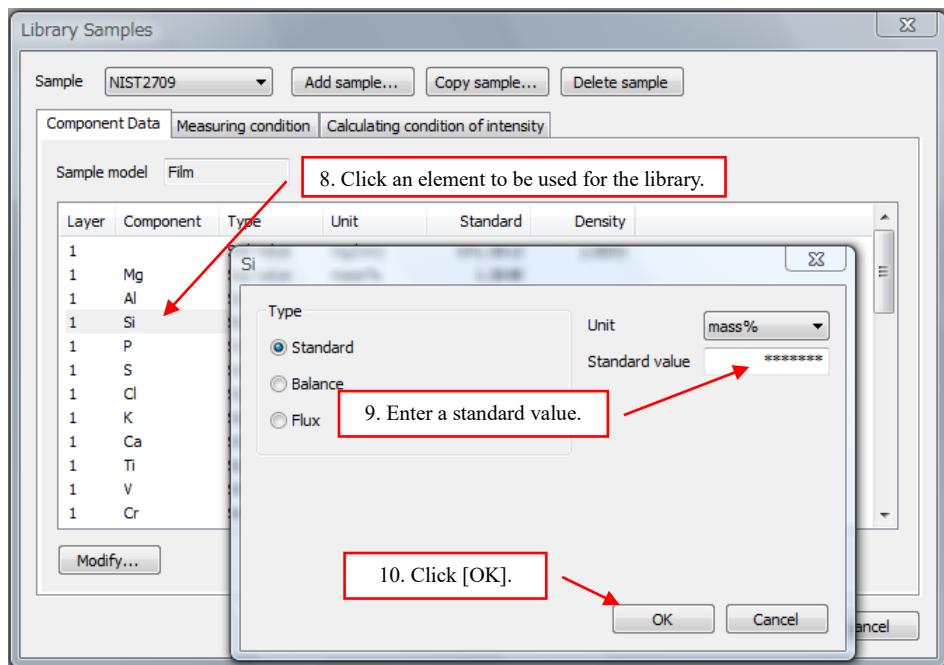
iii. Library Samples

Since standard samples are copied from the RPF-SQX measurement results, the measuring conditions etc. of library samples basically need not be determined. Only the input of standard values is needed for objective elements.

1. Click [Library Samples] on the flow bar.
2. Click [Add sample].
3. The [Add library sample] dialog appears. Click [Import].
4. The [Import] dialog appears. Select the file of a measured sample (to be used as a standard sample) from the analyzed results.
5. Click [OK].
6. Click [OK].
7. The file is read out.



3. SETTING PROCEDURE FOR MATCHING LIBRARY



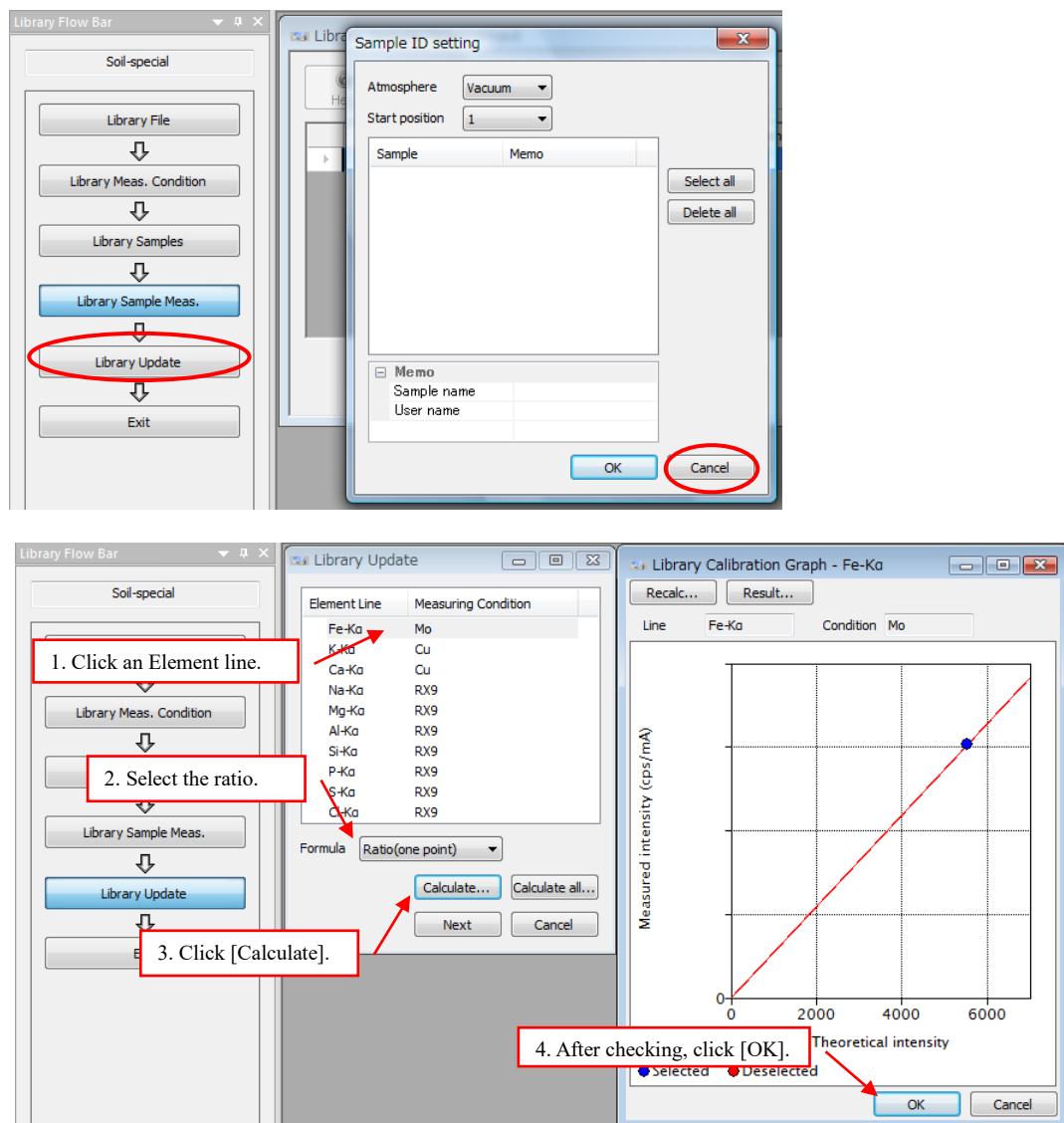
8. Click an element to be used for the library.
9. An analysis value is displayed. Enter a standard value.
10. Click [OK].
11. In the same way, enter the standard values of other elements to be used for the library.
12. Click [Next] when finished.



- Since the balance is automatically set for a sample measured using the scattered ray RPF-SQX analysis, registration can be carried out easily only by entering a standard value.
- In the case of the RPF-SQX analysis that does not use scattered rays, an error message will appear if the total of concentrations is not 100%. In this case, registration becomes possible when the element with the highest concentration is set as the balance.

iv. Library Update

After the registration of library samples, the library sample measurement screen appears automatically. Click [Cancel] on the [Sample ID setting] dialog, and click [Library Update] on the flow bar.

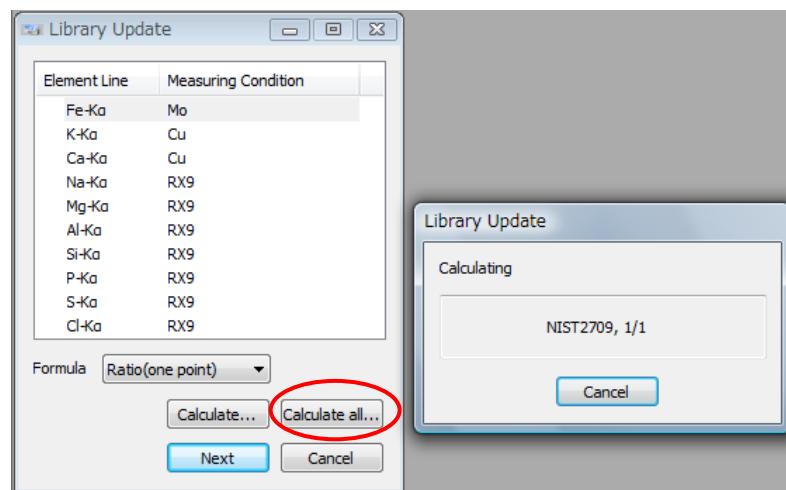


1. Click an Element line for which to draw a sensitivity graph.
2. Select the ratio (one-point method), straight line (linear equation) or curve (quadratic equation) for the formula. Because only one standard sample is used, select the ratio.
3. Click [Calculate].
4. The [Library Calibration Graph] dialog appears. After checking, click [OK].
5. After entering all sensitivity values, click [Next] to complete the procedure.

3. SETTING PROCEDURE FOR MATCHING LIBRARY



- A negative intercept is not recommended in the sensitivity registration. When it becomes negative, the ratio (one-point method) is recommended.
- When two or more standard samples and elements are used, by clicking [Calculate all], the sensitivity calibration can be carried out taking account of all correlations.



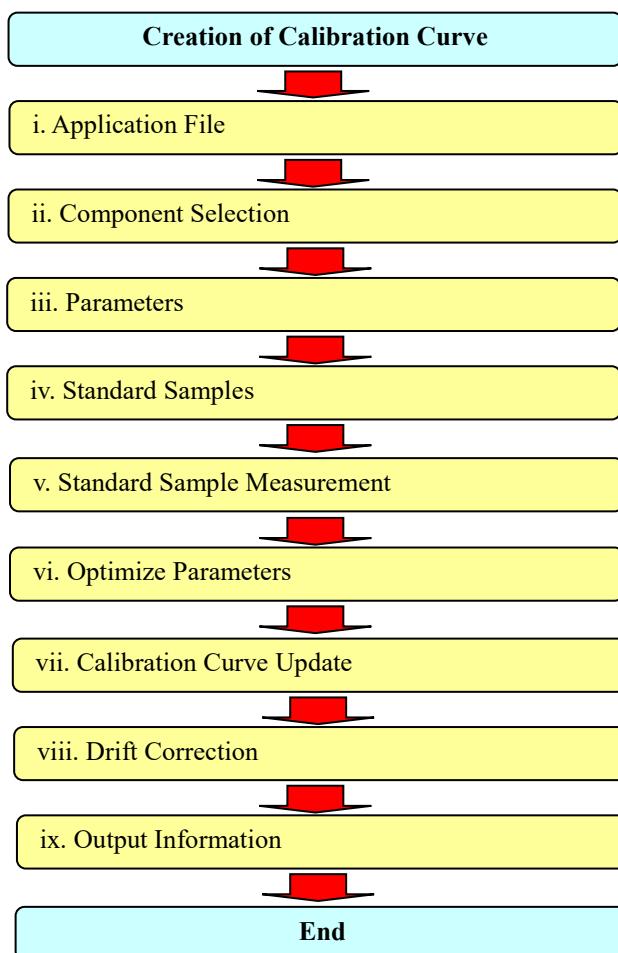
4. CREATION OF EMPIRICAL APPLICATION

4.1 OUTLINE

A calibration curve for a quantitative analysis can be created using assayed standard samples. For the analysis of trace sulfur in light oil, the procedures for creating a calibration curve using an empirical application and for the routine control analyses including the drift correction are introduced below as an example.

4.2 CREATION OF CALIBRATION CURVE

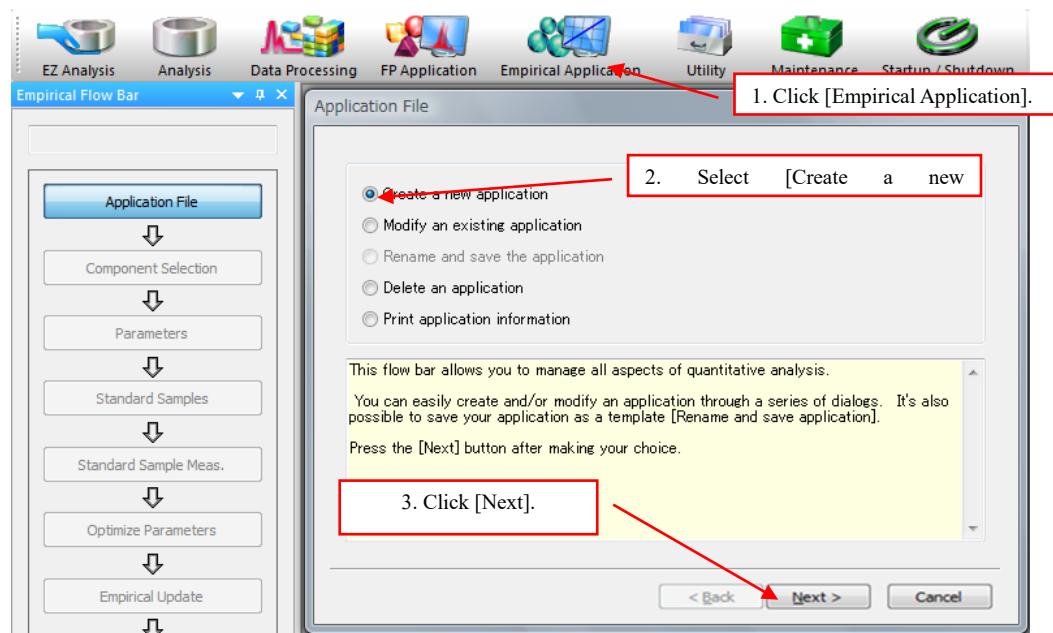
Shown below is the flow of the creation of a calibration curve using an empirical application. Descriptions are given according to this flow.



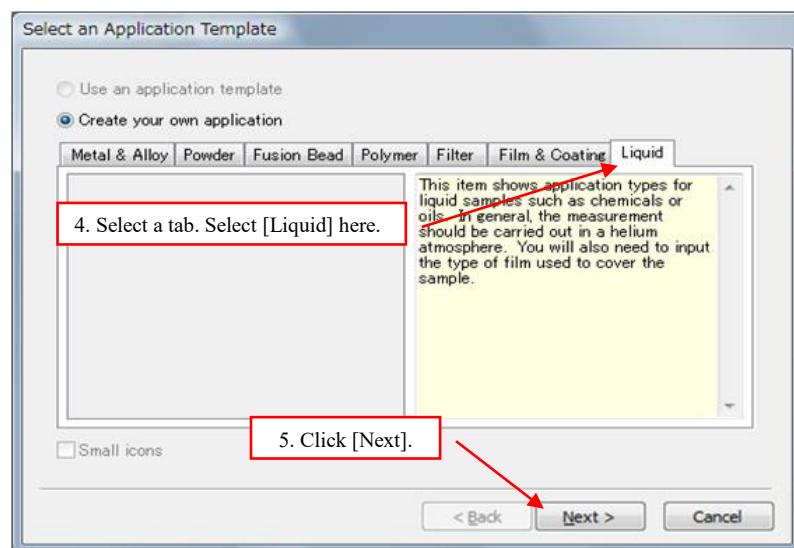
4. CREATION OF EMPIRICAL APPLICATION

i. Creation of Application File

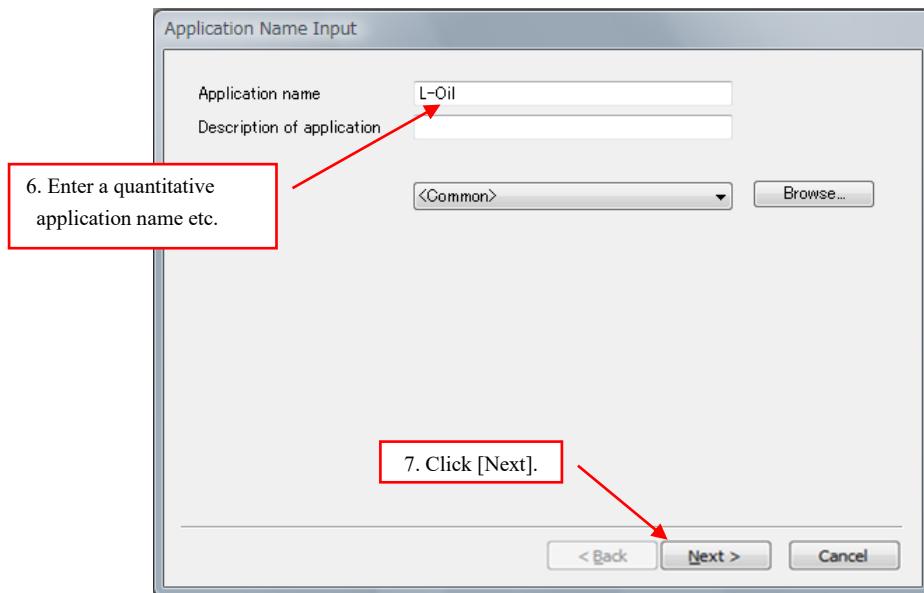
1. Click [Empirical Application] on the tool bar.
2. Select [Create a new application] on the [Application File] dialog.
3. Click [Next].



4. On the [Select an Application Template] dialog, select [Create your own application] and an appropriate application tab. Select [Liquid] here.
5. Click [Next].

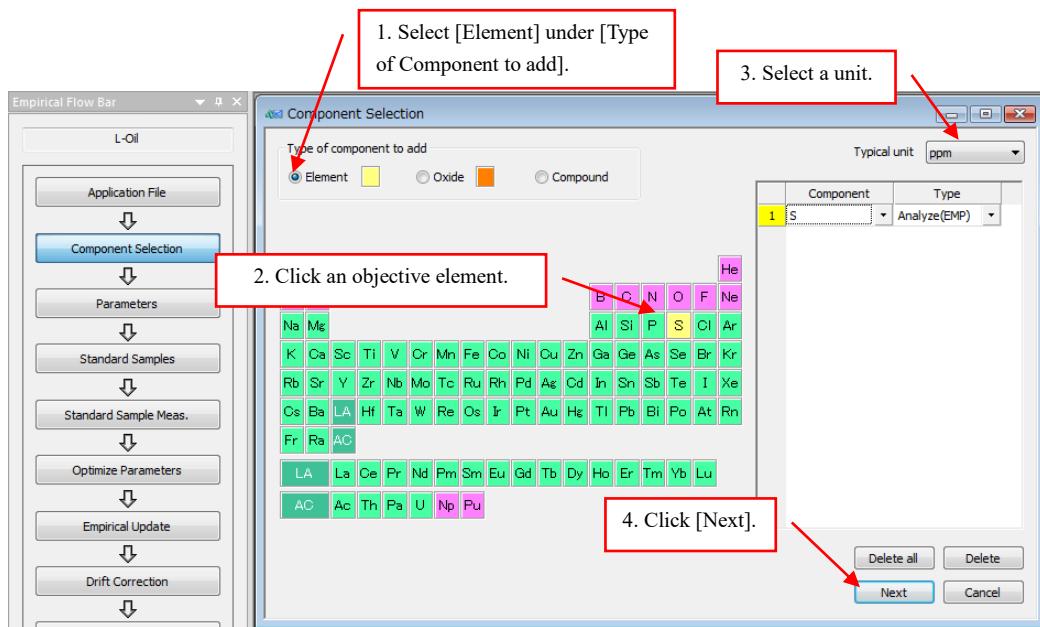


6. Enter an application name and a description. Select a folder in which to save an empirical application. The folder for saving can be selected or created using [Browse]. Since sulfur in light oil is to be analyzed, the quantitative application is named “L-Oil”.
7. Click [Next].



ii. Component Selection

1. Select an element for a quantitative analysis on the [Component Selection] dialog. Select [Element], [Oxide] or [Compound] under [Type of component to add]. Because an element in light oil is to be analyzed, select [Element] here.
2. Click an objective element. Select “S” here.
3. Select a unit. Select “ppm” here.
4. Click [Next].



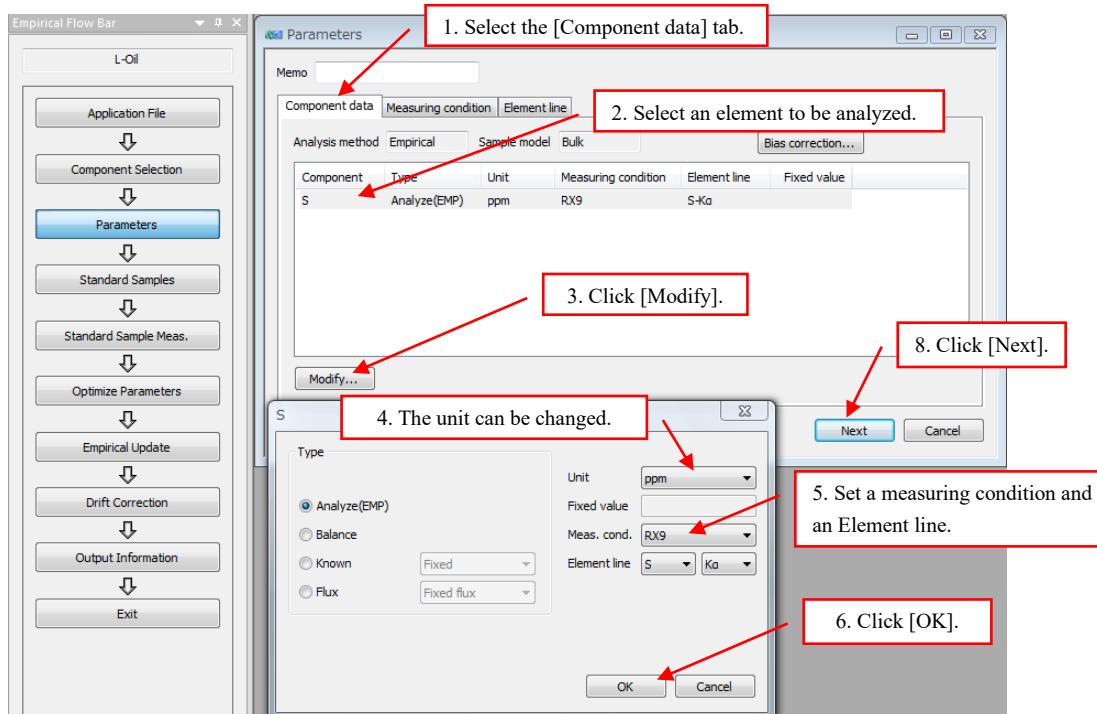
iii. Analysis Parameter Setting

Set parameters on the [Component data], [Measuring condition] and [Element line] tabs.

(A) Setting Procedure for Component Data

Set here a unit, measuring condition (secondary target) and Element line.

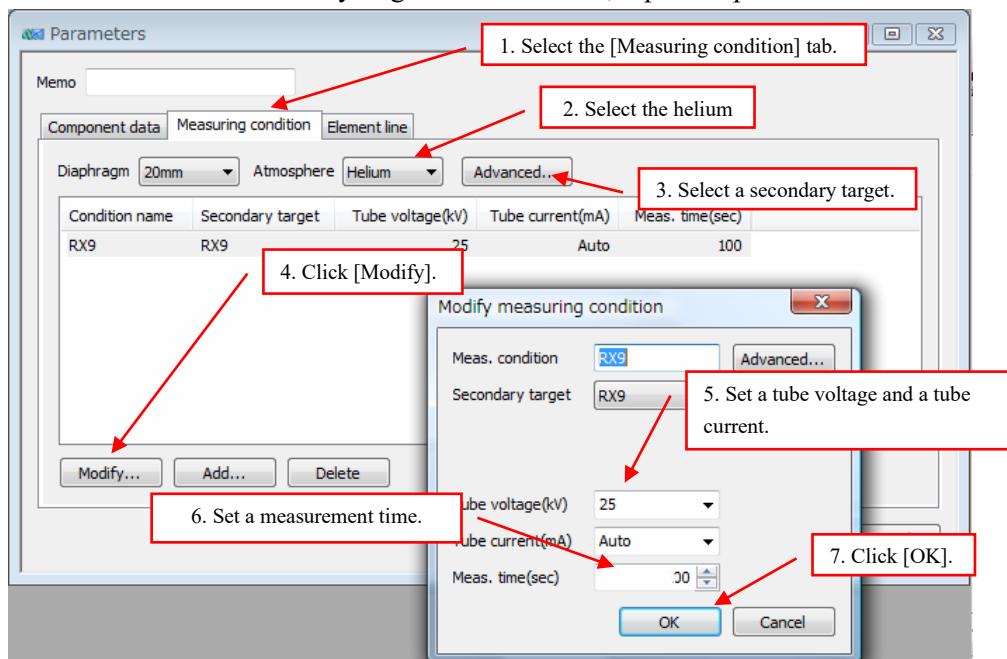
1. Select the [Component data] tab on the [Parameters] dialog.
2. Select an element to be analyzed.
3. Click [Modify].
4. The unit can be changed.
5. Set a measuring condition and an analysis line. Select the S-Ka line for the analysis line.
6. Click [OK].
7. When two or more elements are to be analyzed, repeat steps 2 to 6.
8. Click [Next].



(B) Setting Procedure for Measuring Condition

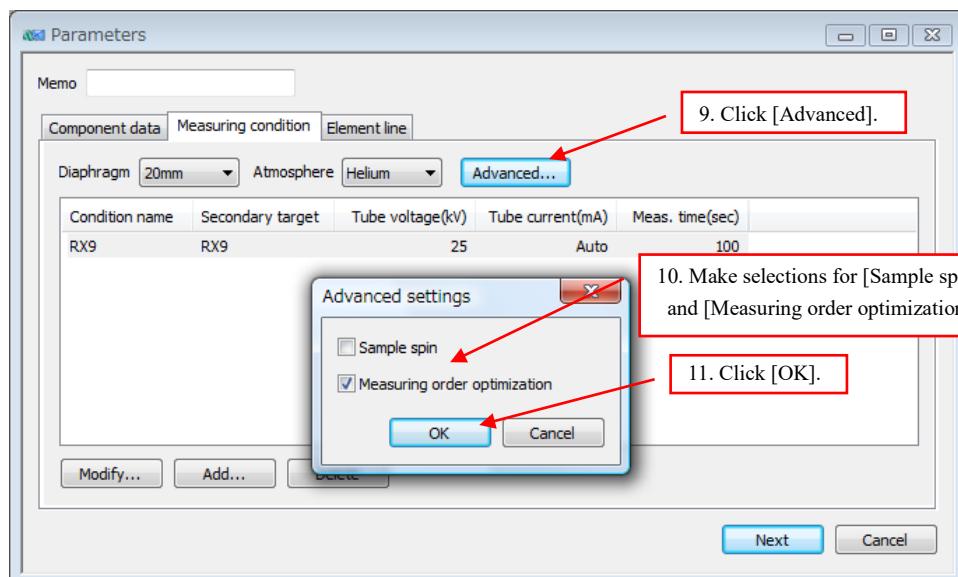
Set here a measurement diameter, atmosphere, tube voltage, tube current and measurement time.

1. Select the [Measuring condition] tab.
2. Select 20mm for the measurement diameter and the helium for the atmosphere because a liquid sample is to be analyzed.
3. Select a secondary target. (Ordinarily, secondary targets that can be used for an objective measurement line are automatically displayed.)
4. Click [Modify].
5. Set a tube voltage and a tube current on the [Parameters] dialog. When sulfur in light oil is to be analyzed using the RX9 secondary target, it is recommended that the tube voltage and current are set to 25kV and 1mA respectively.
6. Set a measurement time for the secondary target.
7. Click [OK].
8. When two or more secondary targets are to be used, repeat steps 3 to 7.



- When [Auto] is set for the tube current, a preparatory measurement is made for a few seconds before a main measurement for each target, and the current is adjusted so that the dead time falls within the predetermined range. When samples for quantitative analyses are similar and dead times are several percent or less, measurement times can be shortened by setting fixed values for the current because preparatory measurements are not made.

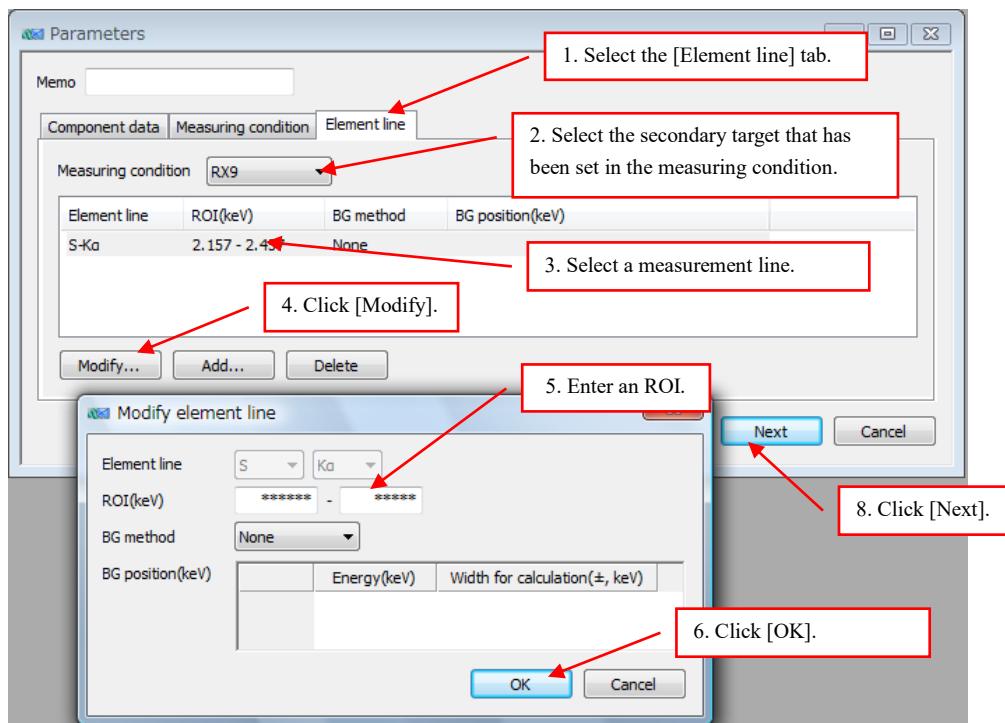
9. Click [Advanced].
10. Make selections for [Sample spin] and [Measuring order optimization]. When the spin unit has been attached, place a check mark for [Sample spin]. When a check mark is placed for [Measuring order optimization], measurements are made from the secondary target with the highest energy in order of energy (Al → Mo → Cu → RX9). Place a check mark for [Measuring order optimization] for the meantime.
11. Click [OK].



(C) Setting Procedure for Element Line

Set mainly the ROI (region of interest) of an Element line here. Detailed settings are made in “Analysis Parameter Setting”.

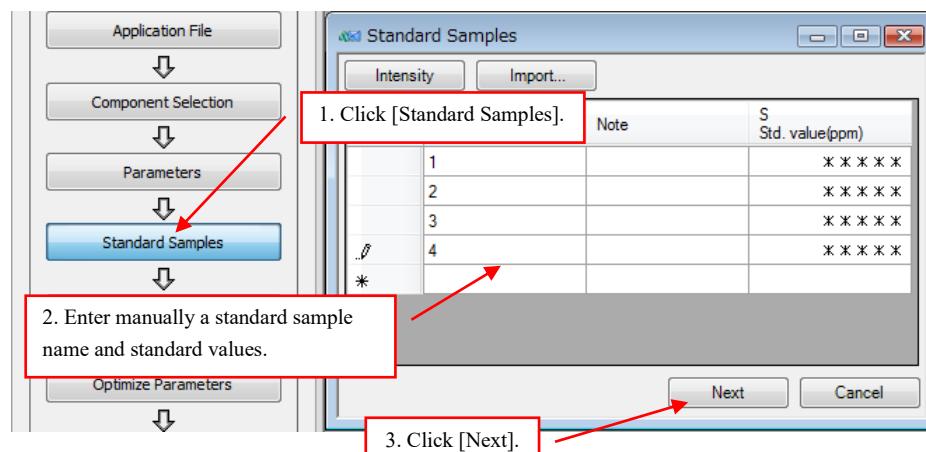
1. Select the [Element line] tab.
2. Select the secondary target that has been set in the measuring condition.
3. Select a measurement line. The ROI has been determined beforehand.
4. Click [Modify].
5. Change the ROI if necessary and set a background.
6. Click [OK].
7. When two or more measurement lines are to be used, repeat steps 2 to 5.
8. When settings on the [Component data], [Measuring condition] and [Element line] tabs have been completed, click [Next].



iv. Standard Sample Setting

Enter the standard values (chemical analysis values) of standard samples.

1. Click [Standard Samples] on the flow bar to invoke the [Standard Samples] dialog.
2. Enter a standard sample name and standard values.
3. Click [Next].

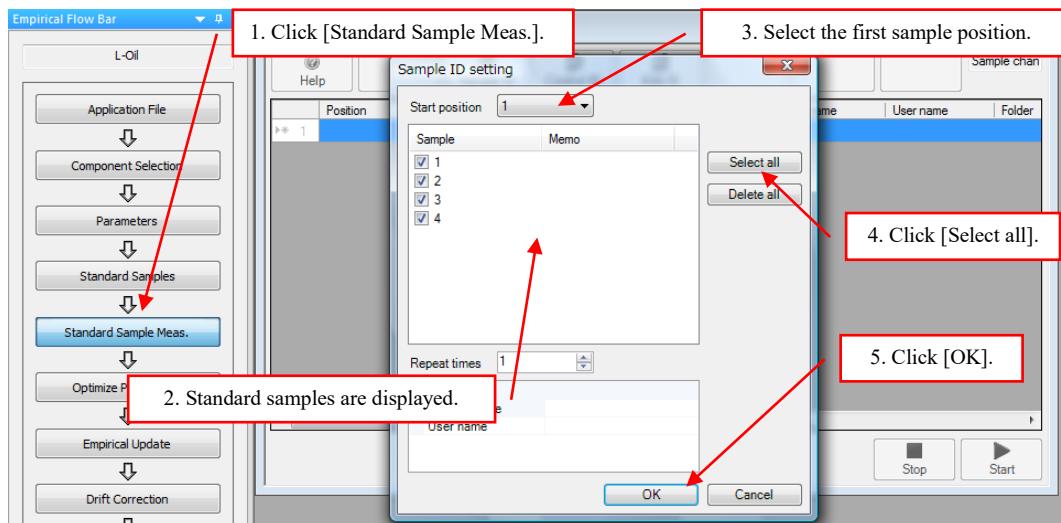


- When “Intensity” is clicked after the standard sample measurement, X-ray intensities are displayed.
- A standard sample name used for another quantitative application can be imported. If elements used are the same, standard values are automatically read in.

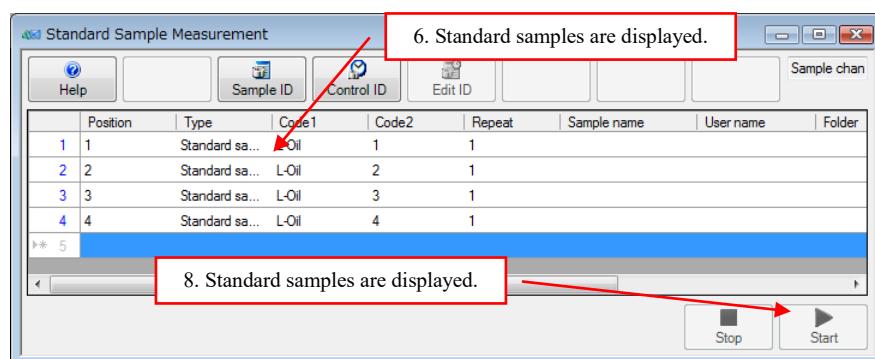
v. Standard Sample Measurement

Set standard samples in the sample chamber for measurements.

1. Click [Standard Sample Meas.] on the flow bar. The [Standard Sample Meas.] screen automatically appears.
2. Standard samples that have been input are automatically displayed on the [Sample ID] dialog.
3. Select the number of the first standard sample to be set on the sample turret.
4. Click [Select all] to place check marks for the standard samples.
5. Click [OK].
6. A list of the standard samples appears on the [Sample ID setting] dialog.



7. Set samples on the sample turret.
8. Click [Start], and measurements will start.
9. When the measurements are completed, the next sequence is displayed.





- When measurements are completed, remove the measured samples immediately from the measurement chamber. If samples that emit volatilized gas are left in the sample chamber periodically, the inside of the instrument may be contaminated. Take special care about acid solutions because they may emit acid corrosive gas and, at worst, damage the optical system of the instrument.
- If a volatile sample such as light oil is measured without boring an air hole in the lid, the pressure in the cell will increase, the analysis window will swell, analysis precision will lower and, furthermore, the analysis window may tear.

vi. Analysis Parameter Setting

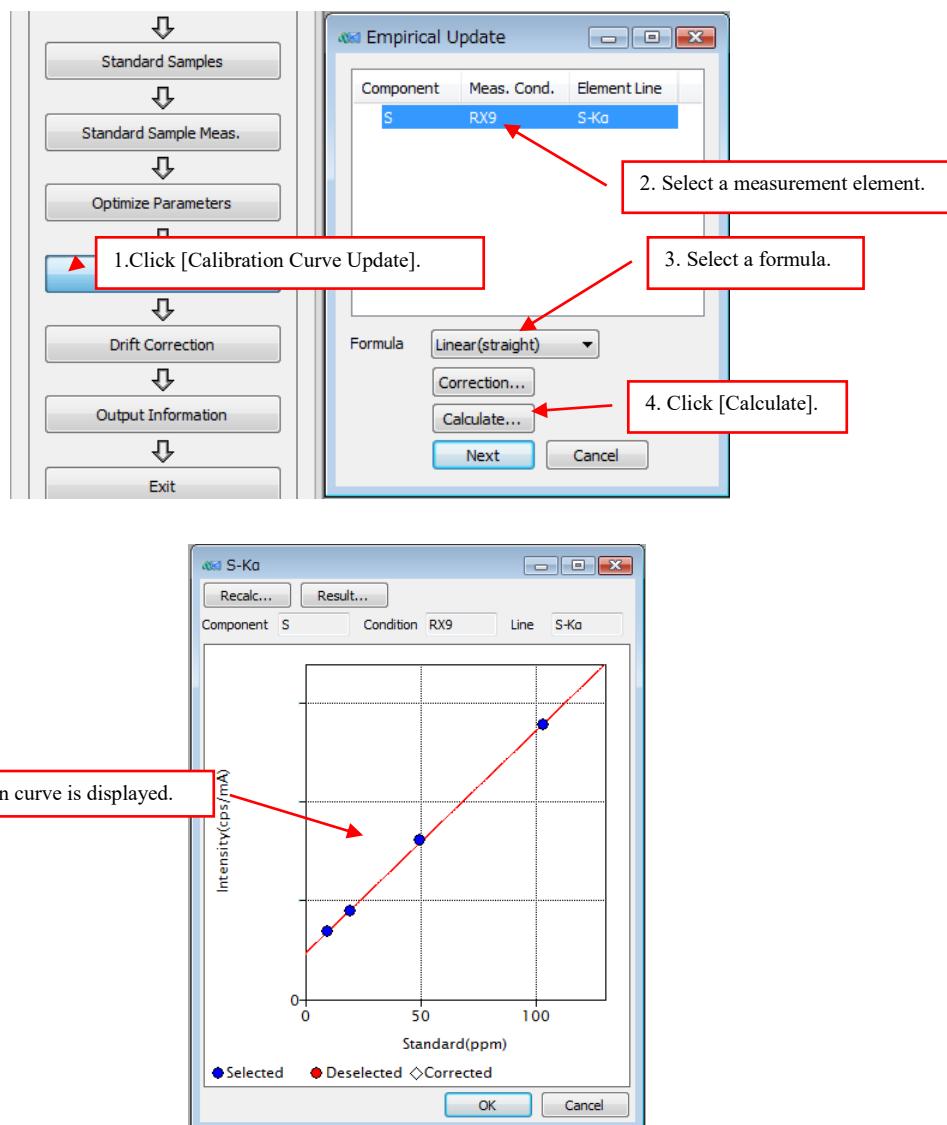
Set detailed conditions such as ROIs using the spectra of measured samples.

1. Invoke the [Optimize Parameters] screen. Select a secondary target used for the measurement for [Measuring condition].
2. From the tabs, select a sample that has the highest intensity of an objective element. An objective analysis line, its ROI, etc. are displayed.
3. To change the ROI, put the cursor on a black boundary line to make a bidirectional arrow appear. Drag the mouse, and the black line (boundary of the ROI) will move.
4. The changed ROI is automatically reflected in the analysis parameters.
5. Repeat steps 1 to 3 for all elements to be set.
6. Click [Next].

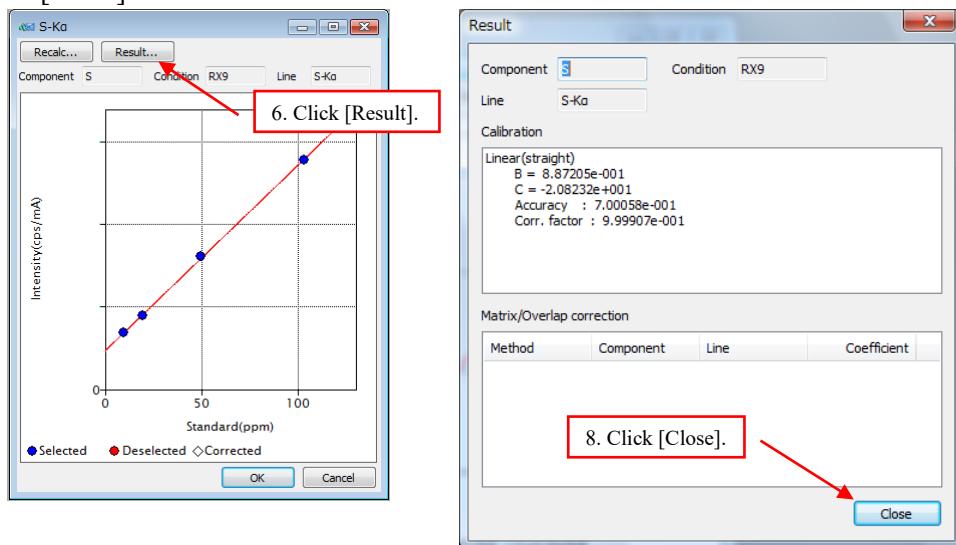


vii. Creation of Calibration Curve

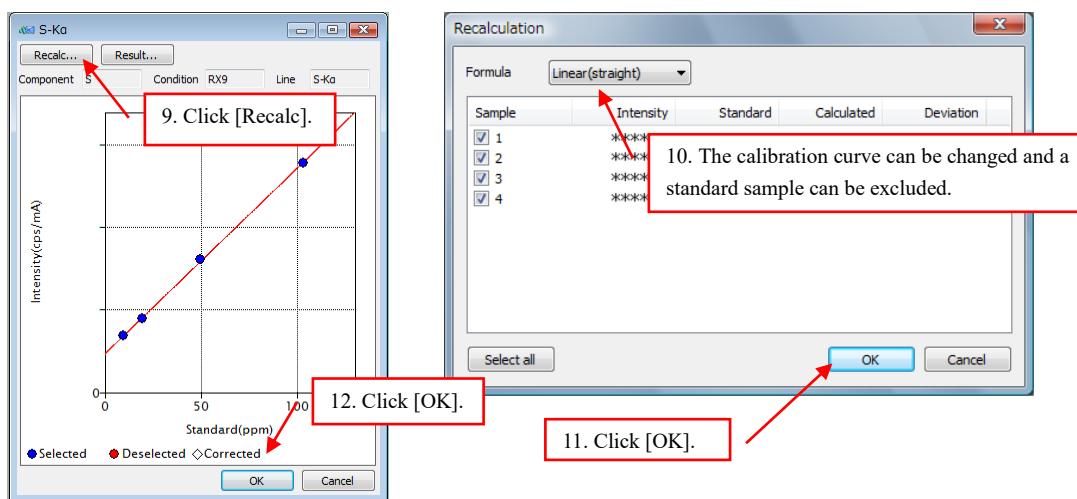
1. Click [Calibration Curve Update] on the flow bar.
2. The [Calibration Curve Update] dialog appears. Select measurement elements. Because only sulfur is used here, select S-Ka.
3. Select a formula to be used for a calibration curve. Select the linear equation here.
4. Click [Calculate].
5. A calibration curve is displayed.



6. To check the calibration curve coefficients, click [Result].
7. The [Result] dialog appears, and the calibration curve coefficients and the accuracy and correlation coefficient of the calibration curve are displayed.
8. Click [Close].

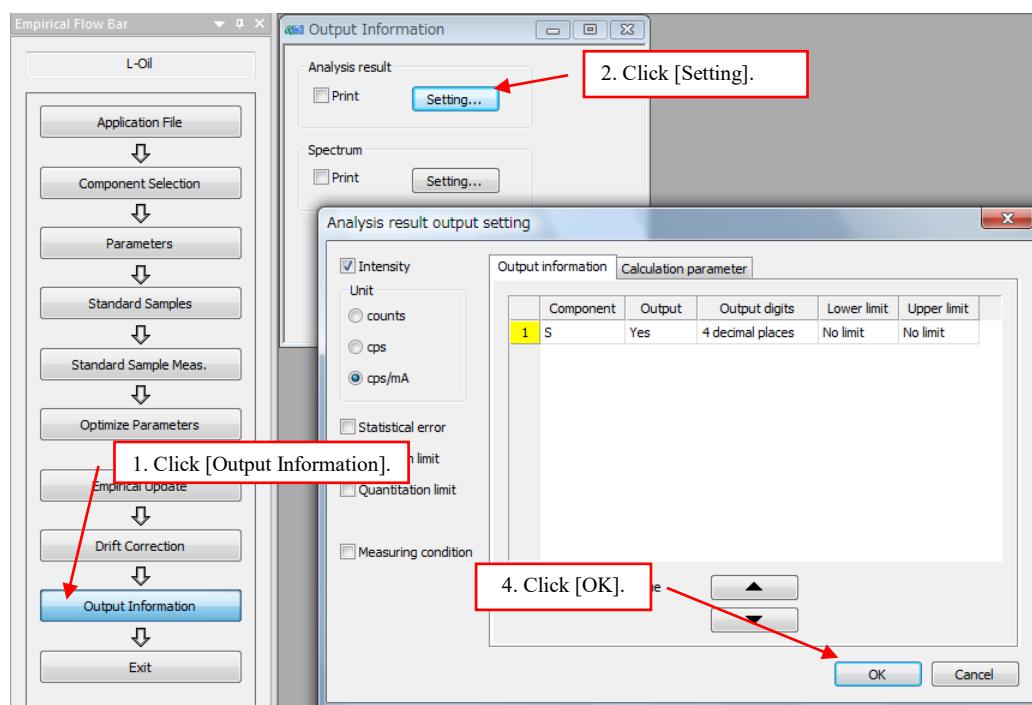


9. Click [Recalc]. The [Recalculation] dialog appears.
10. The calibration curve formula can be changed. If there is a sample far from the calibration curve, it can be excluded.
11. Click [OK].
12. Click [OK].
13. The mark appears and calibration curve coefficients are stored.
14. When there is another measurement element for which to create a calibration curve, repeat steps 2 to 13.
15. Click [Next] on the [Calibration Curve Update] dialog.



viii. Printout Setting

1. Click [Output Information] on the Empirical flow bar.
2. When you want a printout after a quantitative analysis, place a check mark for [Print]. To edit contents to be printed, click [Setting].
3. The [Analysis result output setting] dialog appears. Set parameters on it.
4. When parameters have been set, click [OK].
5. Click [Next].



- X-ray intensities and analyzed results are printed on different sheets of paper.

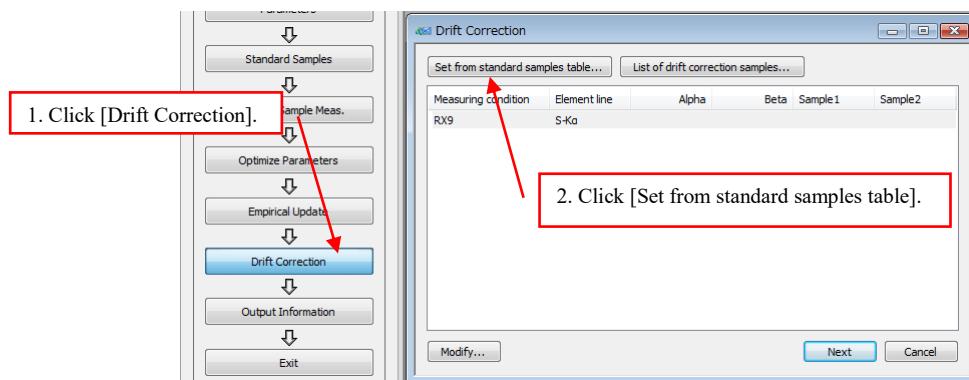
ix. Registration of Drift Correction Sample

In routine control analyses, the periodic drift correction (also called the standardization) of calibration curves are necessary to use them for a long period.

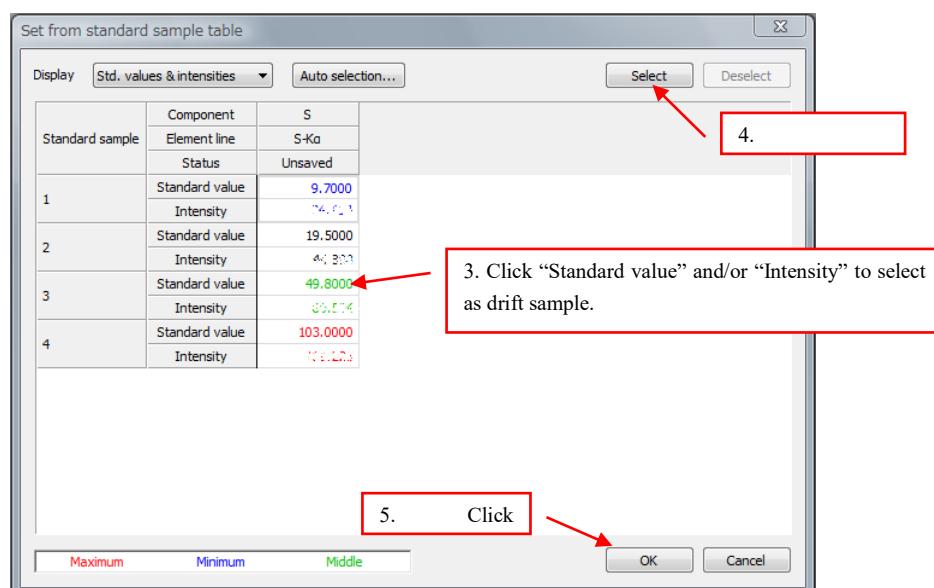
There are two methods to register the drift correction samples. One is to set from standard samples table and another is to set from the list of drift correction samples.

(A) Set from standard samples table

1. Click [Drift Correction] on the Empirical flow bar.

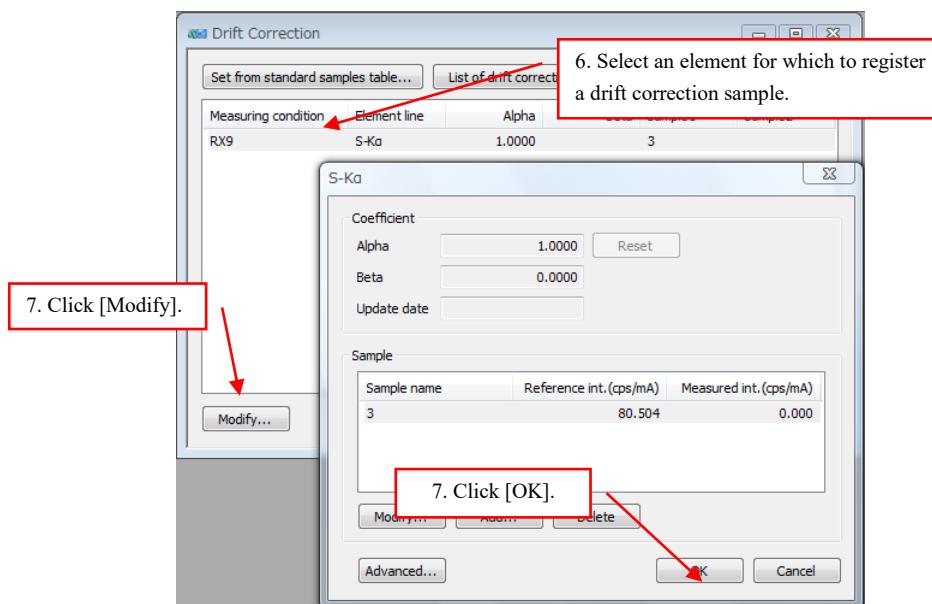


2. Click [Set from standard samples].
3. Click “Standard value” and/or “Intensity” to select as the drift samples.
4. Click [Select].
5. Click [OK].



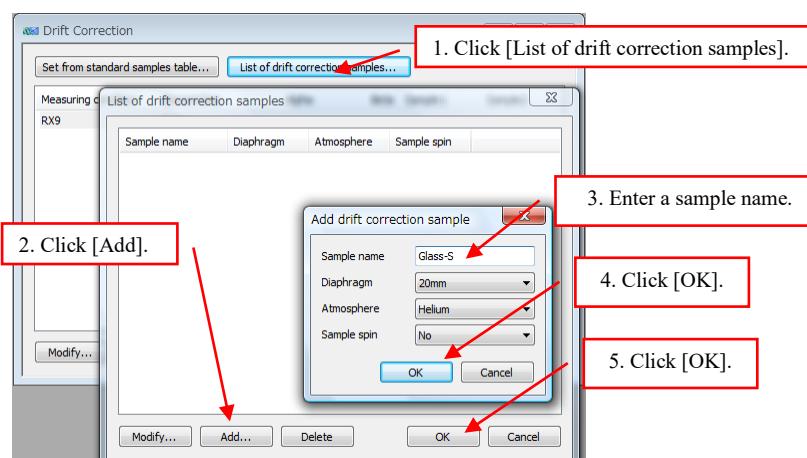
4. CREATION OF EMPIRICAL APPLICATION

6. Select an element for which to register a drift correction sample on the [Drift Correction] dialog.
7. Click [Modify]. A dialog appears to enter a drift correction sample for a measurement line. Enter here a drift correction sample name, reference intensity (of the drift correction sample measured when a calibration curve is created) and measured intensity (of the drift correction sample measured after some days or months). The drift correction is made by comparing the measured intensity to the reference intensity.
8. Click [OK] to close the dialog. (Only a check is made here.)

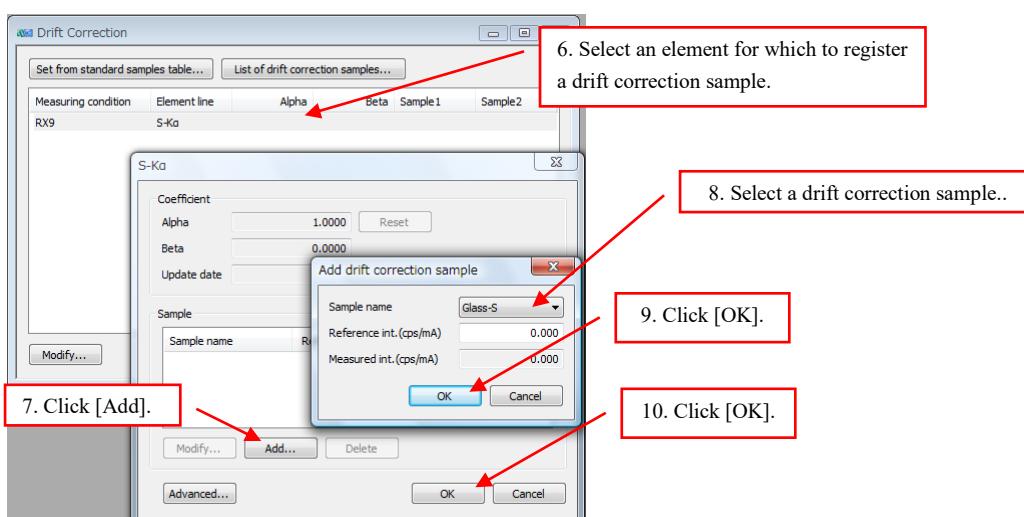


(B) List of drift correction samples

1. Click [List of drift correction samples].
2. Click [Add], and the [Add drift correction sample] dialog will appear.
3. Enter a sample name. Parameters such as the measurement atmosphere can be changed.
4. Click [OK].
5. Click [OK].



6. Select an element for which to register a drift correction sample on the [Drift Correction] dialog.
7. Click [Add]
8. Select a drift correction sample.
9. Click [OK].
10. Click [OK].

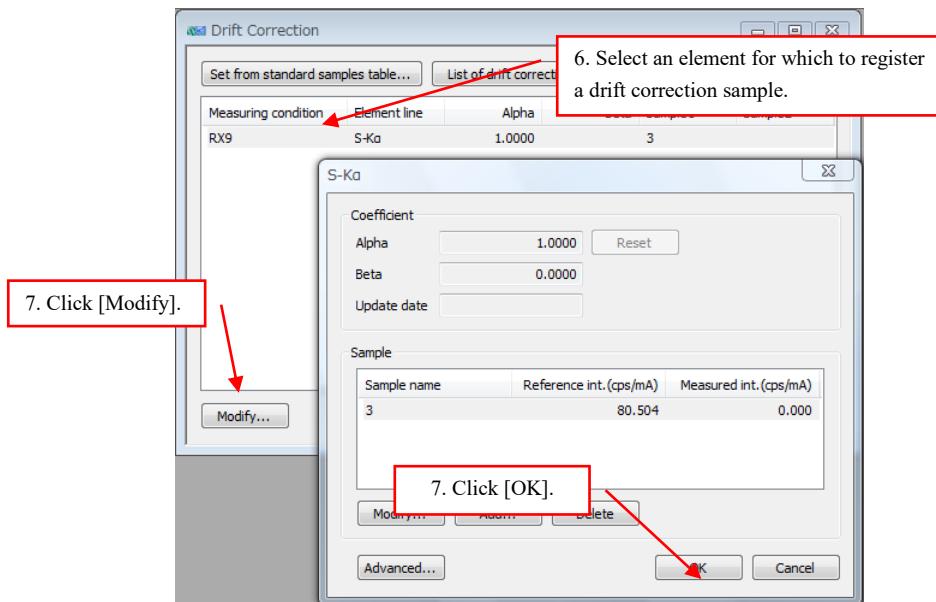


11. Select an element for which to register a drift correction sample on the [Drift Correction] dialog.
12. Click [Modify]. A dialog appears to enter a drift correction sample for a measurement line. Enter here a drift correction sample name, reference intensity (of the drift correction sample measured when a calibration curve is created) and measured intensity (of the drift

4. CREATION OF EMPIRICAL APPLICATION

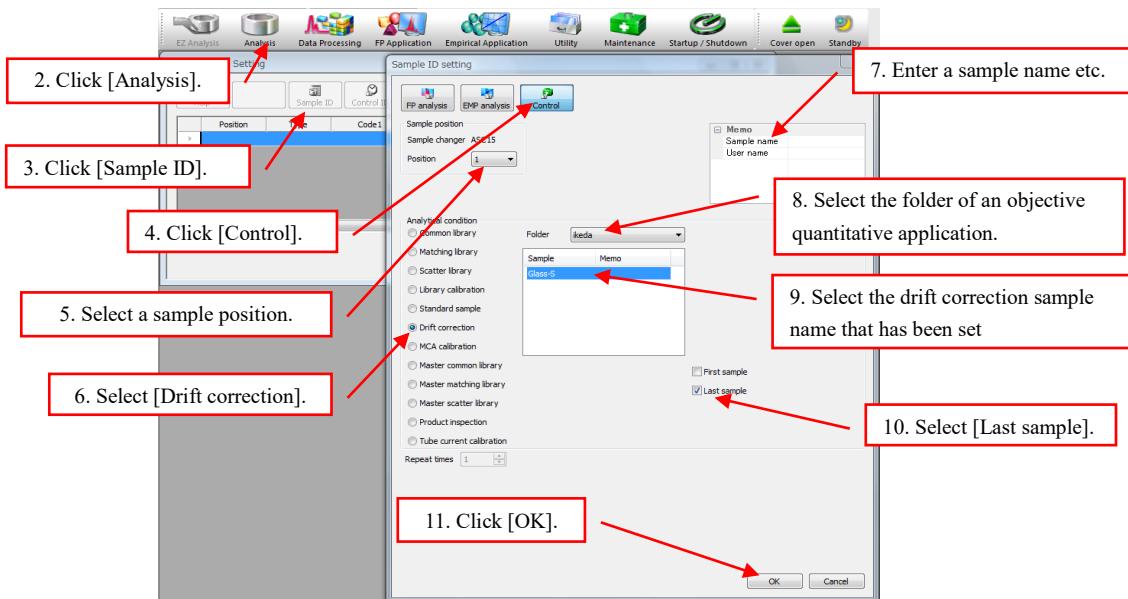
correction sample measured after some days or months). The drift correction is made by comparing the measured intensity to the reference intensity.

13. Click [OK] to close the dialog. (Only a check is made here.)

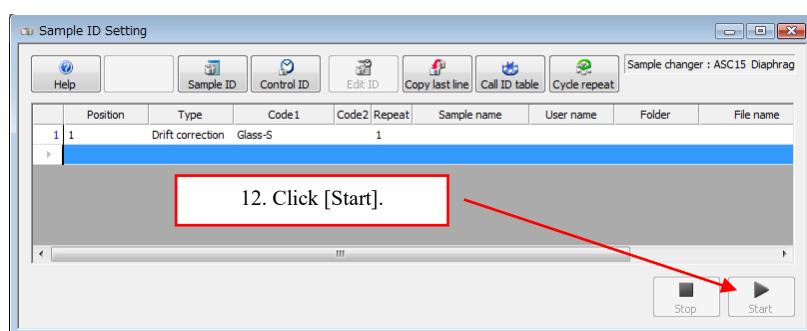


Measurement of drift correction samples

1. Open the lid of the instrument, and put the drift correction sample on the turret.
2. Click [Analysis].

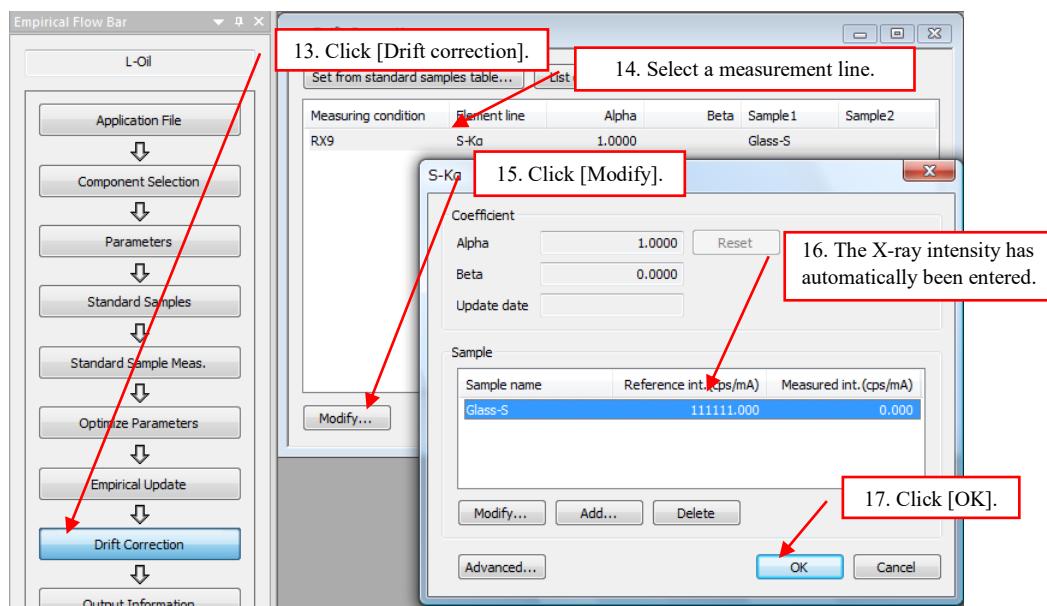


3. Click [Sample ID] on [Sample ID Setting].
4. Click [Control] on the [Sample ID Setting] dialog.
5. Select a sample position.
6. Select [Drift correction].
7. Enter a sample name (memo) etc.
8. Select a folder in which an objective quantitative application has been stored.
9. Select the drift correction sample name that has been set in the quantitative application.
10. Select the [Last sample] flag (In the case of one drift correction sample registered). When there are some drift correction samples, select the [First sample] in the first sample and select the [Last sample] in the last sample.
11. Click [OK].
12. After making sure that the drift correction sample has been set, click [Start].



4. CREATION OF EMPIRICAL APPLICATION

13. To check the X-ray intensity of the drift correction sample, click [Drift Correction].
14. Select a measurement line on the [Drift Correction] dialog.
15. Click [Modify].
16. The X-ray intensity of the drift correction sample has been automatically entered for the reference intensity and measured intensity. When the drift correction sample is measured after this, the intensity is automatically entered only for the measured intensity. (Make a check.)
17. Click [OK].
18. Close the Empirical application program.

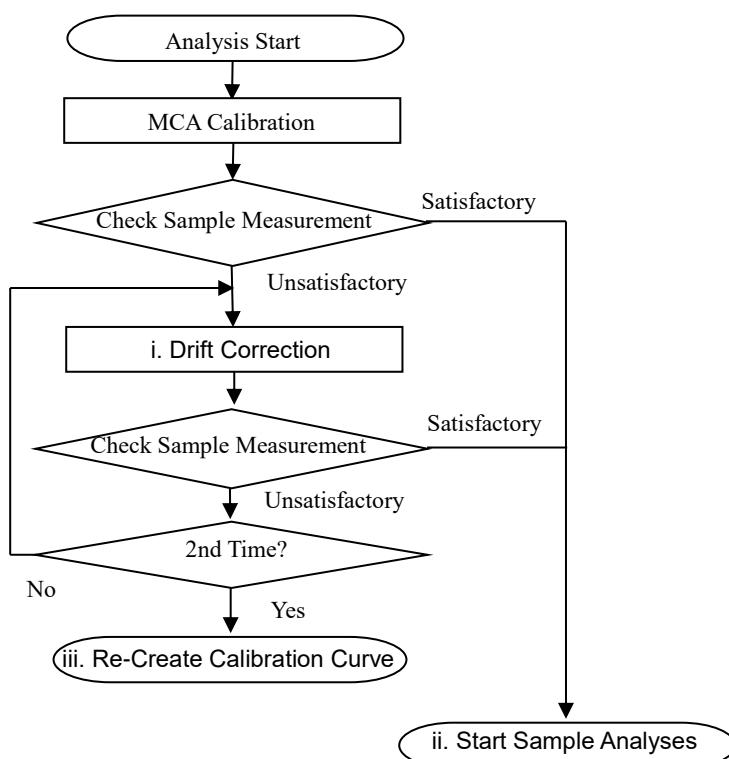


4.3 ROUTINE CONTROL ANALYSES

The sequence of routine analyses is shown below. First, before routine analyses, calibrate the energy axis of the detector using the MCA (multi-channel analyzer) calibration. Next, to make sure whether or not a quantitative application to be used is satisfactory, measure a check sample. When the result of the check sample is satisfactory, main analyses can be started.

When the value of the check analysis is not satisfactory (determine an appropriate control range), make a drift correction. Measure the check sample again after the drift correction. When the result is satisfactory, main analyses can be started.

When the value of the check sample still has a discrepancy, you can make a drift correction again, but we recommend that the calibration curve be re-created.

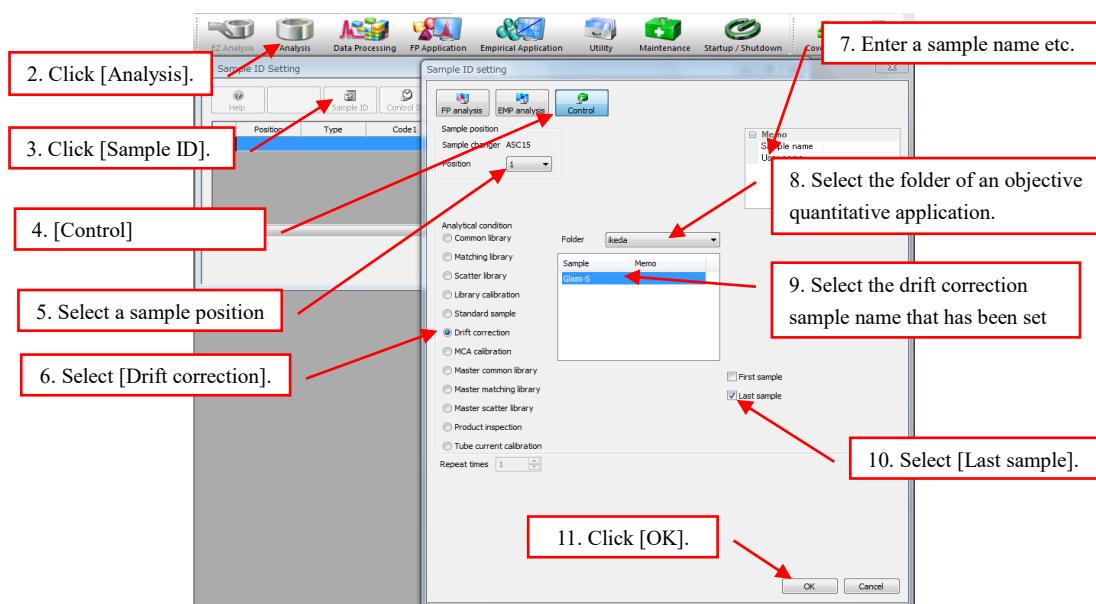


- If the analysis value of a light element in a check sample measured in the helium mode is extraordinarily small, the sample protection film in the sample chamber may be damaged. When the protection film is replaced with a new one, the normal analysis value may be restored.

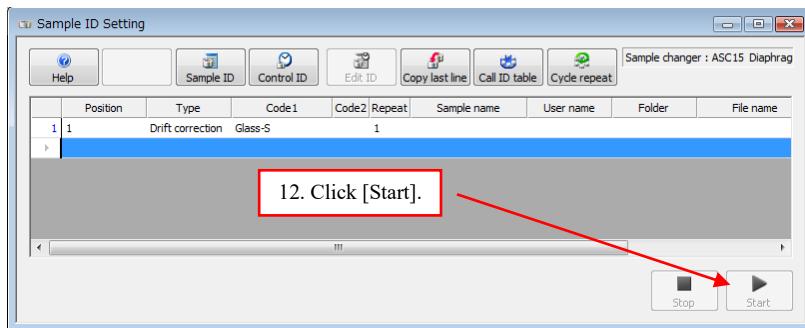
i. Drift Correction

The procedure is basically the same as that in “4.2 ix. Registration of Drift Correction Sample”.

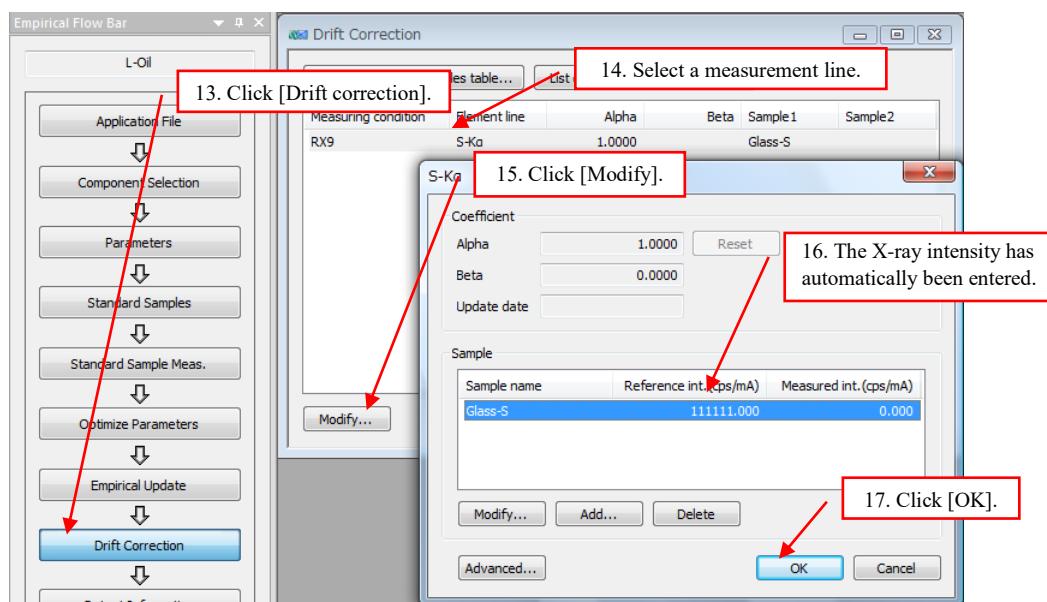
1. Open the lid of the instrument, and put the drift correction sample on the turret.
2. Click [Analysis].
3. Click [Sample ID] on [Sample ID Setting].
4. Click [Control] on the [Sample ID Setting] dialog.
5. Select a sample position.
6. Select [Drift correction].
7. Enter a sample name (memo) etc.
8. Select a folder in which an objective quantitative application has been stored.
9. Select the drift correction sample name that has been set in the quantitative application.
10. Select the [Last sample] flag (In the case of one drift correction sample registered). When there are some drift correction samples, select the [First sample] in the first sample and select the [Last sample] in the last sample.
11. Click [OK].



12. After making sure that the drift correction sample has been set, click [Start].



13. To check the X-ray intensity of the drift correction sample, click [Empirical Application] on the tool bar and click [Drift correction] on the flow bar.
 14. Select a measurement line on the [Drift correction] dialog.
 15. Click [Modify].
 16. Make sure that the X-ray intensity of the newly measured drift correction sample has automatically been entered for the measured intensity.
 17. Click [OK].



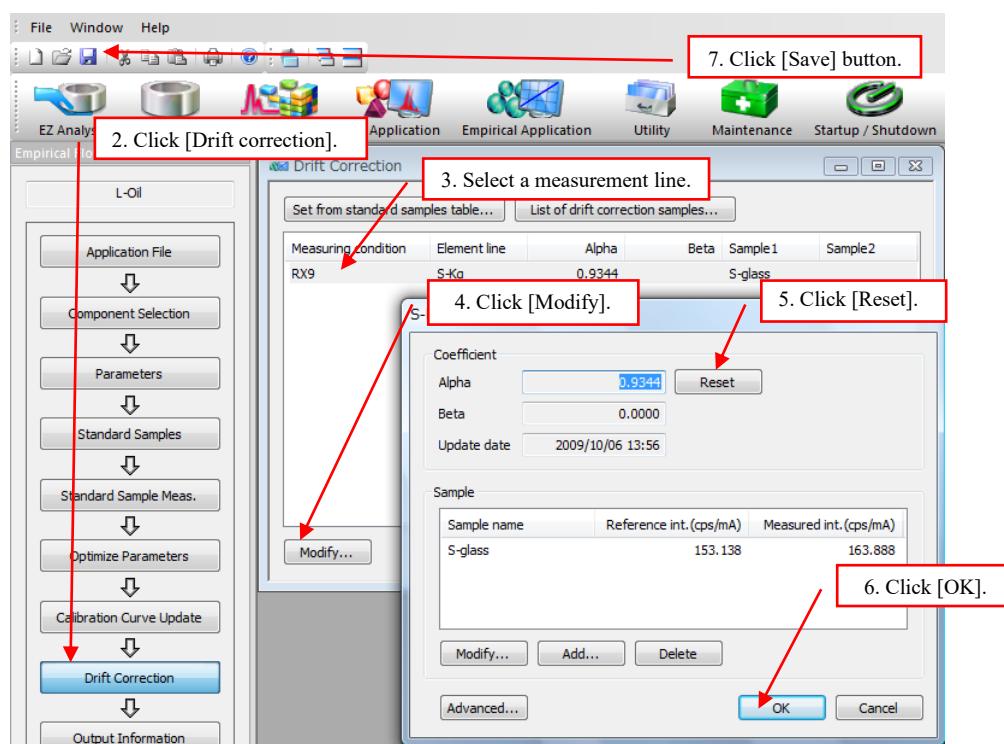
ii. Measurement

For the procedure for a measurement, see “1. ANALYSIS OPERATION”.

iii. Re-Create Calibration Curve

When the calibration curve is re-created, reset the values of $\alpha\beta$ before measuring the standard samples.

1. Click [Empirical Application] on the tool bar.
2. Click [Drift correction] on the flow bar.
3. Select a measurement line on the [Drift correction] dialog.
4. Click [Modify].
5. Click [Reset]
6. Click [OK].
7. Click [Save] button.



5. SAMPLE PREPARATION

5.1 OUTLINE

Described in this chapter are examples of methods for setting liquid and powder samples.

5.2 LIQUID SAMPLE

1. Described below is an example of a method using the sample cell Cat. No.1330 (with a lid) manufactured by Chempex. This cell consists of three components.
2. Although there should not be contaminations on a new sample cell, they may contaminate a sample to be measured in rare cases. So wipe away dirt completely using wiping paper (or similar) lightly soaked with ethanol.
3. Apply film on the sample cell. Ordinarily, use the supplied 4 μm Prolene film (Cat. No.416) manufactured by Chempex. For some kinds of solutions, use Mylar films etc. When applying the film, eliminate wrinkles as much as possible.



Sample Cell Manufactured by Chempex
Cat. No.1330 (With Lid)
This consists of 3 components. Other suppliers can be used.



The 4 μm Prolene film (Cat. No.416) manufactured by Chempex has been applied on a sample cell as an analysis window.



- The resistance of films to chemicals is shown on the next page.

5. SAMPLE PREPARATION

- Ordinary Resistance of Films to Chemicals and X-Rays

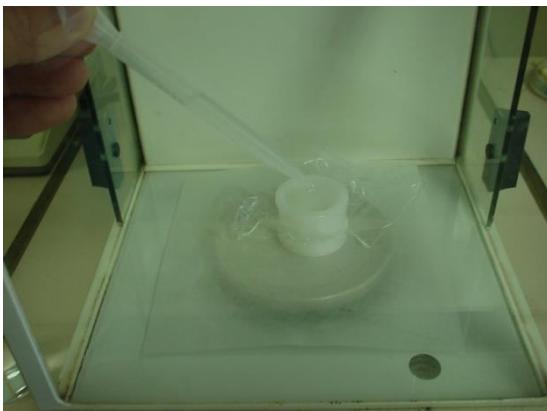
Chemical Classification	Mylar	Polypropylene	Polyimide (Kapton)	Prolene	Ultra-Polyester
Acids, dilute or weak	G	E	F	G	G
Acids, concentrated	N	F	F	E	N
Alcohols, aliphatic	E	E	G	E	E
Aldehydes	U	E	E	E	U
Alkalies, concentrated	N	E	N	E	N
Esters	N	G	G	G	N
Ethers	F	N	U	N	F
Hydrocarbons, aliphatic	G	G	G	G	G
Hydrocarbons, aromatic	F	F	F	F	F
Hydrocarbons, halogenated	F	N	F	N	F
Ketones	E	G	G	G	E
Oxidizing agents	F	F	N	F	F
X-Ray Resistance	G	G	E	G(N*)	N (F)

E: Excellent, G: Good, F: Fair, N: Not Recommended, U: Unknown



- * It is recommended that Prolene not be used for liquid analyses using a large X-ray fluorescence spectrometer.
- Since chemical resistance varies a little depending on the literature, be sure to conduct a test using a small quantity of solution before a measurement.

4. Pour sample into the sample cell using a transfer pipette, syringe, or similar device. The sample quantity can be read accurately by pouring it on a scale. The recommended quantity is 4g (4mL).
5. Lay down powder paper so that the surface of the analysis window is not contaminated, put the weighed cell on it, and put the lid on.



Pour sample into the cell using a transfer pipette or similar device.



Lay down powder paper, put the weighed cell on it, and put the lid on.



- Especially when analyzing volatile sample such as naphtha, measure it quickly and remove it from the sample chamber immediately after the measurement. This caution applies also to ordinary samples.
- When the phase separation occurs with a sample, it is recommended that the sample be measured quickly after stirring it sufficiently or that the upper and lower solutions be measured separately after the phase separation and an average be calculated using the weight ratio.

5.3 POWDER SAMPLE (LOOSE POWDER METHOD)

1. Weigh the sample on powder paper. To obtain a better result, crush the sample into 200-mesh or finer powder if possible.
2. Put the sample in a sample cell. Assemble the sample cell as in the case of liquid.
3. As in the case of solution, lay down powder paper on a desk to avoid contamination, put the weighed cell on it, and put the lid on.



Weigh the sample on powder paper.



Put the sample in a sample cell.

5.4 PELLET SAMPLE

i. Aluminum Ring Method



Shown on the left is a 35mm dia. powder sample aluminum ring No.3481M1 for the 10-sample turret. Shown on the right is a 28mm dia. aluminum ring No.3481H1 for the 15-sample turret.



Die for Aluminum Ring Method (Rigaku AL-40)



Put sample.



Press the sample.
The pressure is approx. 10 to 20 tons.



The sample preparation has been completed.
The pressure is approx. 10 to 20 tons.

ii. Die Method

Merit of Die Method:

If the aluminum ring method is used for bulky powder sample such as CaCO₃, a ring will be crushed and sample cannot be formed properly. In this case, we recommend the die method. Of course, ordinary powder can also be formed securely.

5. SAMPLE PREPARATION



An example of a die manufactured by Chemplex is shown above. One for the 15-sample turret is Cat. No.4030, and one for the 10-sample turret is Cat. No.4040. Rigaku Service's dies of various sizes are also available.



An aluminum cup is shown on the right part (one for the 15-sample turret is Cat. No.4505, and one for the 10-sample turret is Cat. No.545). A plastic cup is shown on the left part (one for the 15-sample turret is Cat. No.552, and one for the 10-sample turret is Cat. No.554). An example of a die manufactured by Chemplex is shown. A formed sample is shown in the lower part.



- In the case that a sample is difficult to form, use binder.



Rigaku recommends SpectroBlend manufactured by Chemplex as binder. (Cat. No.660: Powder, Cat. No.675: 1/4g tablets, Cat. No.690: 1/2g tablets)
Mix 10 to 20% of sample.
When mixing, use an agate mortar or a crusher.

6. CALIBRATION AND MAINTENANCE

6.1 OUTLINE

To keep the spectrometer in good condition, daily maintenance is necessary. So be sure to carry out the following maintenance activities:

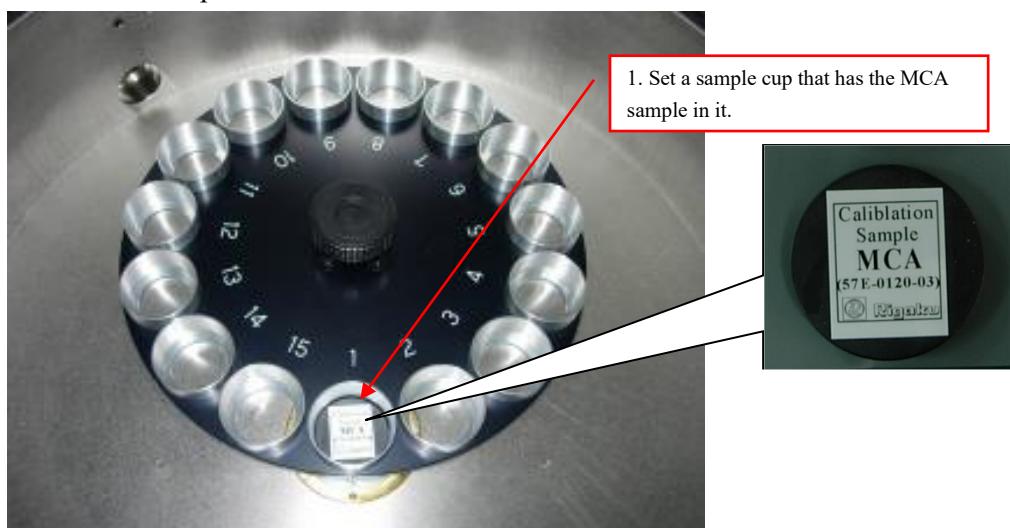
- MCA Calibration: Once a day
- Library Calibration: Once a month
- Replacement of Diaphragm Unit Protection Films: if the films are torn, dirty or slack.
- Cleaning of Sample Chamber: Determine the period of cleaning according to the operation condition.

6.2 MCA CALIBRATION

The MCA calibration is a function to calibrate the relationship between the channels of spectrum data and the energy of fluorescent X-rays using the MCA calibration sample (hereinafter referred to as the MCA sample) that Rigaku supplies for each instrument. If this calibration is not carried out for a long period, the peak positions of fluorescent X-rays will shift from right positions on a spectrum chart and erroneous identification and analysis values will result.

Be sure to carry out the MCA calibration once a day.

1. Set a sample cup that has the MCA sample in it on the sample changer.
2. Close the sample chamber cover.

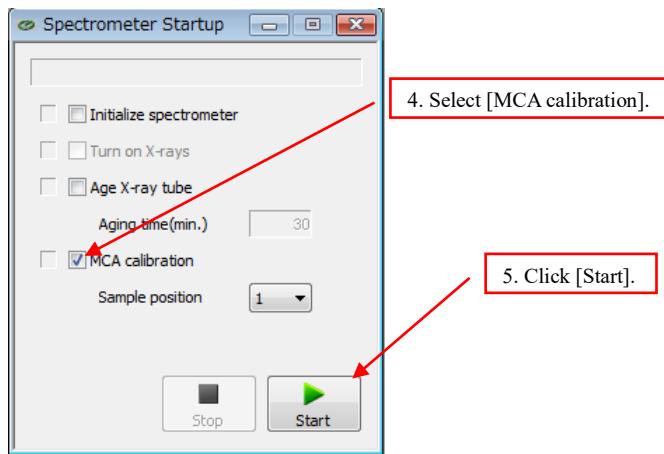


6. CALIBRATION AND MAINTENANCE

3. Select [Startup / Shutdown] → [Startup] on the tool bar.



4. Select [MCA calibration].
5. Make sure that the MCA sample has been put in the predetermined sample position, and click [Start].



- Helium purge can be used for all matrix types: solid, powder, pellet and liquids. Vacuum cannot be used with powder or liquid samples, and is only used for solid or pellet samples.
- Analyzers equipped with helium purge and no vacuum will measure the MCA Calibration and Library Calibration using helium purge.
- Analyzers equipped with both helium and vacuum will measure MCA Calibration and Library Calibration in vacuum.

6.3 LIBRARY CALIBRATION

The library calibration is a function to carry out automatically the peak profile calibration of the spectrometer and the sensitivity library calibration through the intensity drift calibration.

In the energy-dispersive X-ray fluorescence spectrometer, a fluorescent X-ray spectrum from a sample has a peak profile that consists not only of a simple Gauss function but also, mainly resulting from the detector, of a shelf, which appears in the position of energy lower than that of fluorescent X-rays, an escape peak, which appears in the position of the energy of fluorescent X-rays minus the energy of the fluorescent X-rays of an element constituting the detector, a sum peak, which appears in the position of twice the energy of fluorescent X-rays, and other components.

In the library calibration, the peak profiles of elements are obtained using the Sn, Cu and SiO₂ samples supplied with the spectrometer, and the peak profiles are calibrated. The fluorescent X-ray intensities of elements contained in the MCA sample are also obtained to carry out the drift calibration of sensitivity library measurement intensities.

If this calibration is not carried out for a long period, library data will not correlate with the data of samples because of variations in peak profiles and X-ray intensities, and erroneous identifications and the shifts of analysis values will result.

Be sure to carry out the library calibration once a month.

List of Library Calibration Samples

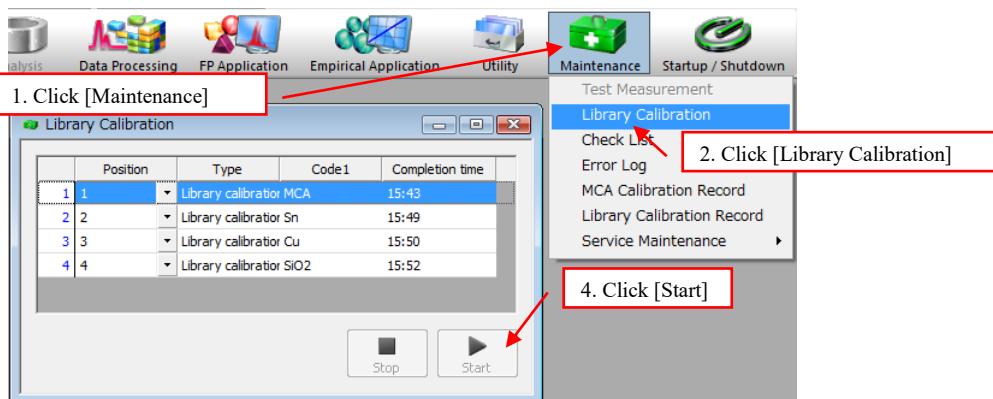
	Sample Name
1	MCA sample
2	Sn
3	Cu
4	SiO ₂



- Be sure to use the MCA sample supplied with the spectrometer. If a sample supplied with another spectrometer is used, calibration cannot be carried out correctly.
- By reason of the calibration process, the MCA sample must be measured first.

6. CALIBRATION AND MAINTENANCE

1. Click [Maintenance] on the tool bar.
2. Click [Library Calibration] on the maintenance list.
3. Make sure that the MCA, Sn, Cu, and SiO₂ samples have been set in the first, second, third and fourth positions of the sample turret respectively, and close the sample chamber cover.
4. Click [Start].
5. When the measurements are completed (after approximately 30 minutes), the library is automatically calibrated.



- If the sample changer has not been chosen in the [Equipment Setting] menu, the library calibration cannot be started.
- If the diaphragm of 20mm has not been chosen in the [Equipment Setting] menu, the library calibration cannot be started.



- Calibration results can be displayed using "Library Calibration Record".



- Helium purge can be used for all matrix types: solid, powder, pellet and liquids. Vacuum cannot be used with powder or liquid samples, and is only used for solid or pellet samples.
- Analyzers equipped with helium purge and no vacuum will measure the MCA Calibration and Library Calibration using helium purge.

Analyzers equipped with both helium and vacuum will measure MCA Calibration and Library Calibration in vacuum.

6.4 CHECKING AND REPLACEMENT OF DIAPHRAGM UNIT PROTECTION FILMS

Prolene protection film and Mylar protection film have been attached to the incident and outgoing sides of the diaphragm unit respectively. If a protection film of the diaphragm unit is torn, dirty or slack, analysis values will be affected. Especially when the helium flush mechanism (option) is used, the flush rise time is affected greatly during an analysis.

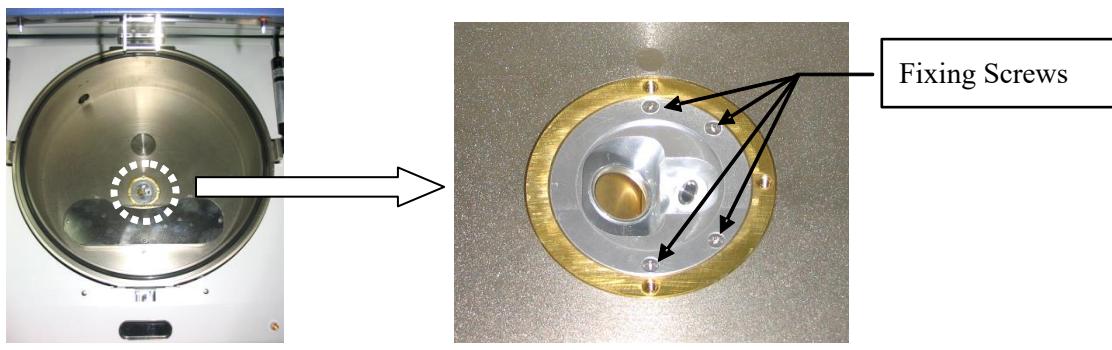
Very thin beryllium windows are used for the tips of the X-ray tube and detector. If a powder or liquid sample is damaged for some reason and contaminates the sample chamber, the beryllium windows may be damaged. Furthermore, if damaged the sample spills and enters the block unit, impurity lines may be generated during an analysis and may make a normal analysis impossible. We recommend replacement of the protection films when the films are torn, dirty or have slack.

i. Checking Procedure for Protection Films

1. Open the sample chamber cover.
2. Remove the four fixing screws in the center of the sample chamber, and remove the diaphragm unit.
3. Make sure whether or not the two protection films are torn, dirty or have slack. If any abnormality is found, that film must be replaced.



- Take care not to drop a screw or the like in the block unit. If any foreign matter is dropped, a serious spectrometer failure may occur.
- An abnormality of a protection film causes abnormal analysis values and a failure.



Diaphragm Unit

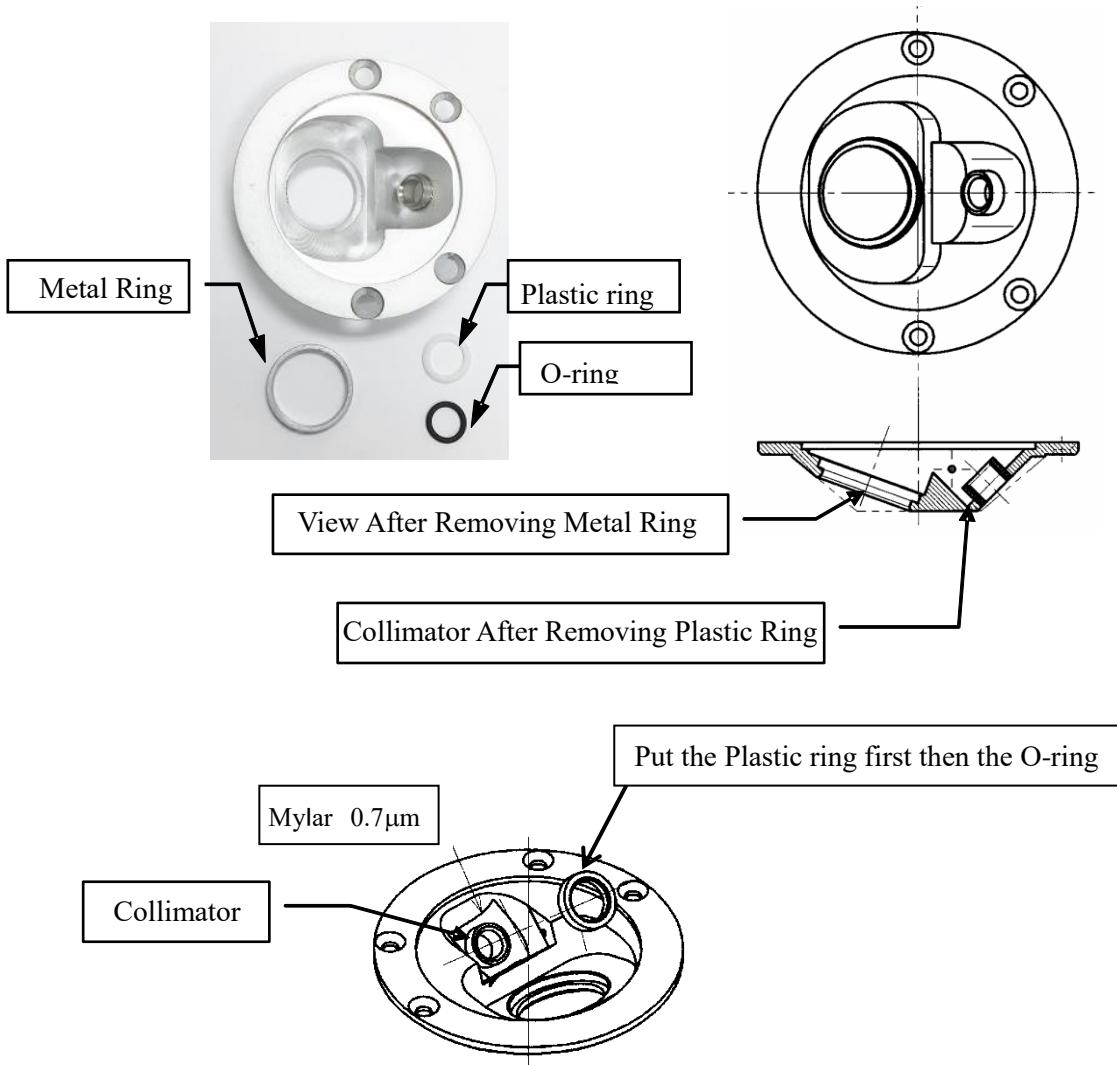
ii. Replacement Procedure for Protection Films

There are two kinds of protection film. One is Mylar film of $0.7\mu\text{m}$ thickness, which is used on the detector side (with the smaller diameter). Another is Prolene film of CH416, which is used on the secondary target side (with the larger diameter).



If the films are set in the wrong way, it causes abnormal analysis value and failure.

1. Open the sample chamber cover, remove the fixing screws and take out the diaphragm unit. Remove the Plastic Ring (on the detector side) to fix the protection film and the Metal ring (on the secondary target side) of the diaphragm unit, and remove the old films.



(A) Replacement method of the protection film on the detector side



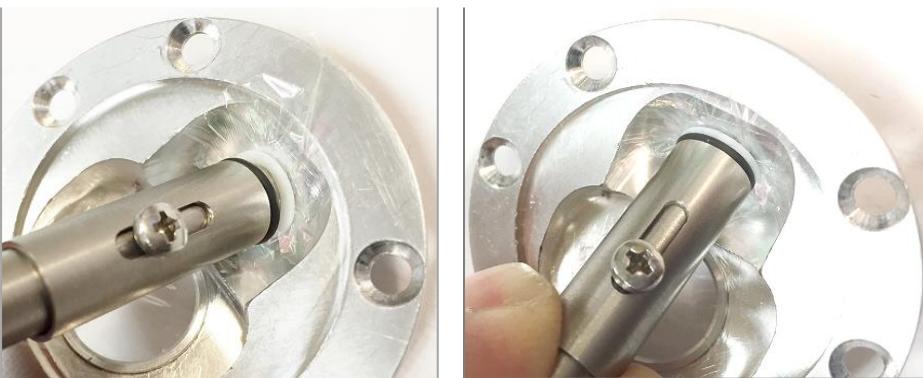
1. Place the 0.7μm Mylar window film (Part # 515635) over the collimator opening.



2. Place the white retainer ring (Part #516742) on top of the film seating it against the collimator.



3. Position the Viton O-ring (Part # 515624) over the hollow end of the installation tool (Part # 516022) as shown in the adjacent photo. Place approximately one millimeter from the end of the tool.



4. Position the installation tool with O-ring over the collimator as shown. Push the installation tools outer sleeve all the way down to attach to O-ring to the collimator.

6. CALIBRATION AND MAINTENANCE

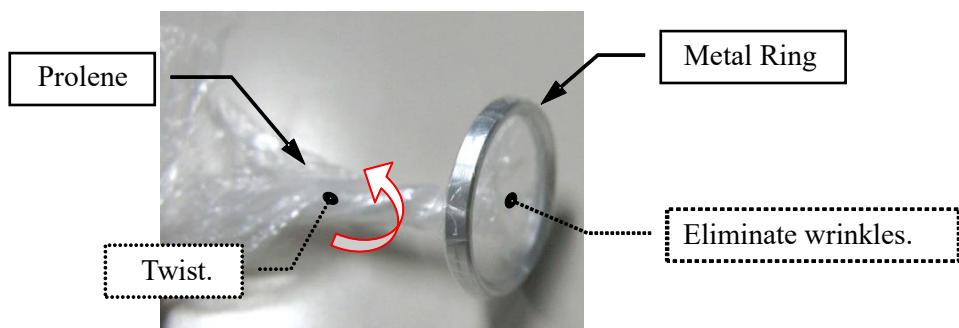
5. With the O-ring securely in place, carefully trim the excess film from the collimator area and confirm no holes or tears are present in the film.



- If a component in the X-ray path or the film is touched with a bare hand, abnormal analysis values may result.
- If part of the film enters the X-ray path, cut the part away using a cutter or the like.

(B) Replacement method of the protection film on the secondary target side

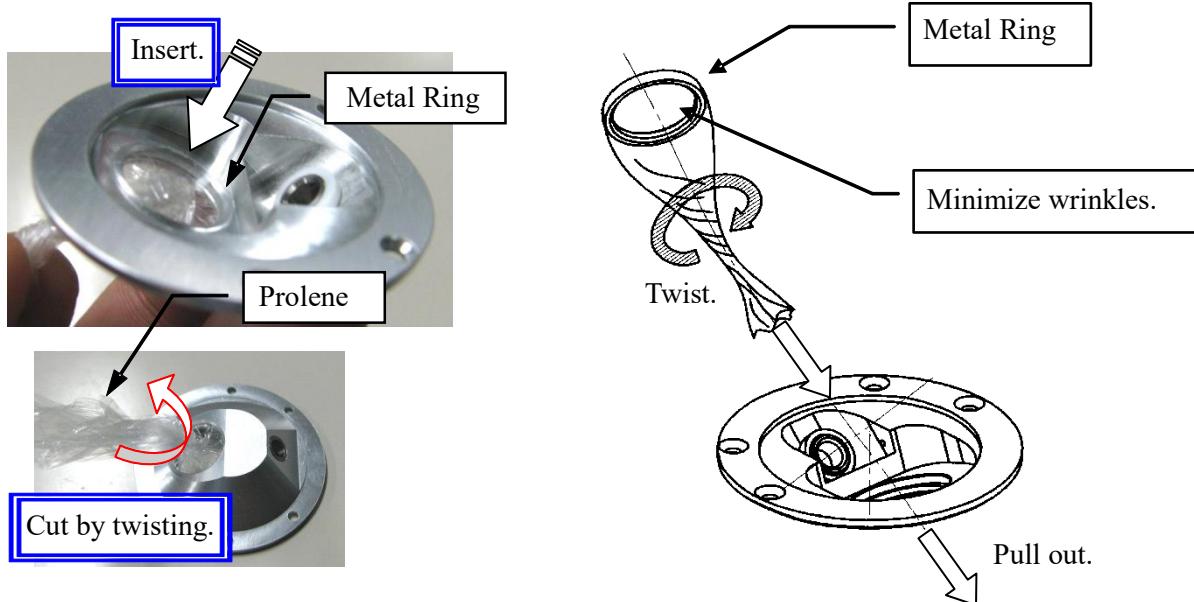
1. Cut a protection film on the secondary target side from the supplied Prolene Ch416. Wrap the fixing ring in the film that has been cut such that the length becomes approximately 60mm, and twist the end of the Prolene film to eliminate the wrinkles in the film.



2. With the end of the film twisted, insert it from the top of the diaphragm unit, and fasten the fixing ring to the unit, taking care not to produce wrinkles.
3. With the fixing ring fastened, cut away the unnecessary part of the protection film by twisting its end.



Make certain of the Metal Ring inserted in the diaphragm unit deeply. If the ring is not inserted deeply, push the edge of the ring to fix it without touching the center of film.



- Fix the diaphragm unit to the block unit as before using the four fixing screws.



- Take care not to drop a screw in the diaphragm unit of the spectrometer. Because the inside of the spectrometer may be damaged if the spectrometer is operated with a screw dropped inside, be sure to contact our service department.
- Because an unnecessary Prolene film obstructing the X-ray path decreases X-ray intensities, cut it away using a cutter or the like after the replacement.

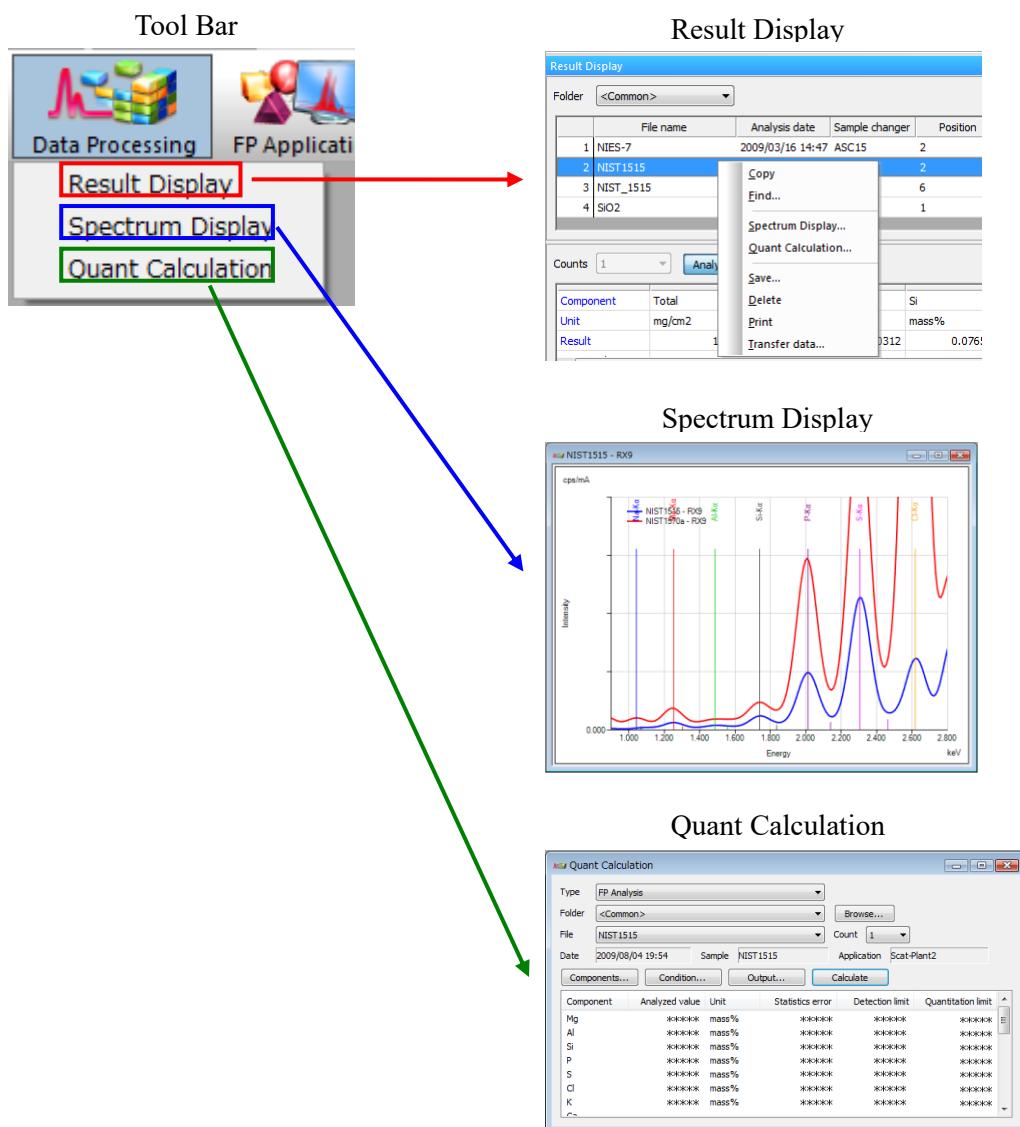


- Always keep the inside of the sample chamber clean. If part of liquid or powder sample sticks to the inside of the sample chamber, gas will be produced from it and cause abnormal analysis values and a failure.
- If part of the sample has stuck, if possible use a vacuum cleaner and gently vacuum, or the absorb it and wipe it away using water or alcohol. (Do not use a solvent such as thinner or acetone.)

7. DATA PROCESSING

7.1 OUTLINE

Analyzed results such as qualitative analysis spectra and quantitative analysis results obtained from measurements can be displayed and edited. By using [Spectrum Display], data can be printed after changing the scale and overlapped spectra can be displayed. By using [Quant Calculation], a recalculation can be made after changing the sample constitution and calculation condition. Described below is the procedure for each process that is used frequently. For the items to be described, see the table of contents.

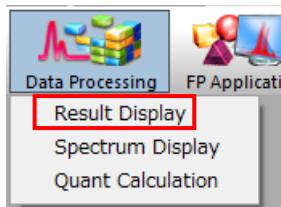


7.2 RESULT DISPLAY

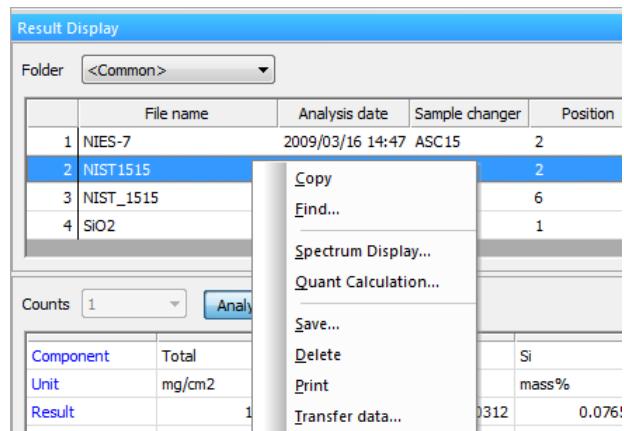
Analyzed results are displayed on the result display screen for each folder saved. Each process can be carried out by selecting a file to be displayed or edited and selecting an item on the right-click menu.

[Result Display] can be displayed from the [Data Processing] tool bar.

Select from the tool bar.



[Result Display] Screen



- Right-Click Menu

Item	Description
Copy	Copied contents can be pasted as a sample ID on the [Sample ID Setting] dialog. Or a quantitative analysis result can be pasted to spreadsheet software.
Find	A selected folder is searched for a file.
Spectrum Display	A spectrum is edited or printed.
Quant Calculation	A calculation condition is changed and a recalculation is made.
Save	A selected file (.ho) is saved in another file.
Delete	A selected file is deleted.
Print	The quantitative analysis result of a selected file is printed.
Transfer data	A selected quantitative analysis result is transferred in the spreadsheet or transmission format.

- To select two or more files, any of the following three procedures can be used:

To select successive files:

(1) Left-click a start file and expand the selected range by pressing the [\downarrow] or [\uparrow] key with the [Shift] key pressed down.

(2) Left-click a start file and left-click an end file with the [Shift] key pressed down.

To select arbitrary files:

❖ Left-click objective files with the [Ctrl] key pressed down.

a. Copying of Analyzed Result

Procedure for Pasting to Spreadsheet Software in Text Format

A copied quantitative analysis result can be pasted to spreadsheet software in the same format as display on [Quant Calculation] (only when one file has been selected).

1. Select a file to be copied, and click [Copy] on the right-click menu.
2. Startup spreadsheet software and paste the data.

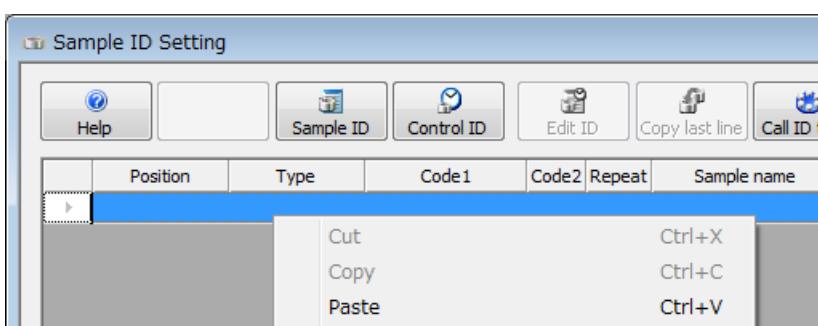
Example:

	A	B	C	D	E	F	G
1	Type	FP Analysis					
2	Folder	*****					
3	File	*****					
4	Count		1				
5	Date	*****					
6	Sample	*****					
7	Application Powder						
8							
9	[Analyzed result]						
10	Component	Analyzed w/ Unit	Statistics	Detection	Quantitation limit		
11	Mg	***** mass%	*****	*****	*****		
12	Al	***** mass%	*****	*****	*****		
13	Si	***** mass%	*****	*****	*****		
14	P	***** mass%	*****	*****	*****		
15	S	***** mass%	*****	*****	*****		
16	Cl	***** mass%	*****	*****	*****		
17	K	***** mass%	*****	*****	*****		
18	Ca	***** mass%	*****	*****	*****		
19	Ti	***** ppm	*****	*****	*****		
20	Mn	***** ppm	*****	*****	*****		
21	Fe	***** ppm	*****	*****	*****		

Procedure for Pasting as Analysis Sample ID

When the spectrometer condition settings (analysis diameter and sample changer) are the same as those for the analyzed result to be copied, it can be pasted as an analysis sample ID.

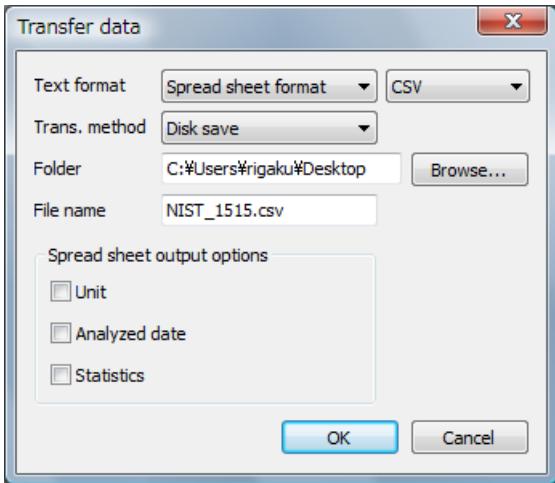
1. Select a file to be copied, and click [Copy] on the right-click menu.
2. Right-click a sample ID line on the [Sample ID Setting] dialog, and select [Paste].



3. If necessary, change the sample ID information such as the file name.

b. Data Transfer of Quantitative Analysis Result in Text Format

1. Select a file or files for a data transfer on the analyzed result list, and right-click the mouse on the selection list.
2. Click [Transfer data] on the right-click menu.



3. Set a transfer method and click [OK].



- When the text format is [Spread sheet format]:
 - Select a file format from [CSV] or [TAB].
 - The transfer method is fixed to [Disk save].
 - The output option [Statistics] is displayed only when two or more files have been selected.
- When the text format is [Transmission format]:
 - Select a transfer method from [Disk save] and [RS232C Transmission].
 - When two or more files have been selected, the data transfer in the transmission format cannot be used.

Example: Plural Files Selected, Text Format: Spreadsheet/CSV

	A	B	C	D	E	F	G
1			Mg		Al		Si
2	NIST1515	Powder	*****		*****		*****
3	NIST1547	Powder	*****		*****		*****
4	NIST1549	Powder	*****		*****		*****

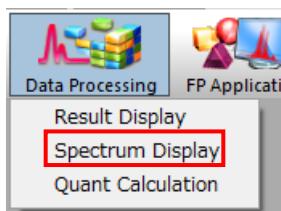
7.3 SPECTRUM DISPLAY

By using the spectrum display function, a spectrum can be displayed, edited and printed.

a. Display of Spectrum

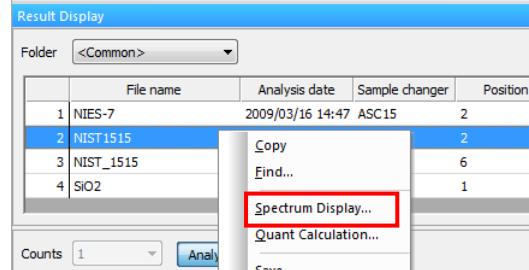
1. Select [Spectrum Display] on the menu of [Data Processing] on the tool bar or on [Result Display].

Selection on Tool Bar



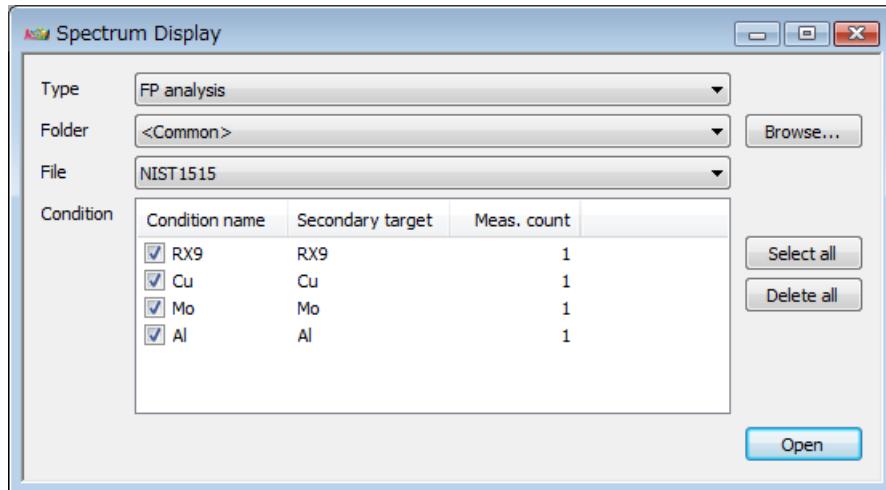
Click [Data Processing] on the tool bar, and make a selection on the menu that appears.

Selection on [Result Display]



Right-click an objective file and make a selection on the menu that appears.

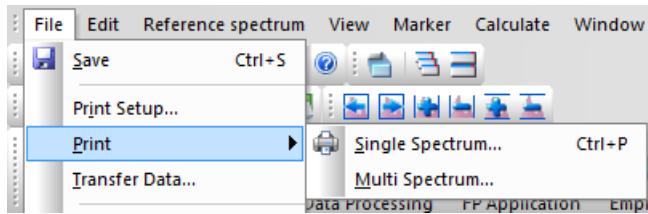
2. Select a file for processing such as editing. Select the type of the analysis condition used for the analysis of the objective file, the folder in which the file has been saved, and a file name.



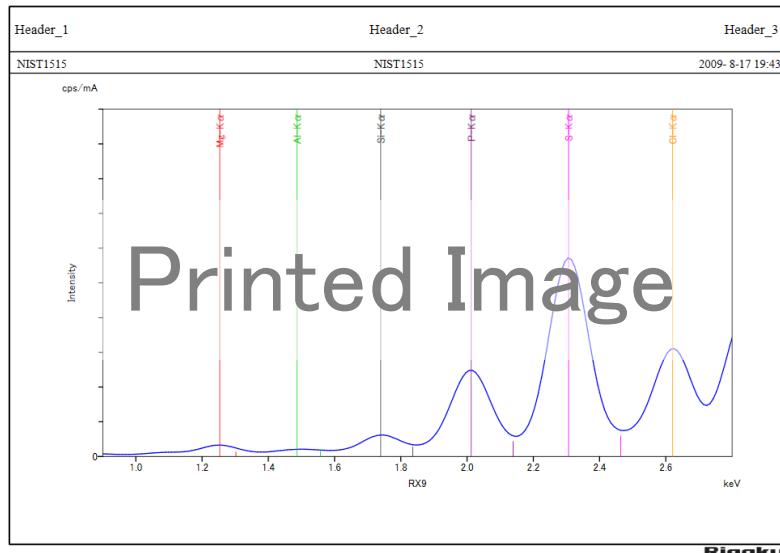
- All the results of [EZ Analysis] are automatically saved in [Common Folder].

b. Printing of Spectrum

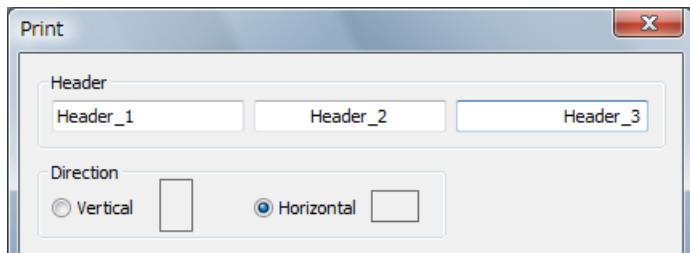
1. Display a spectrum to be printed.
2. Select [File] → [Print] on the menu to print the spectrum. Select [Single Spectrum] or [Multi Spectrum] for the printing.



Printing of Single Spectrum



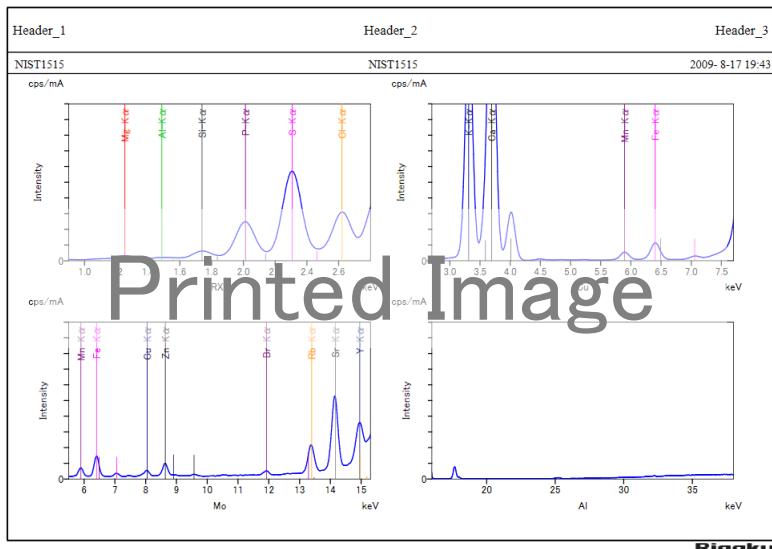
3. Left-click a spectrum to be printed to make it displayed in the foreground.
4. Click [File] on the menu and select [Print] → [Single Spectrum].
5. Set a header and paper direction.



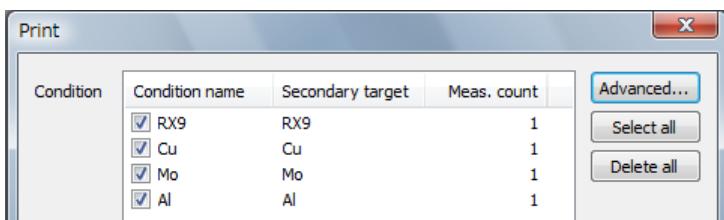
6. Click [OK].

7. DATA PROCESSING

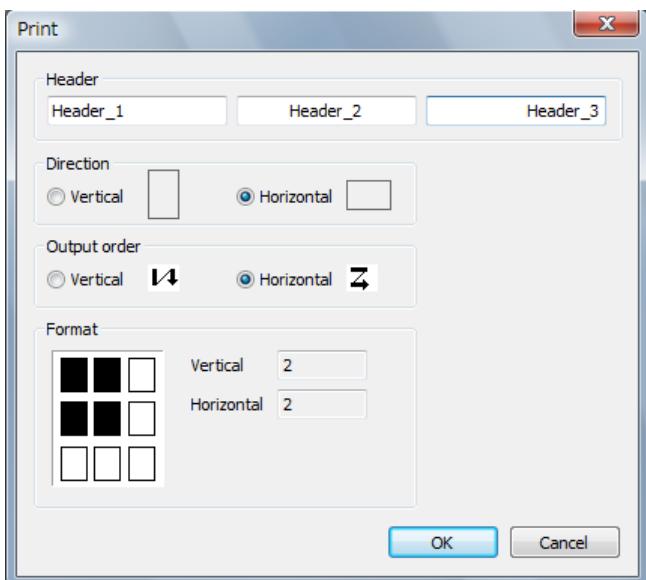
Printing of Multiple Spectra



3. Click [file] on the menu and select [Print] → [Multi Spectrum].
4. Select the check boxes of conditions to be printed.



5. Click [Advanced].
6. Set a header, paper direction and arrangement, and click [OK].

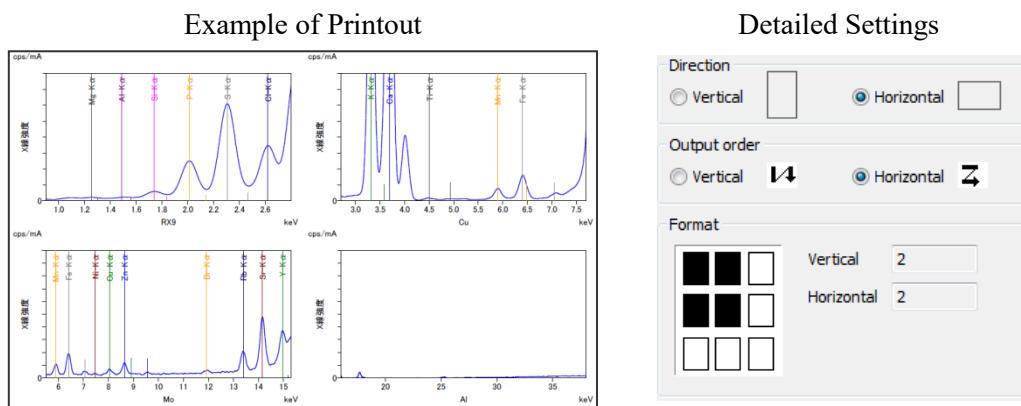


7. Click [OK].

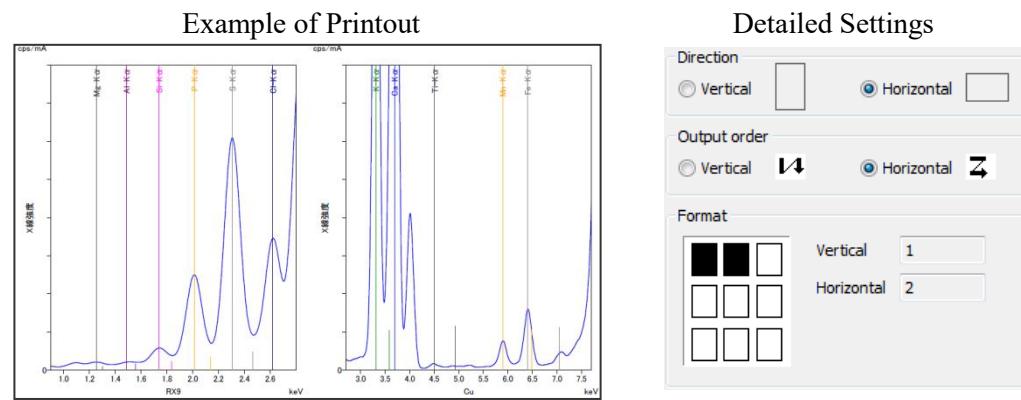


- Examples of printouts and detailed settings are shown below:

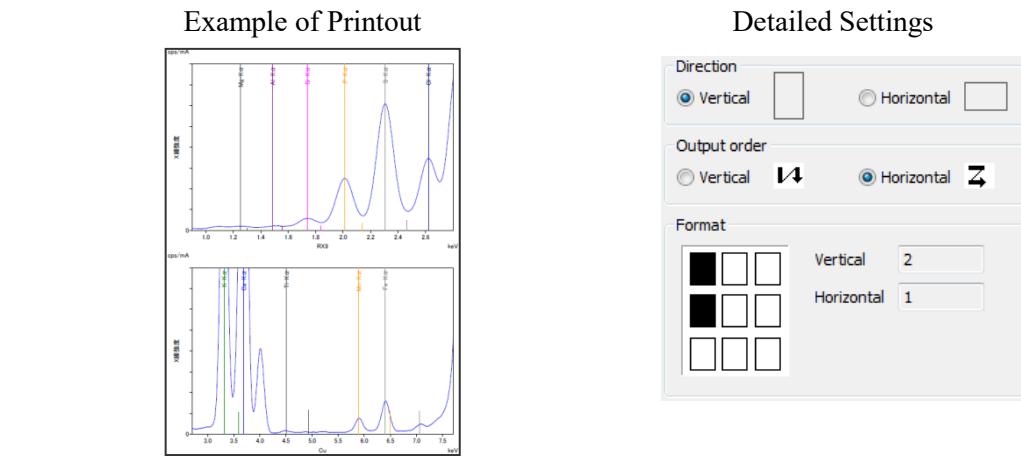
- Example of Printout (1)



- Example of Printout (2)



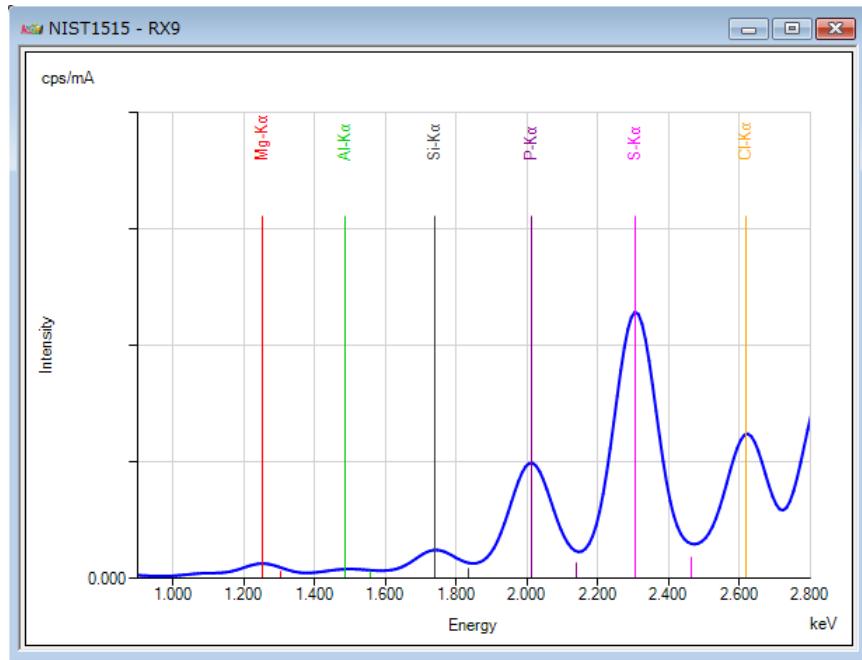
- Example of Printout (3)



c. Copying of Spectrum Image to Clipboard

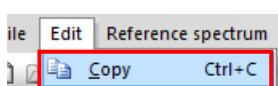
A spectrum now on display can be copied to the clipboard (only in the case of a single spectrum). The copied spectrum can be pasted to other software as an image (bitmap file).

1. Display a spectrum to be copied.

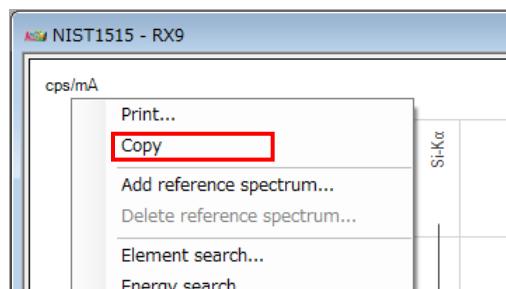


2. Left-click the window of the spectrum to be copied to make it displayed in the foreground, and then select [Edit] → [Copy] on the menu. Or right-click the mouse on the window of the spectrum, and select [Copy] on the menu that appears.

Selection on Menu



Selection on Right-Click Menu



d. Editing of Element Marker

Addition of Element Marker

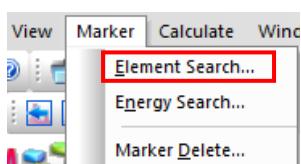
To add a new element marker, a procedure can be selected from (1) [Element Search] and (2) [Energy Search].

1. Display a spectrum for which to add an element marker.

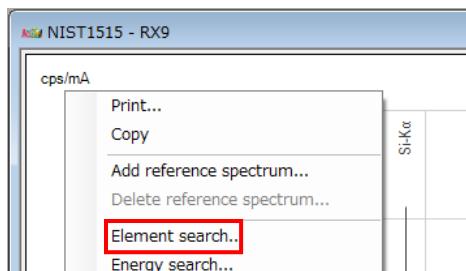
- (1) Addition of Element Marker From [Element Search]

2. Display a spectrum to be edited.
3. Select [Marker] → [Element Search] on the menu. Or right-click the mouse on the window of the spectrum, and select [Element Search] on the menu that appears.

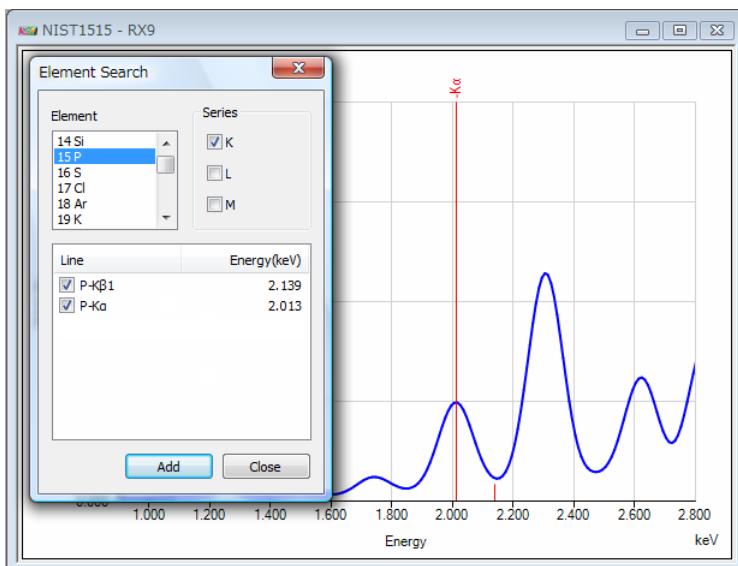
Selection on Menu



Selection on Right-Click Menu



4. Select an element and the check box of a line type (series and characteristic line) to be added. A characteristic line for which to add a marker appears in red in the display range of the spectrum.

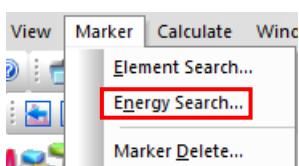


5. Click [Add].
6. Select [File] → [Save] on the menu, and the changed contents will be saved.

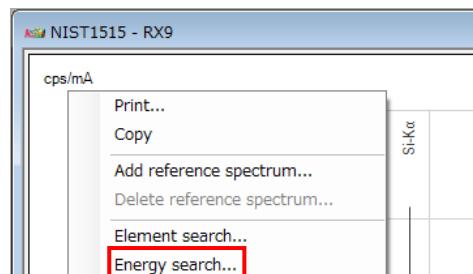
(2) Addition of Element Marker From [Energy Search]

2. Left-click the place of the characteristic line energy to be selected for a position selection.
3. Select [Marker] → [Energy Search] on the menu. Or right-click the mouse on the window of the spectrum, and select [Energy Search] on the menu that appears.

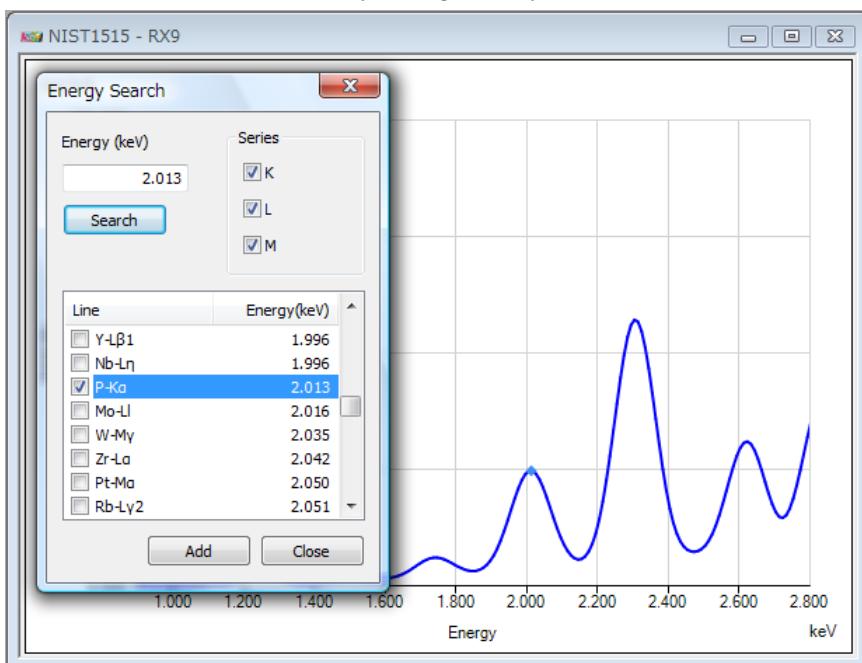
Selection on Menu



Selection on Right-Click Menu



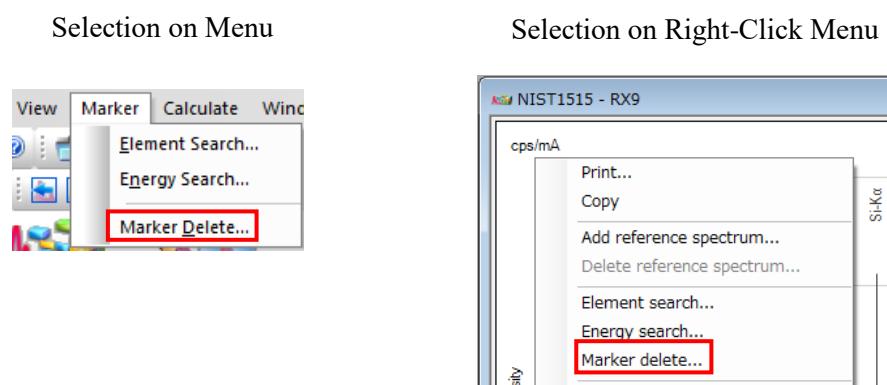
4. The energy for which the position has been selected has automatically been entered. Click [Search] without any changes. Or you can enter a value directly.



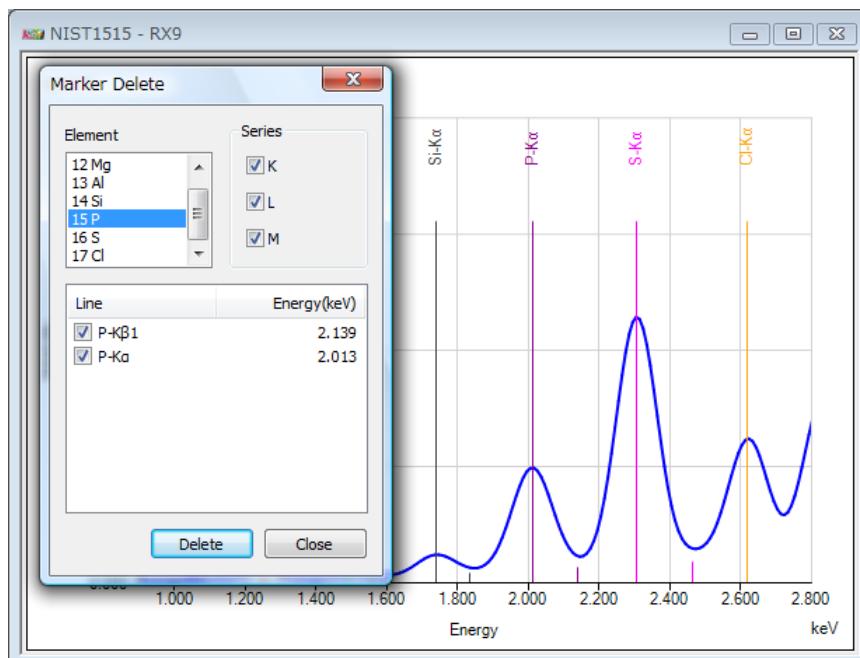
5. From the displayed lines, select the check box of a characteristic line to be added.
6. Click [Add].
7. Select [File] → [Save] on the menu, and the changed contents will be saved.

Deletion of Element Marker

1. Display a spectrum for which to delete an element marker.
2. Select [Marker] → [Marker Delete] on the menu. Or right-click the mouse on the window of the spectrum, and select [Marker Delete] on the menu that appears.



3. Select a characteristic line to be deleted, and click [Delete].

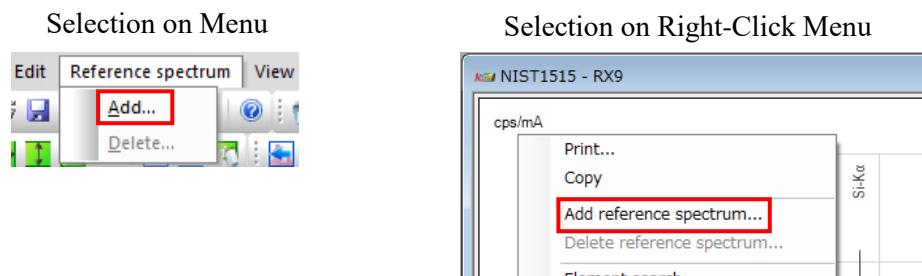


4. Select [File] → [Save] on the menu, and the changed contents will be saved.

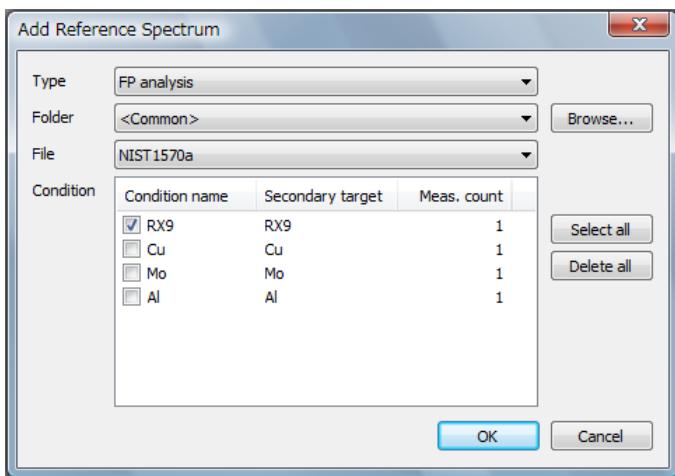
e. Overlapping Display of 2 or More Spectra

Addition of Reference Spectrum

1. Display a spectrum.
2. Select [Reference spectrum] → [Add] on the menu. Or right-click the mouse on the window of the spectrum, and select [Add reference spectrum] on the menu that appears.

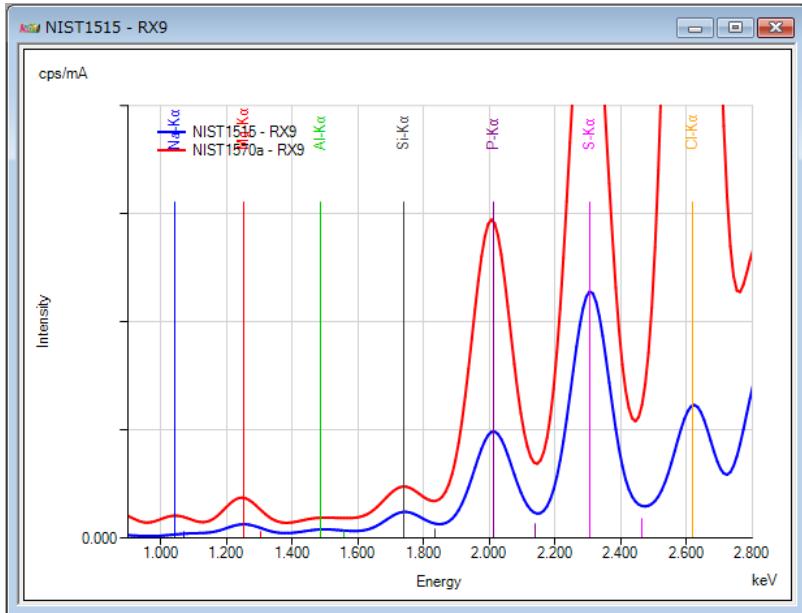


3. Select the type, folder and file of a reference spectrum, and select a measuring condition.



- A spectrum with the same condition (secondary target) as that of an objective spectrum should ordinarily be selected as a reference spectrum to be overlapped.

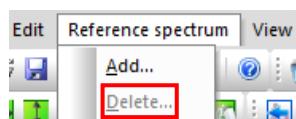
4. Click [OK].



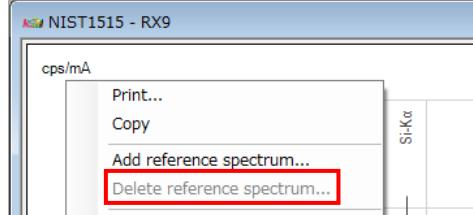
Deletion of Reference Spectrum

1. Left-click the window of a reference spectrum to be deleted to make it displayed in the foreground.
2. Select [Reference spectrum] → [Delete] on the menu. Or right-click the mouse on the window of the spectrum, and select [Delete reference spectrum] on the menu that appears.

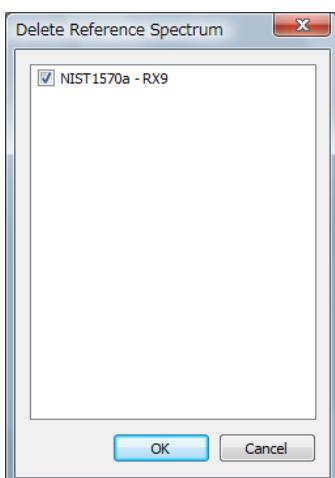
Selection on Menu



Selection on Right-Click Menu

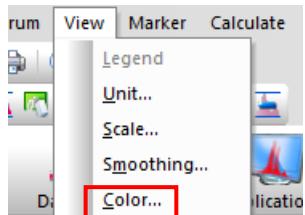


3. Select the reference spectrum to be deleted, and click [OK].

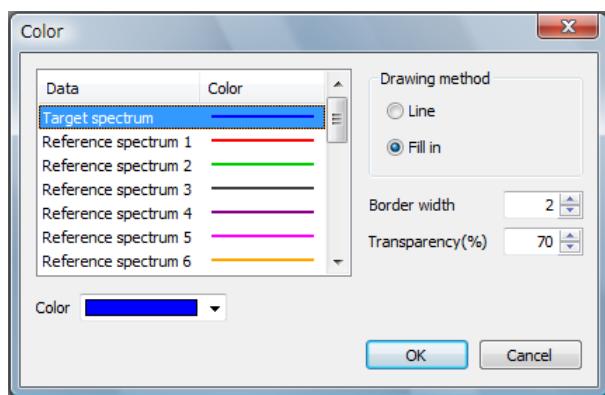


f. Change of Spectrum Color

1. Display a spectrum.
2. Select [View] → [Color] on the menu.



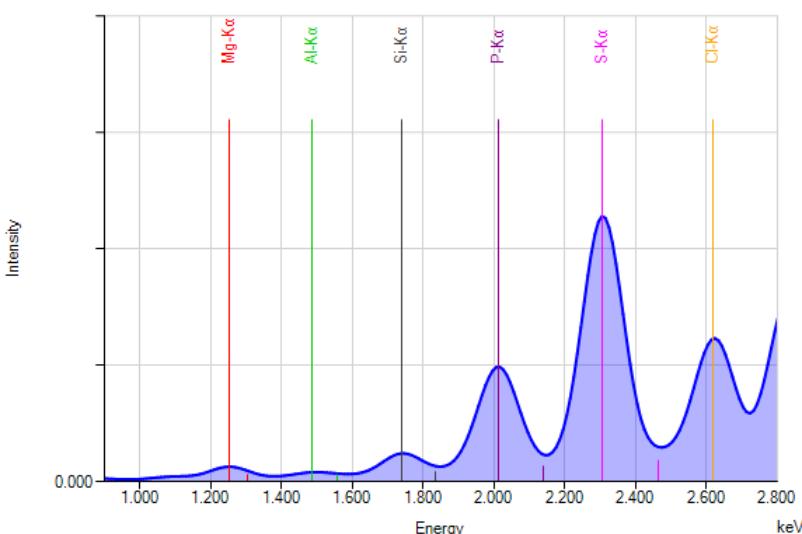
3. Select a spectrum for which to change the settings, and set a drawing method, color, width of a boundary and transparency (only when filling is selected).



4. Click [OK].
5. Select [File] → [Save] on the menu, and the changed contents will be saved.

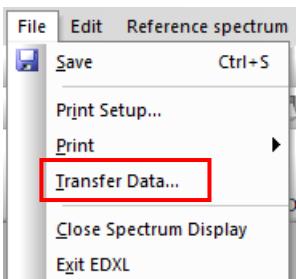
Example: Drawing Method: Filling, Color: Blue, Width of Boundary: 2, Transparency: 70%

cps/mA

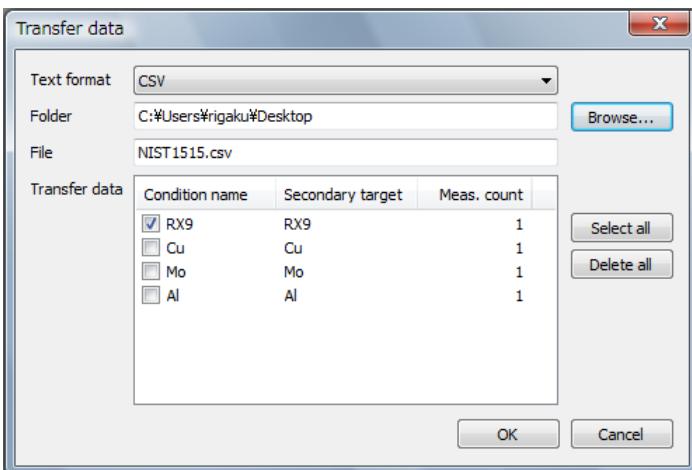


g. Transfer of Spectrum Data in Text Format

1. Display a spectrum for a data transfer.
2. Select [File] → [Transfer Data] on the menu.



3. Set a text format ([CSV] or [TAB]), folder for saving and file name, and select a condition name (secondary target) for a data transfer.



4. Click [OK].

Example:

	A	B
1	1	
2	RX9	
3	keV	cps/mA
4	0.008	209.748
5	0.018	204.543
6	0.028	191.663
7	0.038	176.43
8	0.048	151.087
9	0.058	126.271
10	0.068	98.825
11	0.070	75.447

h. List of Operation Items

Operation Items on Menu



Menu	Item	Description
File	Save	A changed scale, element marker, etc. are saved.
	Print	A spectrum is printed on the printer.
	Transfer Data	Spectrum data is transferred in the text format.
Edit	Copy	A spectrum that has now been selected is copied to the clipboard.
Reference spectrum	Add	Overlapped spectra are displayed.
	Delete	A reference spectrum is excluded from overlapping display.
View	Legend	The legend is displayed.
	Unite	The unit is changed.
	Scale	The scale to display a spectrum is changed.
	Smoothing	The number of smoothing points is changed.
	Color	The color and line width of a spectrum is changed.
Marker	Element Search	Characteristic lines are retrieved from an element name, and an element marker is added.
	Energy Search	Characteristic lines are retrieved from energy, and an element marker is added.
	Marker Delete	An element marker is deleted.
Calculate	ROI intensity	An integrated intensity in a selected region is displayed.
	Resolution	Energy resolution is displayed.
	Normalize	An objective spectrum is normalized to the intensity of a reference spectrum in a specified energy region.
	Subtract	The intensities of an objective spectrum minus those of a reference spectrum are displayed.

Operation Items on Menu Bar

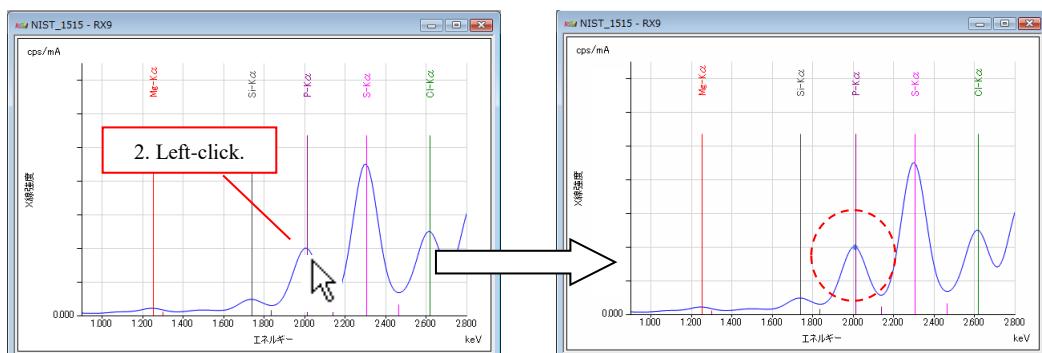
Icon	Description
	A check mark is placed on a spectrum. An energy position is selected.
	A region along the X-axis is selected.
	A region along the Y-axis is selected.
	Regions along the X- and Y-axes are selected simultaneously.
	The scale is changed to a selected region.
	The scale is returned to the default scale (initial display region).
	The scale is changed according to the maximum intensity in the display region.
	The position and region selections are canceled.
	The spectrum is moved to the low-energy side (left).
	The spectrum is moved to the high-energy side (right).
	The spectrum is expanded in the X-axis direction.
	The spectrum is reduced in the X-axis direction.
	The spectrum is expanded in the Y-axis direction.
	The spectrum is reduced in the Y-axis direction.



- A position or a region can be selected using the mouse. By using a right-click menu, operations can be carried out efficiently.

Position Selection Using Mouse

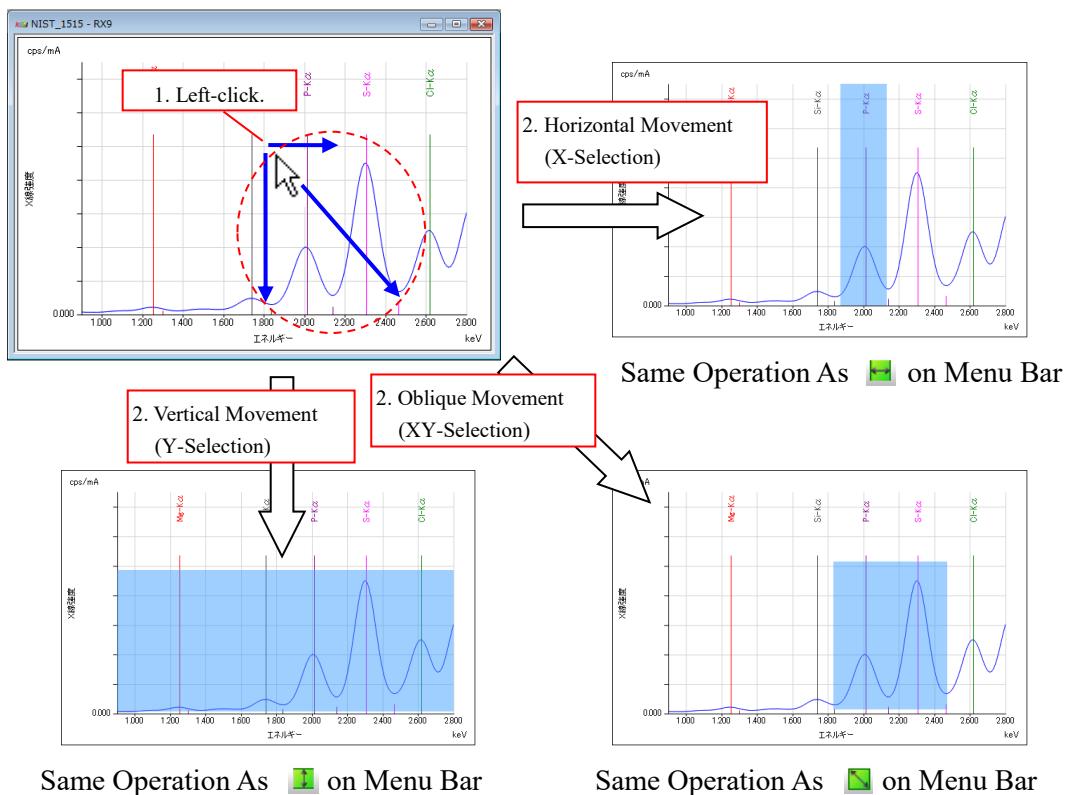
1. Move the mouse cursor to a position to be selected.
2. Left-click the mouse. The selected energy value is automatically entered when executing [Energy Search].



Same Operation As on Menu Bar

Region Selection Using Mouse

1. Move the mouse cursor to the start point of a region to be selected, and left-click the mouse.
2. With the left mouse button pressed down, move the mouse cursor to the end point of the region to be selected. The region in which the left mouse button has been pressed down is read as an energy region.
3. When the icon or [Execute] on the right-click menu is clicked, the selected region is expanded. The selected region is automatically entered for [Scale], [ROI intensity], etc.



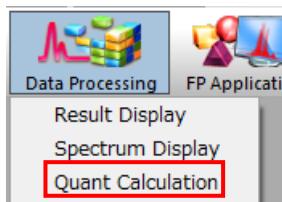
7.4 QUANTITATIVE CALCULATION

Quantitative calculation results can be recalculated and printed by using the quantitative calculation function.

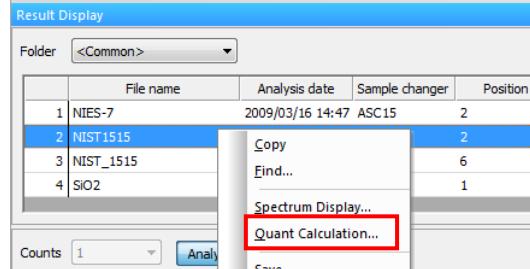
a. Display of Quantitative Calculation

1. Select [Data Processing] on the tool bar or [Quant Calculation] on [Result Display].

Selection on Tool Bar



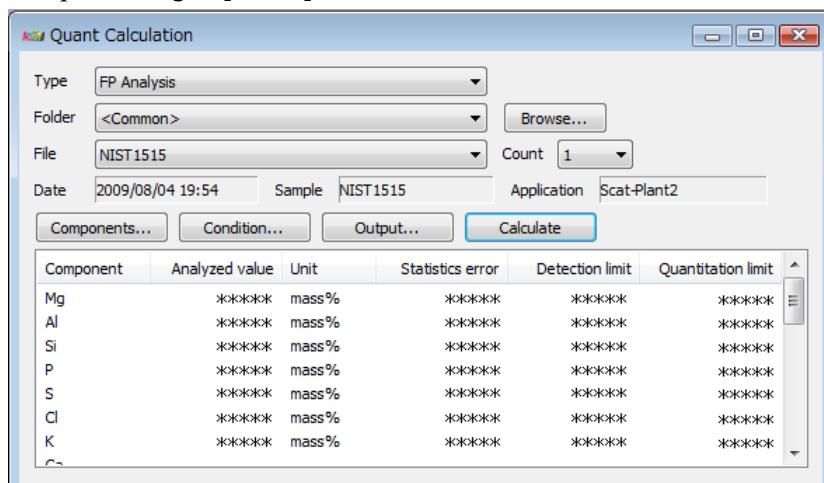
Selection on [Result Display]



Click [Data Processing] on the tool bar, and select an item on the menu that appears.

Right-click an objective file, and select an item on the menu that appears.

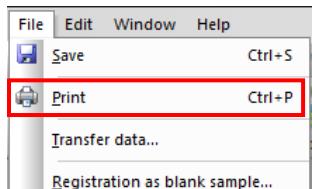
2. Select a file for processing such as editing. Select the type of an analysis condition used for the analysis of the objective file, a folder in which the file has been saved and a file name. When two or more measurements have been made, select a repeat counter for processing at [Count].



- All the results of analyses using [EZ Analysis] are automatically saved in the common folder.

b. Printing of Quantitative Calculation Result

1. Display a quantitative calculation result to be printed.
2. Select [File] → [Print] on the menu.



Example:

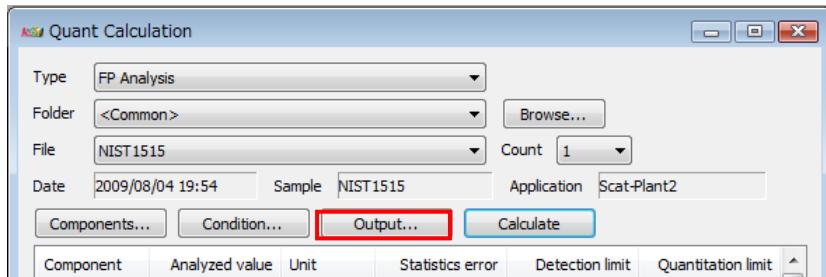
Analyzed result

No.	Component	Result	Unit	Statistical error	Detection limit	Quantitation limit
1.	Mg	*****	mass%	*****	*****	*****
2.	Al	*****	mass%	*****	*****	*****
3.	Si	*****	mass%	*****	*****	*****
4.	P	*****	mass%	*****	*****	*****
5.	S	*****	mass%	*****	*****	*****
6.	Cl	*****	mass%	*****	*****	*****
7.	K	*****	mass%	*****	*****	*****
8.	Ca	*****	mass%	*****	*****	*****
9.	Mn	*****	mass%	*****	*****	*****
10.	Fe	*****	mass%	*****	*****	*****
11.	Ni	*****	mass%	*****	*****	*****
12.	Cu	*****	mass%	*****	*****	*****
13.	Zn	*****	mass%	*****	*****	*****
14.	Br	*****	mass%	*****	*****	*****
15.	Rb	*****	mass%	*****	*****	*****
16.	Sr	*****	mass%	****	****	****
17.	Y	*****	mass%	*****	*****	*****
18.	Ba	*****	mass%	*****	*****	*****
19.	C6H10O5	*****	mass%	*****	*****	*****
20.	Total	*****	mg/cm ²			

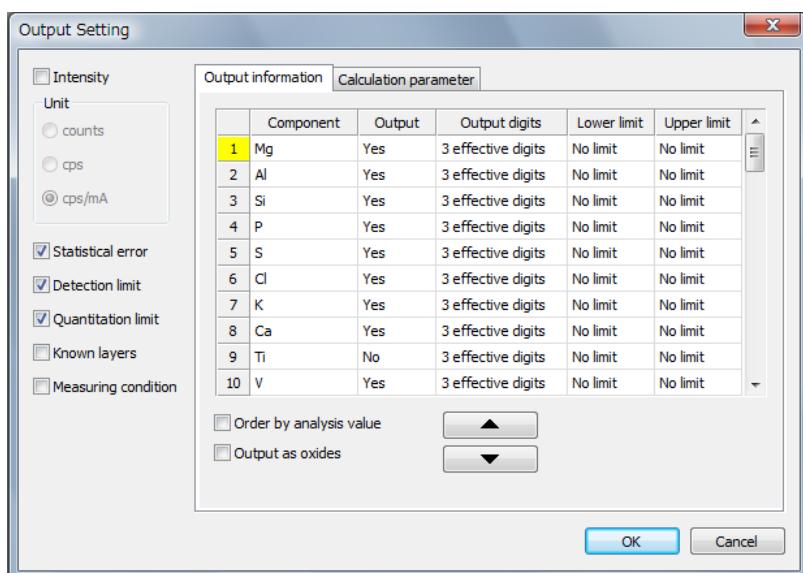
Rigaku

c.Change of Output Setting (Printing Order, Output Digits, Output/No Output)

1. Display a quantitative calculation result.
2. Click [Output].



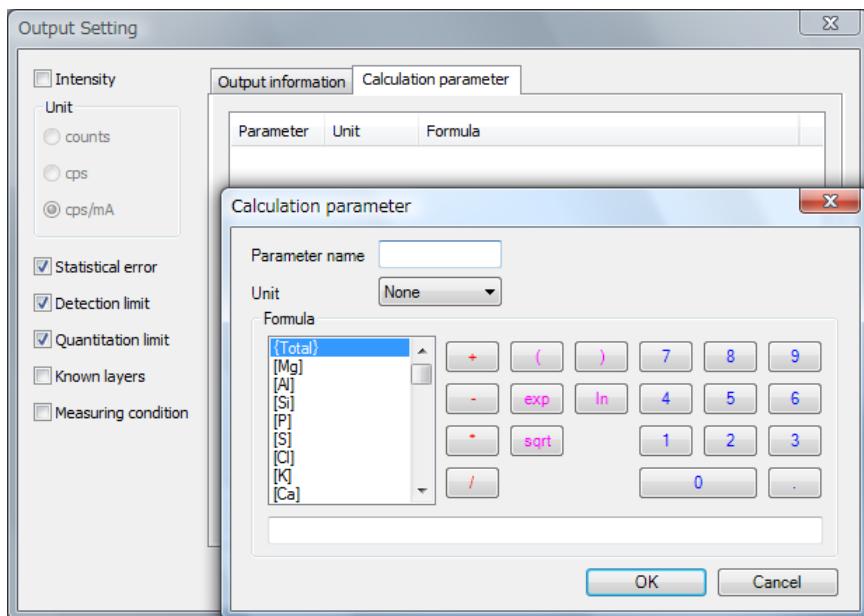
3. Set printing conditions.
 - Select the check boxes of items to be printed.
 - For each component, settings can be made for whether it is to be output or not, the number of output digits, lower limit and upper limit.
 - By selecting a component and clicking [\blacktriangle] or [\blacktriangledown], printing order can be changed.
 - By selecting the check box [Order by analysis value], components can be output in order of analysis value from the largest to the smallest.
 - When the check box [Output as Oxides] is selected, the concentrations of all components are converted from those of elements (Si, Fe, etc.) into those of oxides (SiO_2 , Fe_2O_3 , etc.) and printed out.



4. Click [OK].
5. Select [File] → [Save] on the menu, and the changed contents will be saved.

Setting of Calculated Parameter

1. Click the [Calculation parameter] tab.
2. Click [Add], and a dialog to input a formula will appear.
3. Set a parameter name and a unit.
4. Create a formula by double-clicking components and single-clicking calculation symbols and numerals.
5. Click [OK].



6. Click [Calculate].



- After editing a calculated parameter, be sure to click [Calculate]. By making a calculation, a formula is reflected in a result.

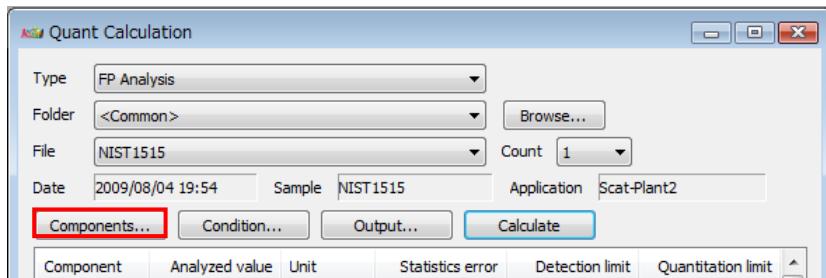
7. DATA PROCESSING

d. Change of Unit (Only for FP Calculation)

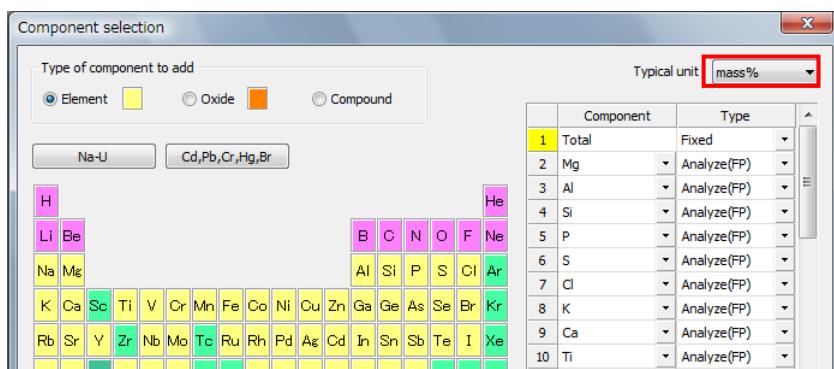
To change a unit or units, (1) the units of all components can be changed at a time or (2) the unit of each component can be changed.

1) Change of Units of All Components

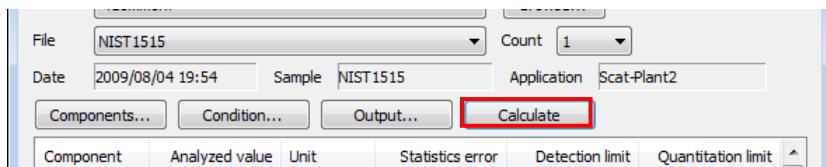
1. Display a quantitative calculation result for which to change units.
2. Click [Components].



3. Change the setting for [Typical unit], and click [OK].



4. Click [Calculate].



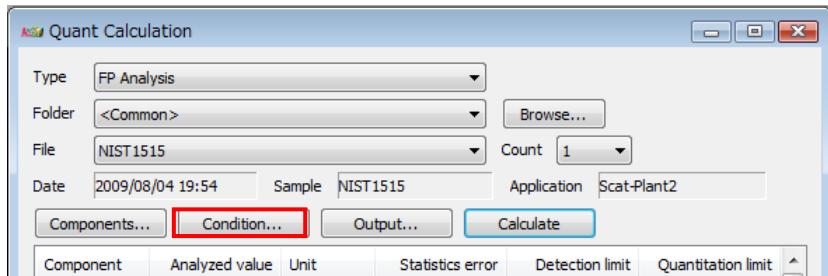
5. Select [File] → [Save] on the menu, and the changed contents will be saved.



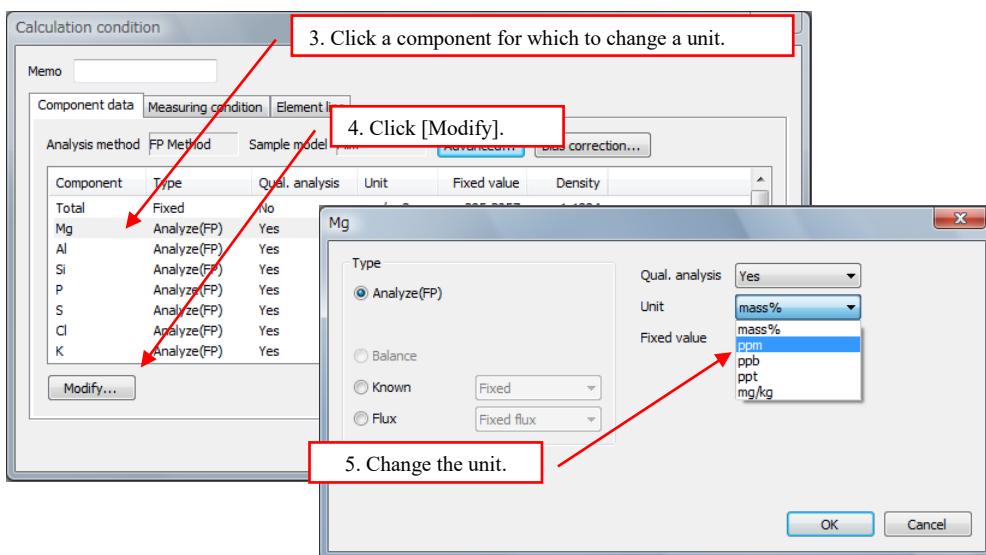
- After changing units, be sure to click [Calculate]. By making a calculation, analysis values are converted according to the units.

2) Change of Unit of Each Component

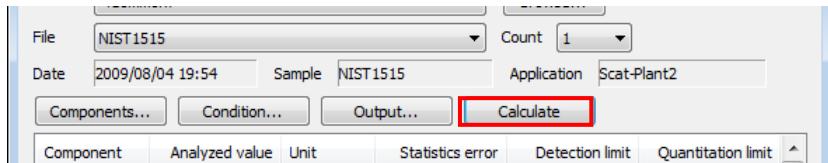
1. Display a quantitative calculation result for which to change a unit.
2. Click [Condition].



3. Click a component for which to change a unit.
4. Click [Modify].
5. Change the unit and click [OK].



6. Click [OK].
7. Click [Calculate].



8. Select [File] → [Save] on the menu, and the changed contents will be saved.

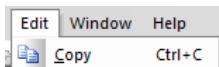


- After changing a unit, be sure to click [Calculate]. By making a calculation, an analysis value is converted according to the unit.

e.Copying of Quantitative Calculation Result to Clipboard in Text Format (Like Printout)

A quantitative calculation result now on display can be copied to the clipboard. The copied quantitative calculation result can be pasted to spreadsheet software in the same format as that for display using [Quant Calculation].

1. Display a quantitative calculation result to be copied.
2. Select [Edit] → [Copy] on the menu.



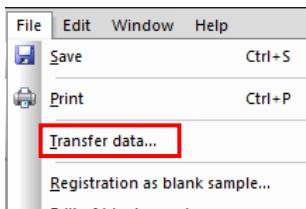
3. Start up spreadsheet software and paste the result.

Example:

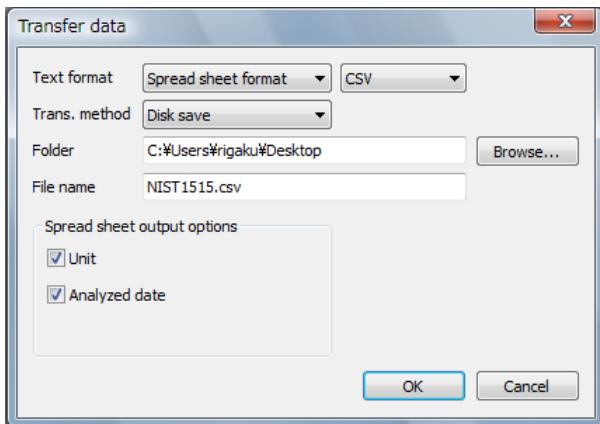
	A	B	C	D	E	F	G
1	Type	FP Analysis					
2	Folder	*****					
3	File	*****					
4	Count		1				
5	Date	*****					
6	Sample	*****					
7	Application	Powder					
8							
9	[Analyzed result]						
10	Component	Analyzed w/ Unit	Statistics	↓ Detection	Quantitation	limit	
11	Mg	***** mass%	*****	*****	*****		
12	Al	***** mass%	*****	*****	*****		
13	Si	***** mass%	*****	*****	*****		
14	P	***** mass%	*****	*****	*****		
15	S	***** mass%	*****	*****	*****		
16	Cl	***** mass%	*****	*****	*****		
17	K	***** mass%	*****	*****	*****		
18	Ca	***** mass%	*****	*****	*****		
19	Ti	***** ppm	*****	*****	*****		
20	Mn	***** ppm	*****	*****	*****		
21	Fe	***** ppm	*****	*****	*****		

f. Data Transfer of Quantitative Calculation Result

1. Display a quantitative calculation result for a data transfer.
2. Select [File] → [Transfer data] on the menu.



3. Set a transfer method and click [OK].



Example: Text Format: Spreadsheet/CSV

	A	B	C	D	E	F	G	H
1				Mg		Al	Si	
2				mass%		mass%	mass%	
3	NIST1515	Powder	2009/8/4 19:54	*****		*****	*****	
4	Statistical error			*****		*****	*****	
5	Detection limit			*****		*****	*****	
6	Quantitation limit			*****		*****	*****	

g.Calculation After Subtracting Impurity and Impurity Line Data (Only for FP Calculation)

Impurity Correction

By specifying [Impurity] in the quantitative calculation, corrections can be made for impurities mixed during a sample preparation such as crushing, an analysis window (sample film) used for a liquid analysis etc. and impurities contained in installation filter paper. This function can also be used to correct the effects of the impurity lines of Zr, Sn, etc. that are generated in the spectrometer. Impurities are registered by editing components detected by measuring a blank sample.



- Although two or more similar functions such as [Impurity] and [Blank sample] can be used, **[Impurity]** should ordinarily be used.

Example: An impurity is registered and a calculation is made for a quantitative calculation result of SiO₂ after subtracting impurity line (Zr and Sn) data.

1. Prepare a sample to be registered as an impurity. Prepare a pure substance (such as a reagent) that has composition similar to that of an unknown sample. When it is difficult to prepare a sample, a calibration sample in the accessories of the NEX CG can be used.

Example of Measurement Sample	Substance to Be Registered as Impurity
Al Alloy, Mg Alloy	Al metal*
Cement, Soil	SiO ₂ glass*, powder reagent
Iron, Steel, Cu Alloy, Solder, Noble Metals	Cu metal*
Plating Solution, Effluent	Distilled water, SiO ₂ glass*
Polymer, Oil	Plastic reagent, organic solvent, SiO ₂ glass*

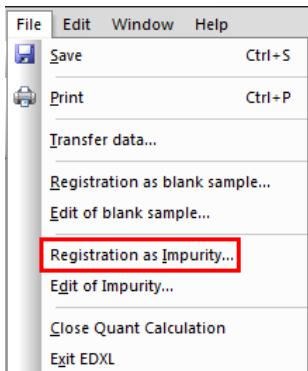
* The Al metal, SiO₂ glass and Cu metal are supplied with the NEX CG.

2. Make an analysis using an FP application.

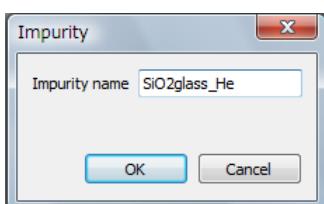


- For the procedure to create an FP application, see “2. CREATION OF FP APPLICATION” in the User manual.
- In the application template, Zr has been excluded from measurement elements. Add it using [Components] on the flow bar. Although it can be added individually after a measurement, we recommend adding it beforehand.

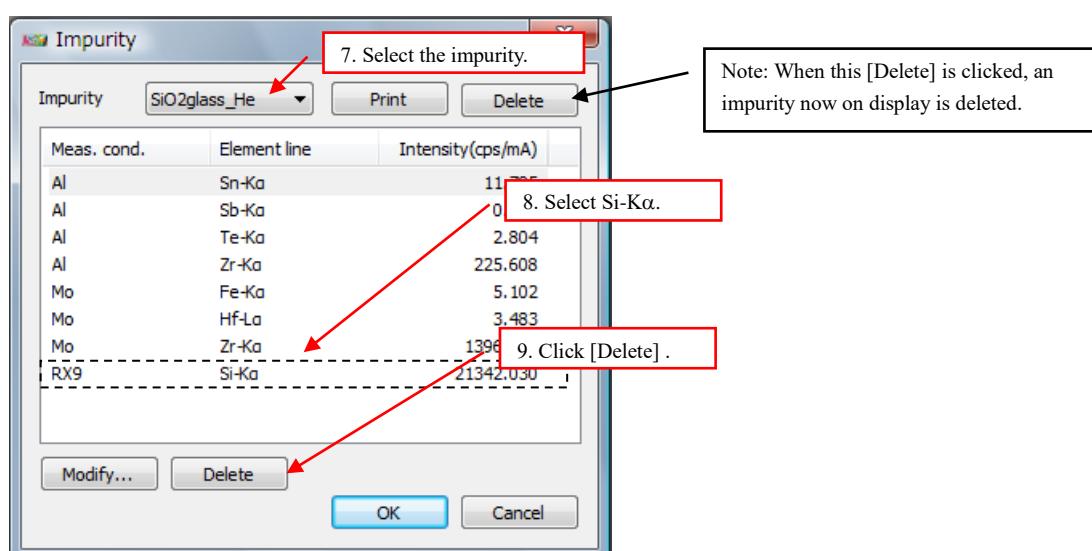
3. Display a quantitative calculation result to be registered as an impurity. When Zr has not been included in the measurement elements of the FP application, add the component using [Components] and then click [Calculate].
4. Select [File] → [Registration as Impurity] on the menu.



5. Enter an impurity name. Click [OK].



6. Click [Edit of Impurity] on the [File] menu.
7. Select the registered impurity.
8. Select Si-K α .
9. Click [Delete] in the lower part.

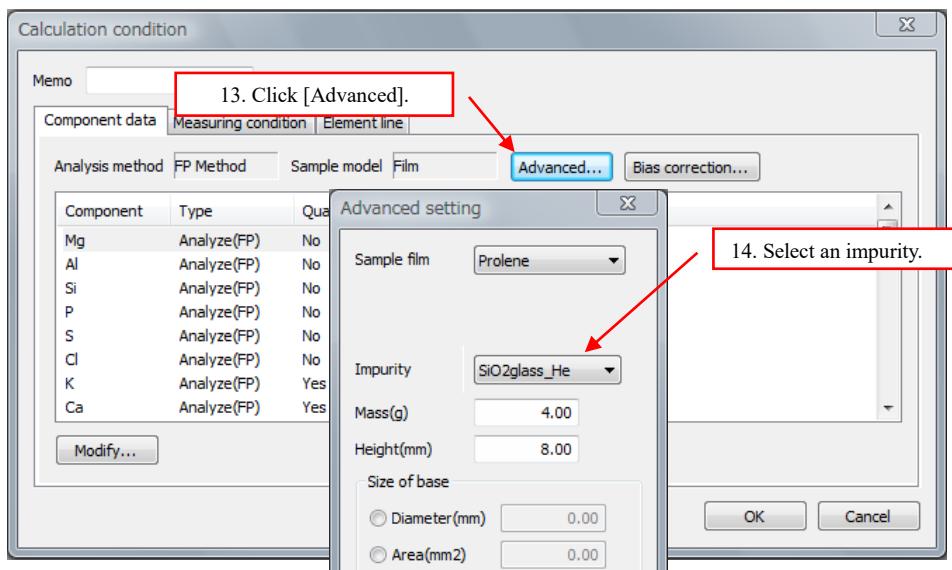


7. DATA PROCESSING

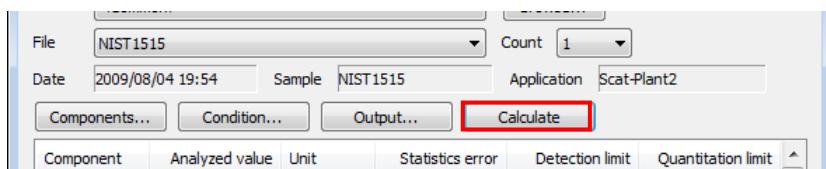


- In the impurity correction, the X-ray intensities of the Element lines used in a specified impurity are subtracted before a calculation. Such subtractions compensate for interfering elements found in the base matrix and also helps to define the background.

10. Click [OK].
11. Display a quantitative calculation result for which to make an impurity correction.
12. Click [Condition].
13. Click [Advanced].
14. Select an impurity and click [OK].
15. Click [OK].



16. Click [Calculate].



- A registered impurity can be used for a result obtained using another application if the measurement atmosphere and measurement diameter in the FP application used are the same.
- An impurity can be registered in an FP application. For the procedure for registration, see “2. CREATION OF FP APPLICATION” in the User Manual.



Appendix I – Language Localization

The factory or authorized Rigaku service representative will enable the analyzer for bilingual language selection between English and one of the supported languages. Language localization applies to the EZ Analysis screen.

Supported languages for EZ Analysis screen:

- English
- Japanese
- Chinese
- Russian
- German
- Spanish
- French
- Italian
- Portuguese

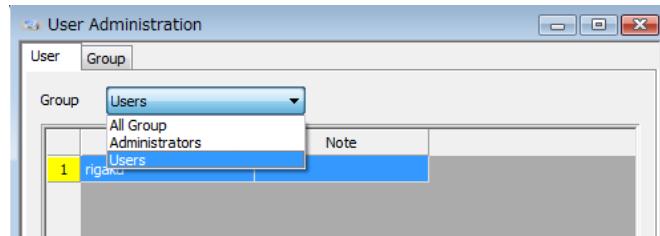
A. User Operation

Toggle between English and selected language

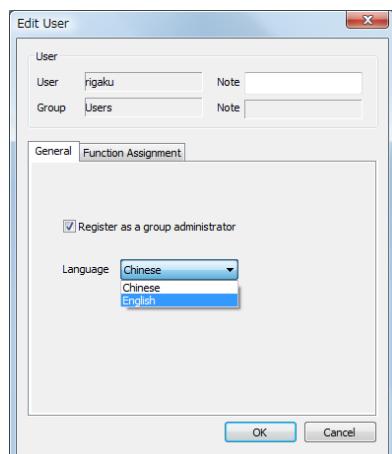
Change the language as follows:

(a) Click [Utility] -> [System management] -> [User administration].

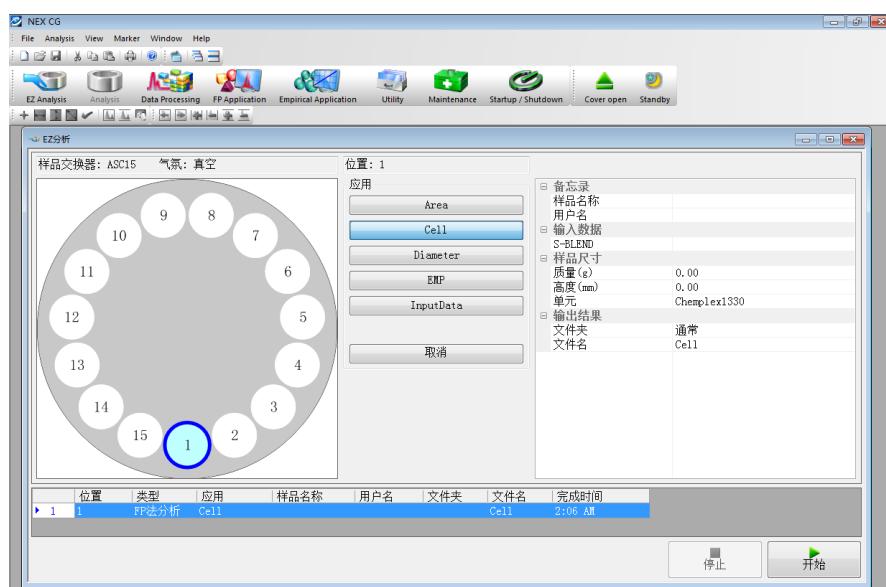
(b) Select [Users] from [Group] list.



(c) Select Users and click [Properties], then choose Language in [Edit User] dialog.

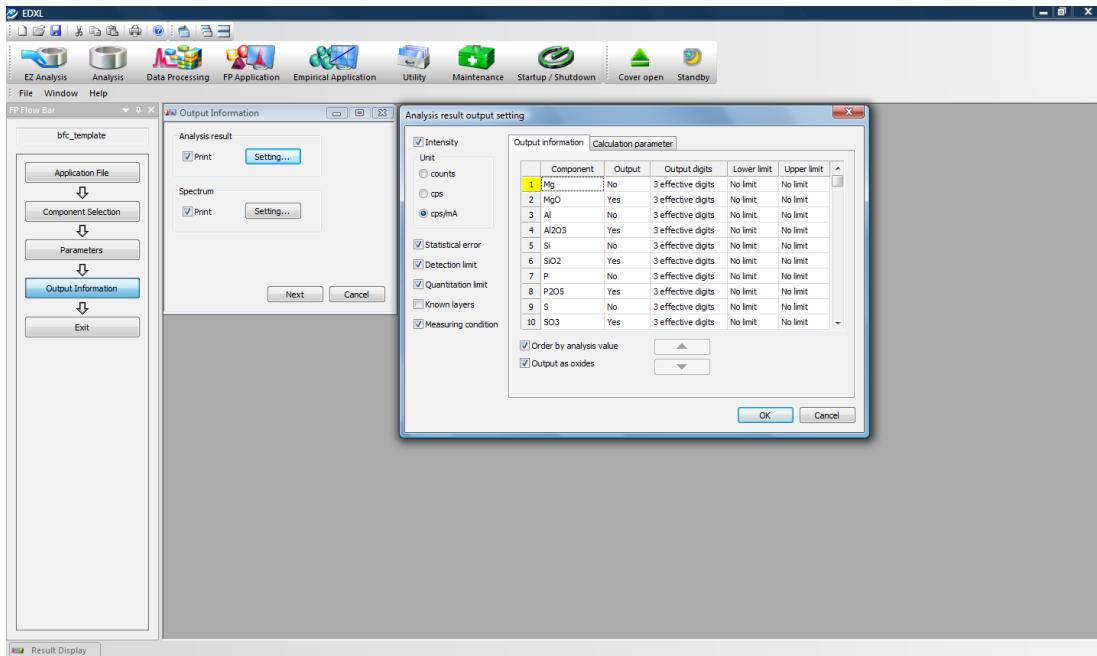


(d) Selected language will be available after restarting NEX CG software.



Appendix II – DATA TRANSMISSION

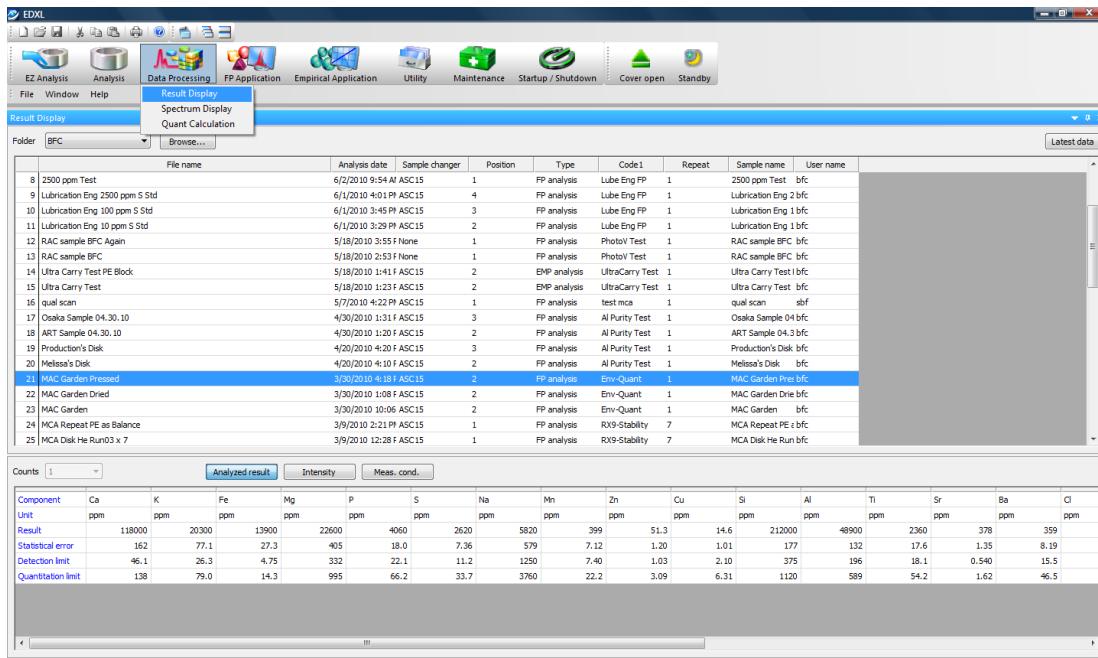
1. DATA OUTPUT PROTOCOLS



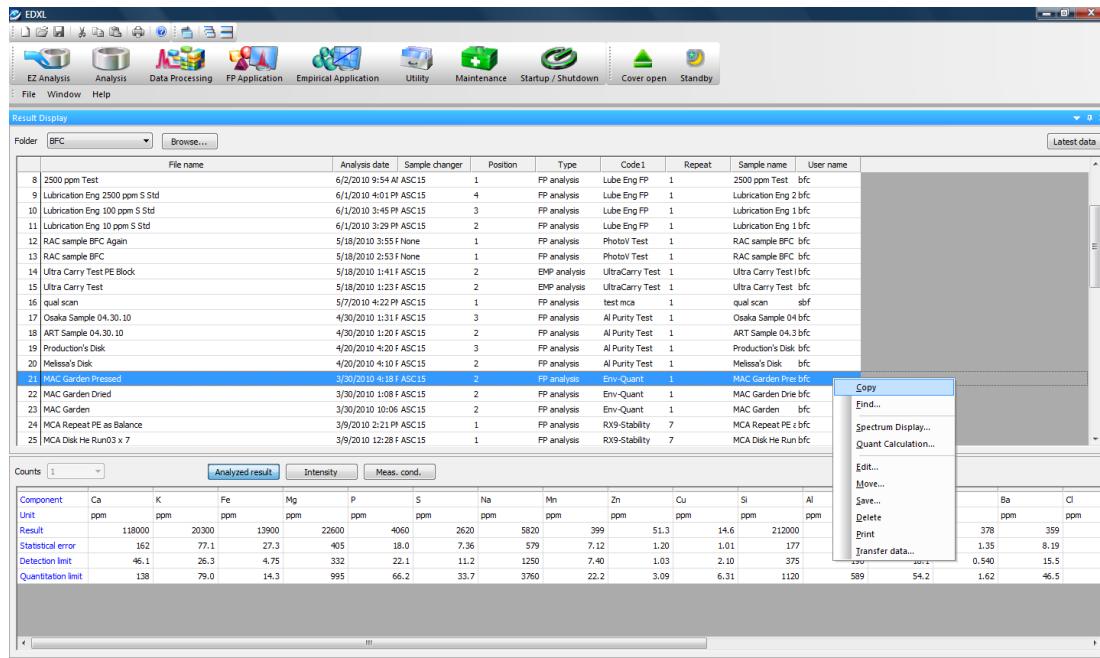
When setting up an analysis method, the output parameters are defined:

1. Selecting the “Print” checkboxes will automatically print results after execution of the analysis.
2. The “Setting” option allows the user to define which elements are output, how many significant figures to display, set upper and lower measurement criteria, display peak intensities, output analytical statistics, and display measurement conditions. The user can also order analytical data from highest to lowest concentration and choose to report elemental results or analogous oxide results

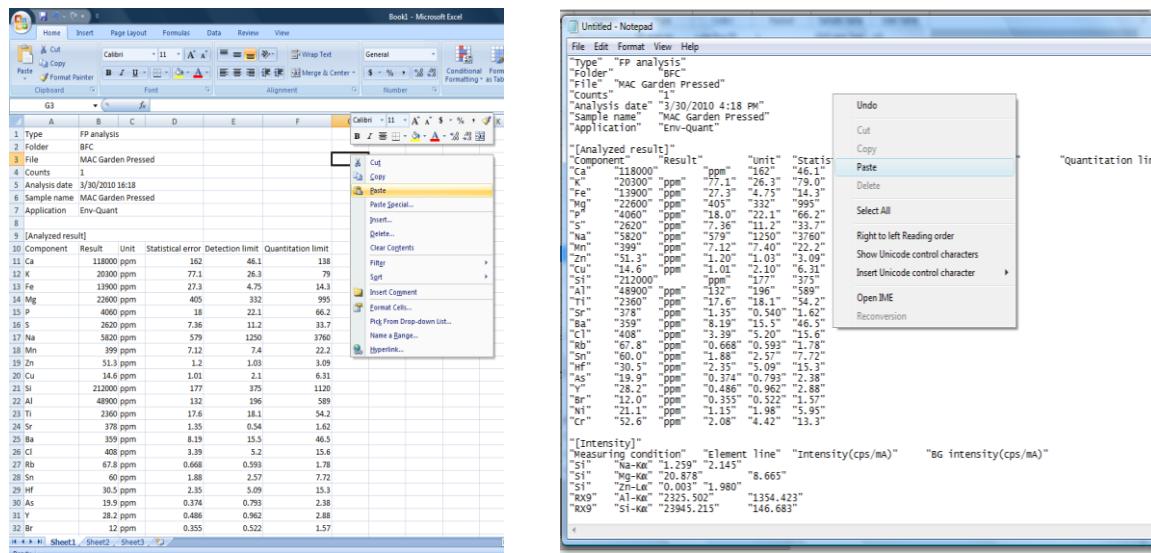
Appendix II



1. After analysis, the user can select “Data Processing” → “Result Display”
2. Select a results file
3. Review the data
 - a) Tab displays quantitative results for all elements in the method
 - b) Tab displays peak intensity information for all elements in the method
 - c) Tab displays the measurement conditions for the sample in question



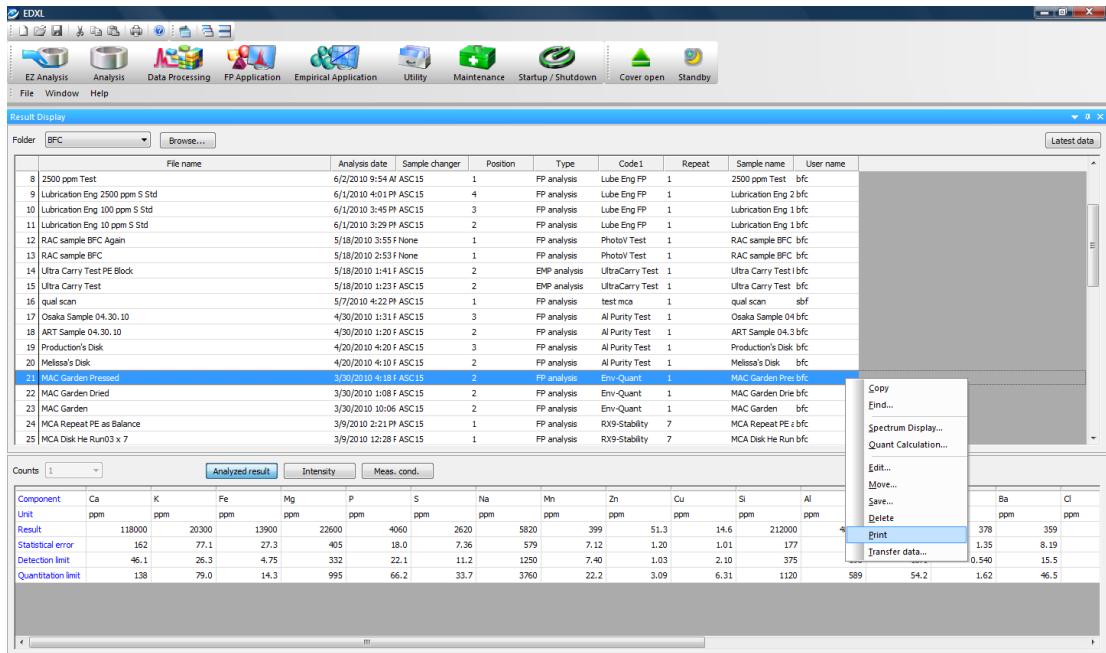
1. Right mouse clicking on individual results brings up a sub-menu where the user can copy results



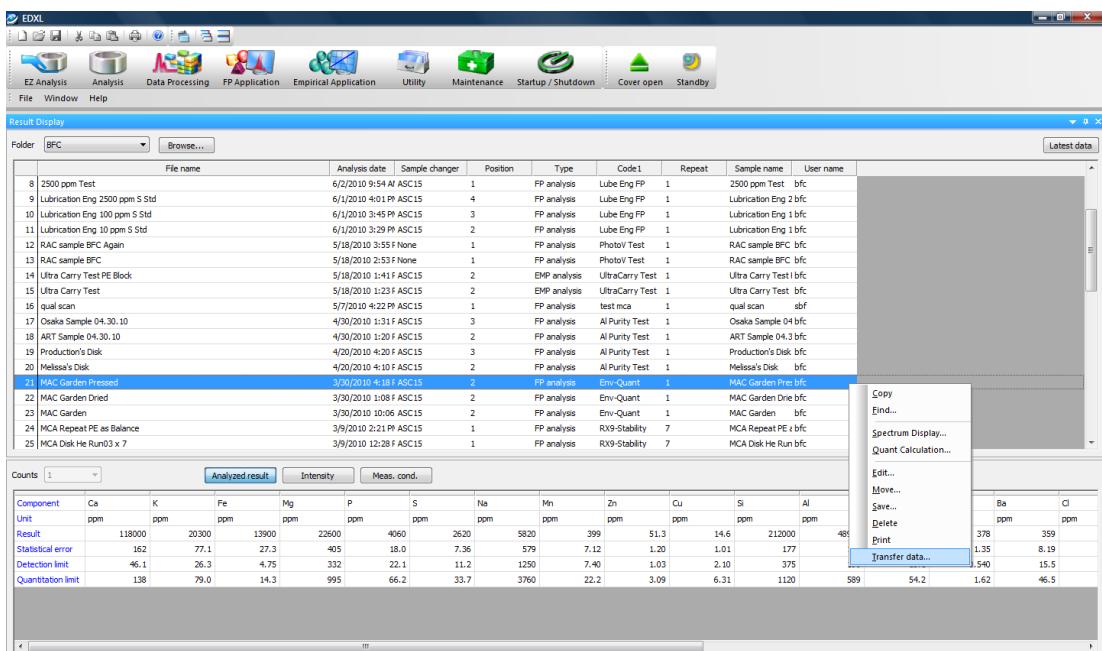
Microsoft Excel

Microsoft Notepad

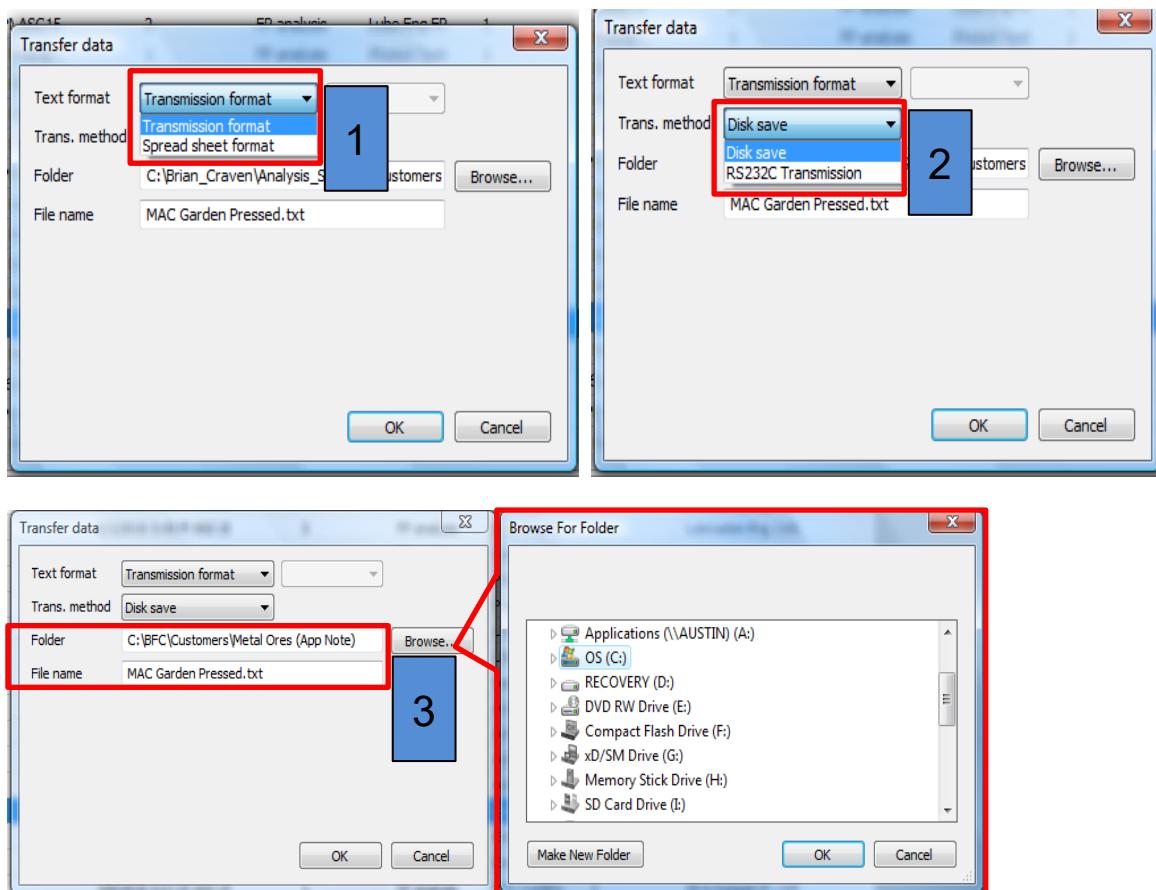
Appendix II



1. Right mouse clicking on individual results brings up a sub-menu where the user can print results

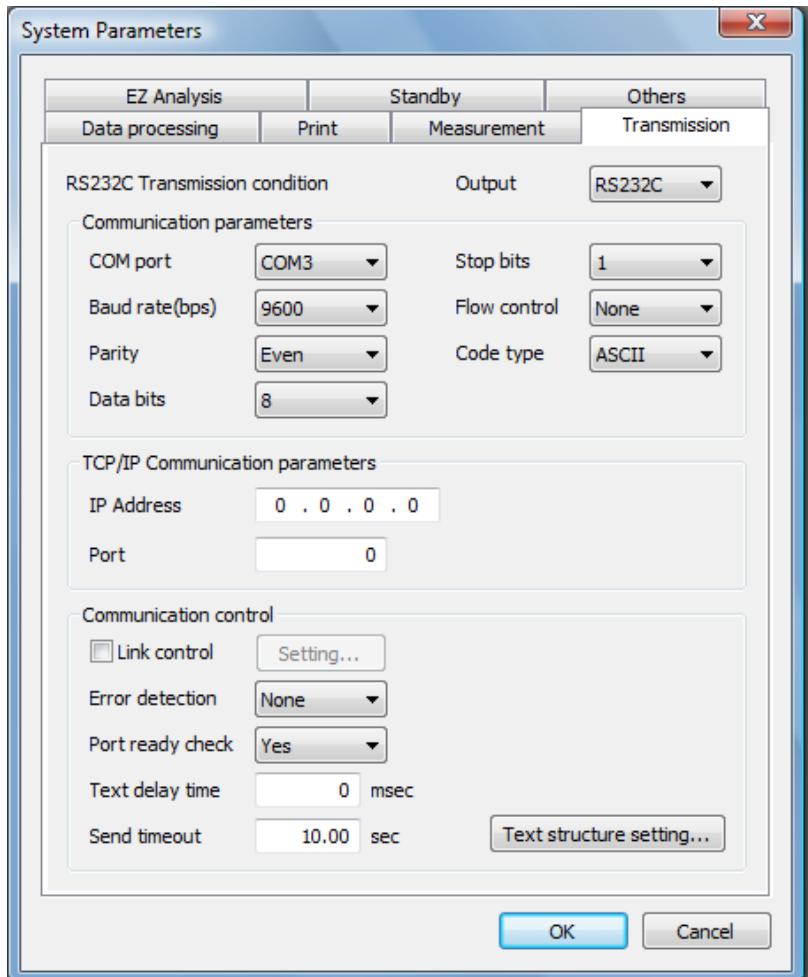


1. Right mouse clicking on individual results brings up a sub-menu where the user can transfer results to a file (see next slide)



If user selects “Transfer Data” (previous slide), several options are available:

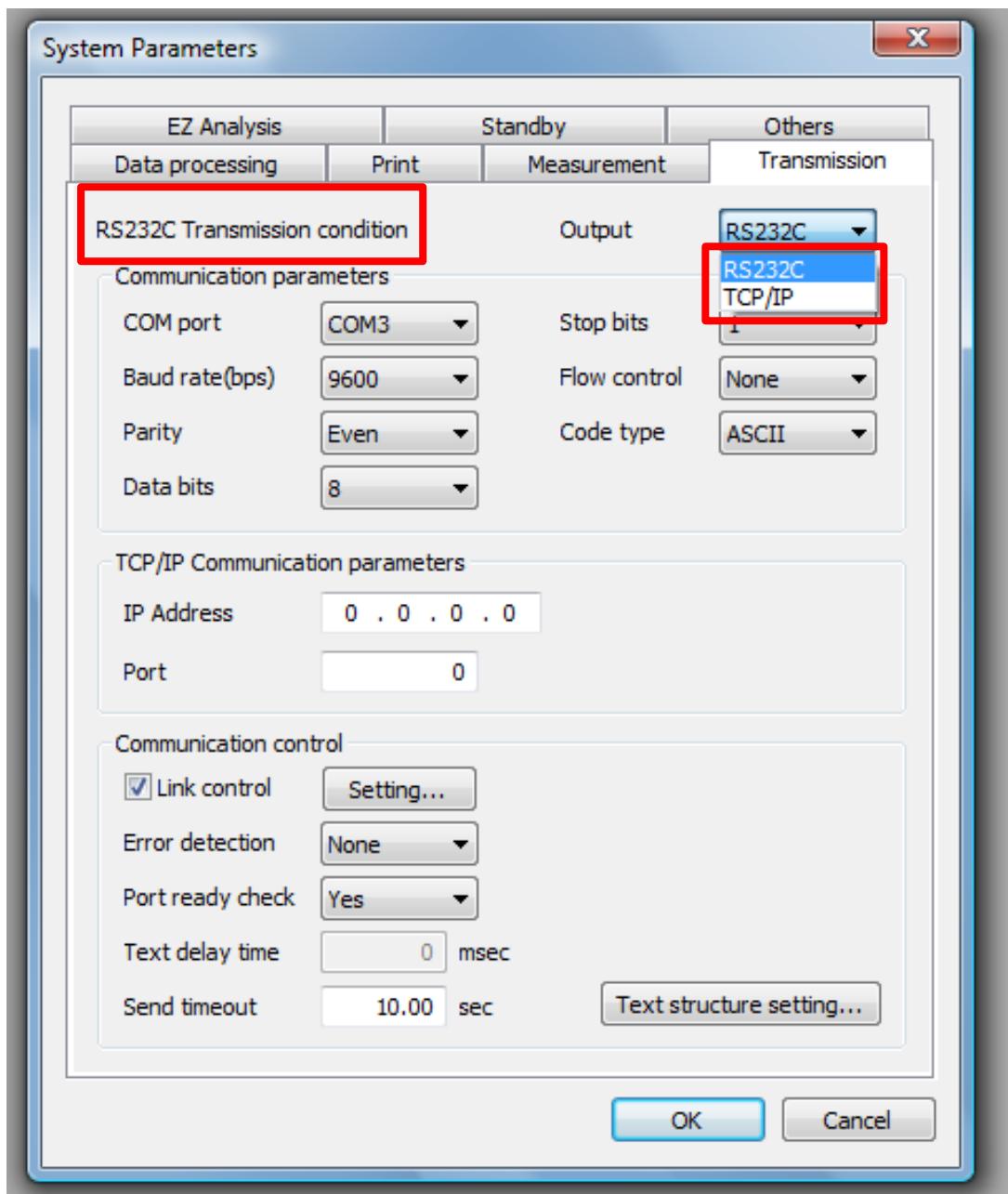
1. User defines “Text Format” as either “Transmission Format” (.txt) or “Spreadsheet Format” (.csv)
2. User defines “Trans. Method” as either “Disk Save” (local or network physical drive) or “RS232C Transmission” (results transferred to network server)
3. If “Disk Save” is chosen (in step #2), User defines destination folder and provides a filename

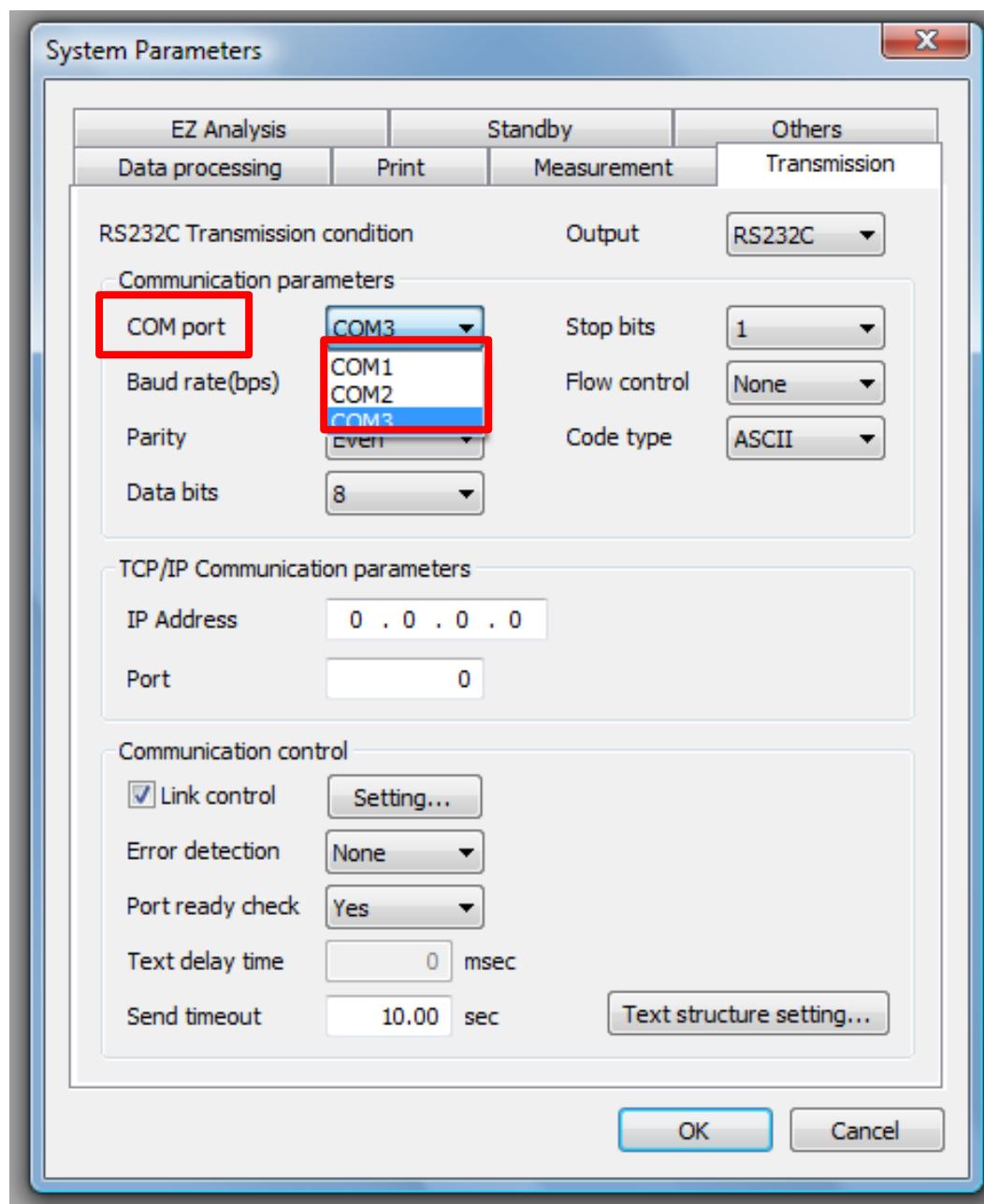


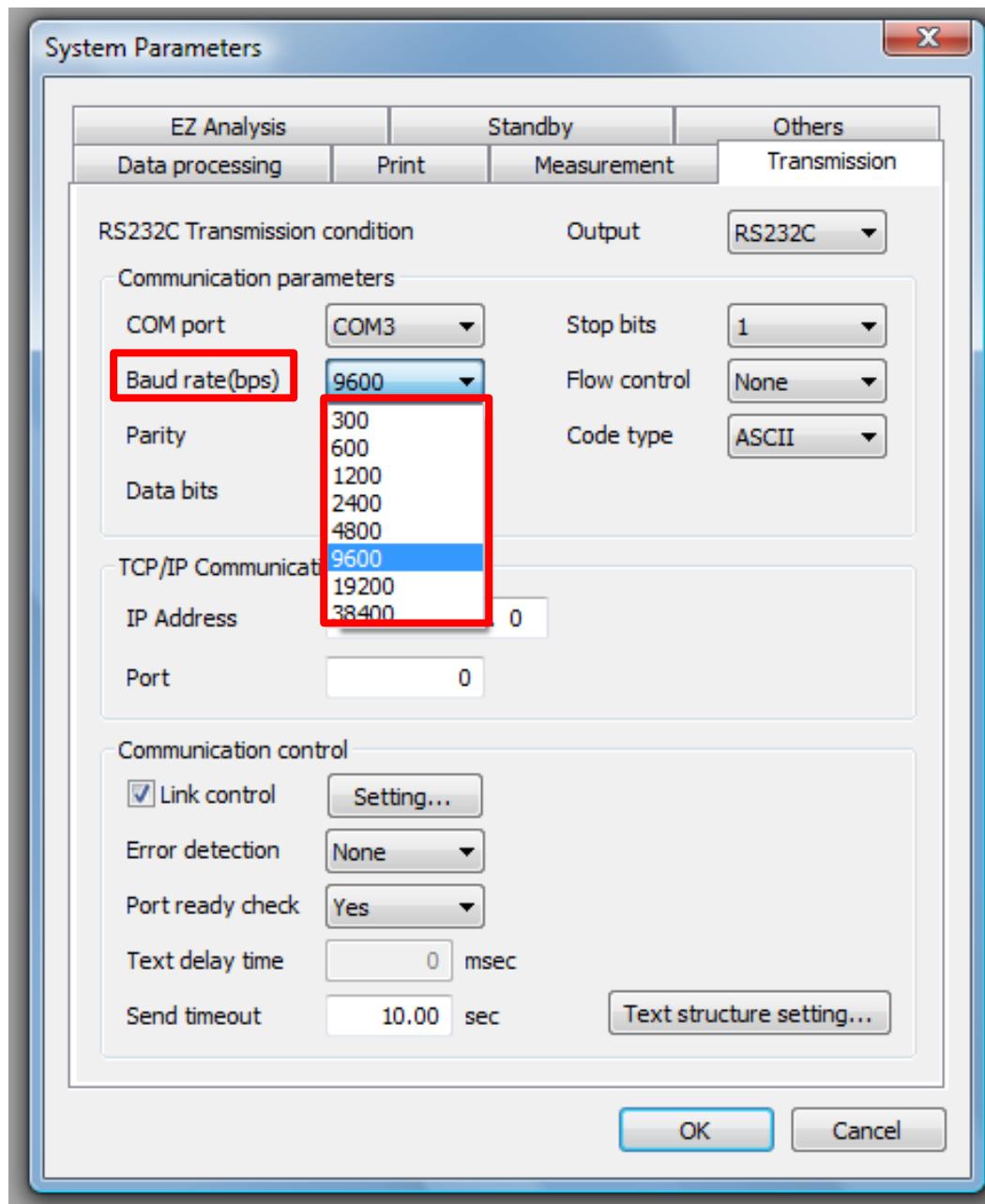
If user selects “RS232C Transmission” (previous slide) to transfer data to a network server, several communications protocols will need to be defined to establish successful communication between analyzer and the Host.

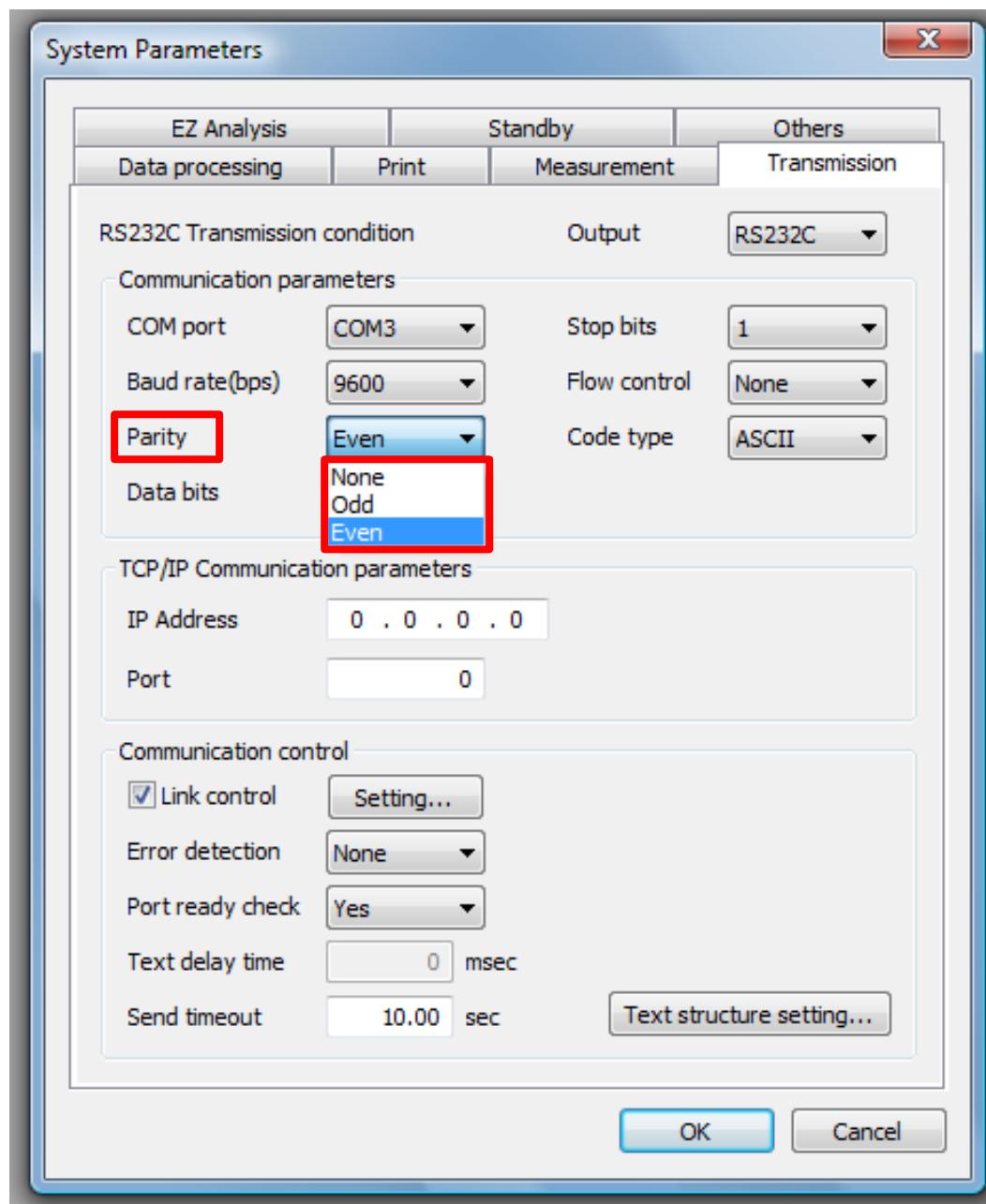
2. ON-LINE TRANSMISSION PROTOCOLS

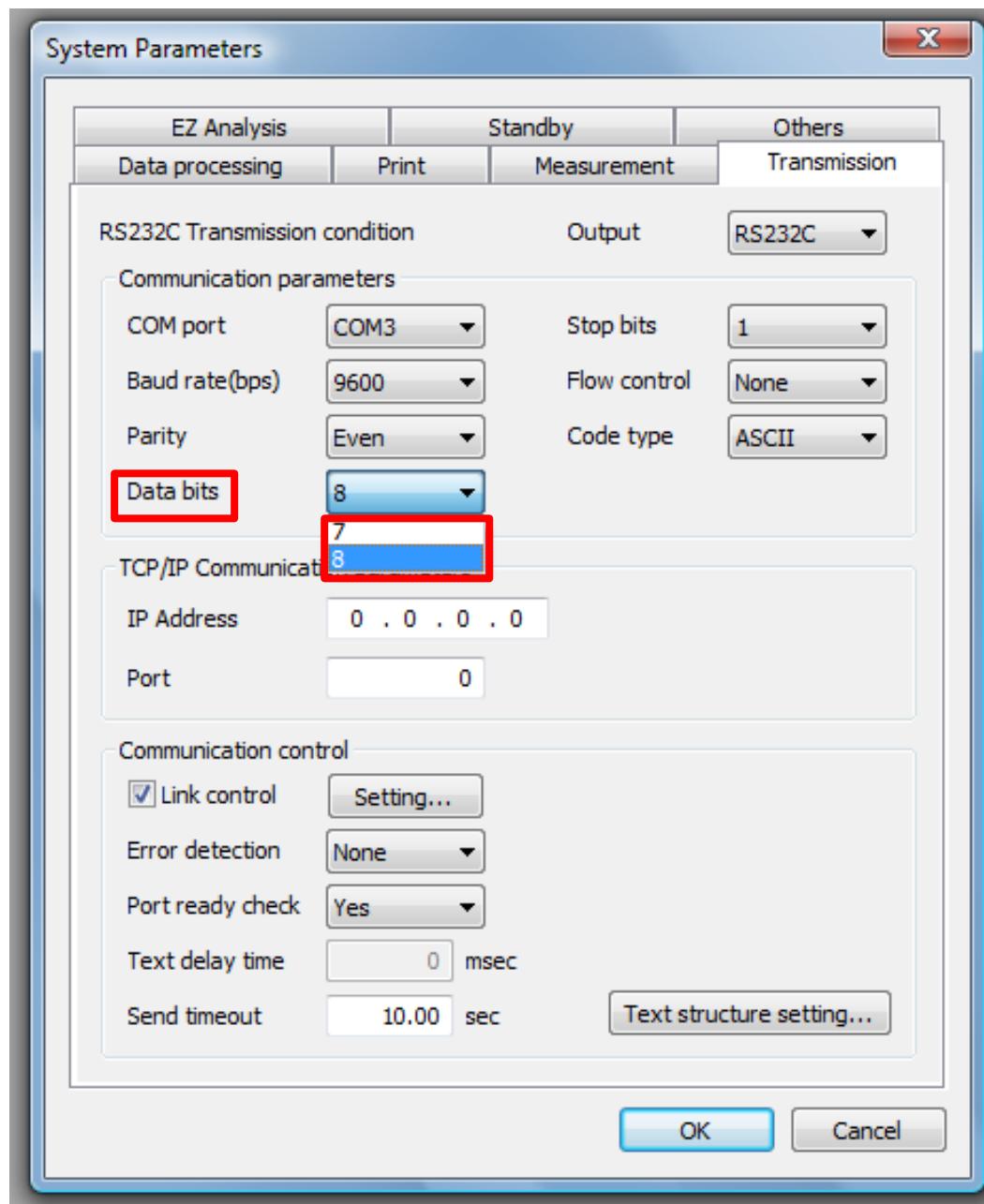
All settings are available for IT technician to setup for on-line transmission or connections with LIMS system.

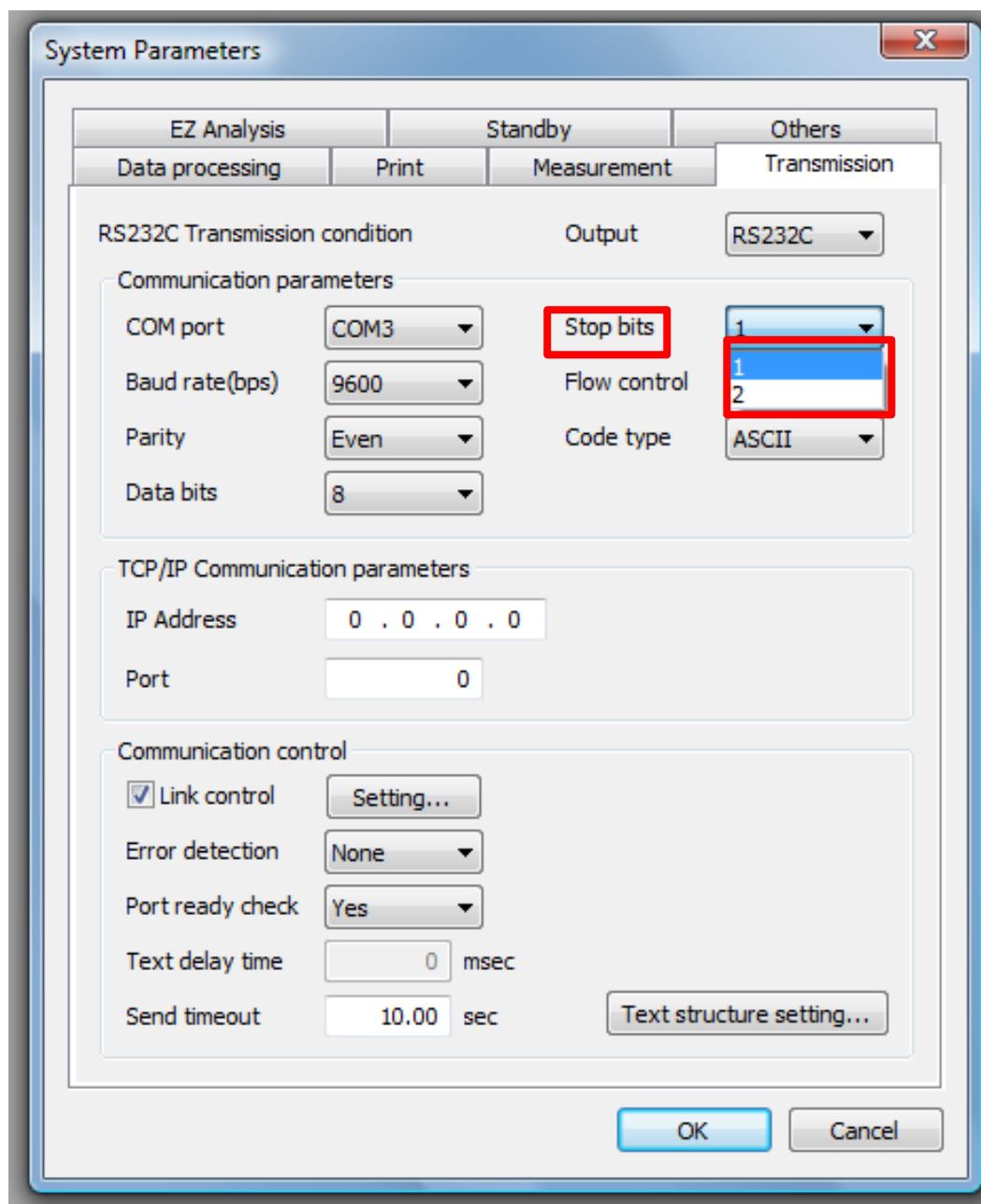


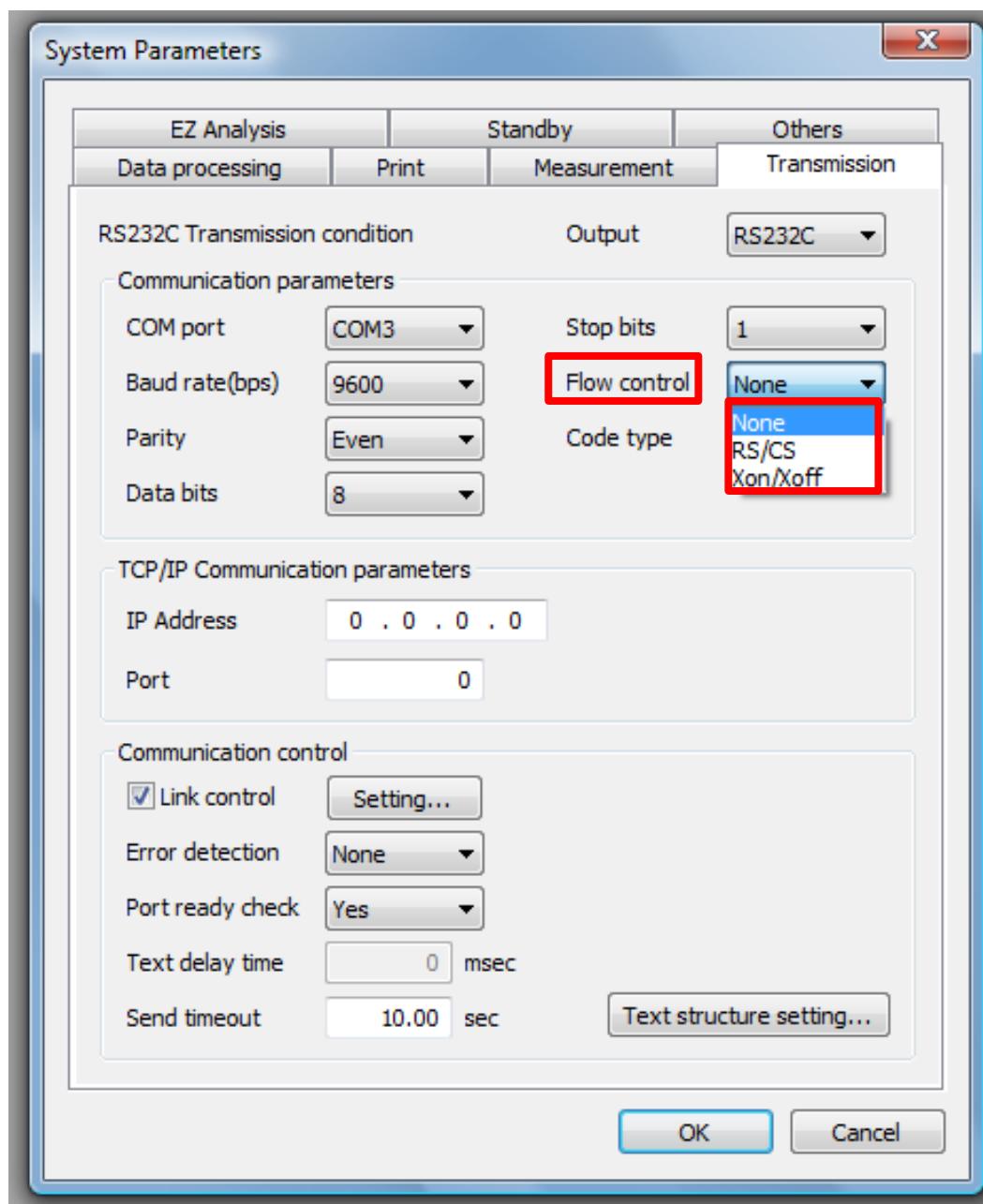


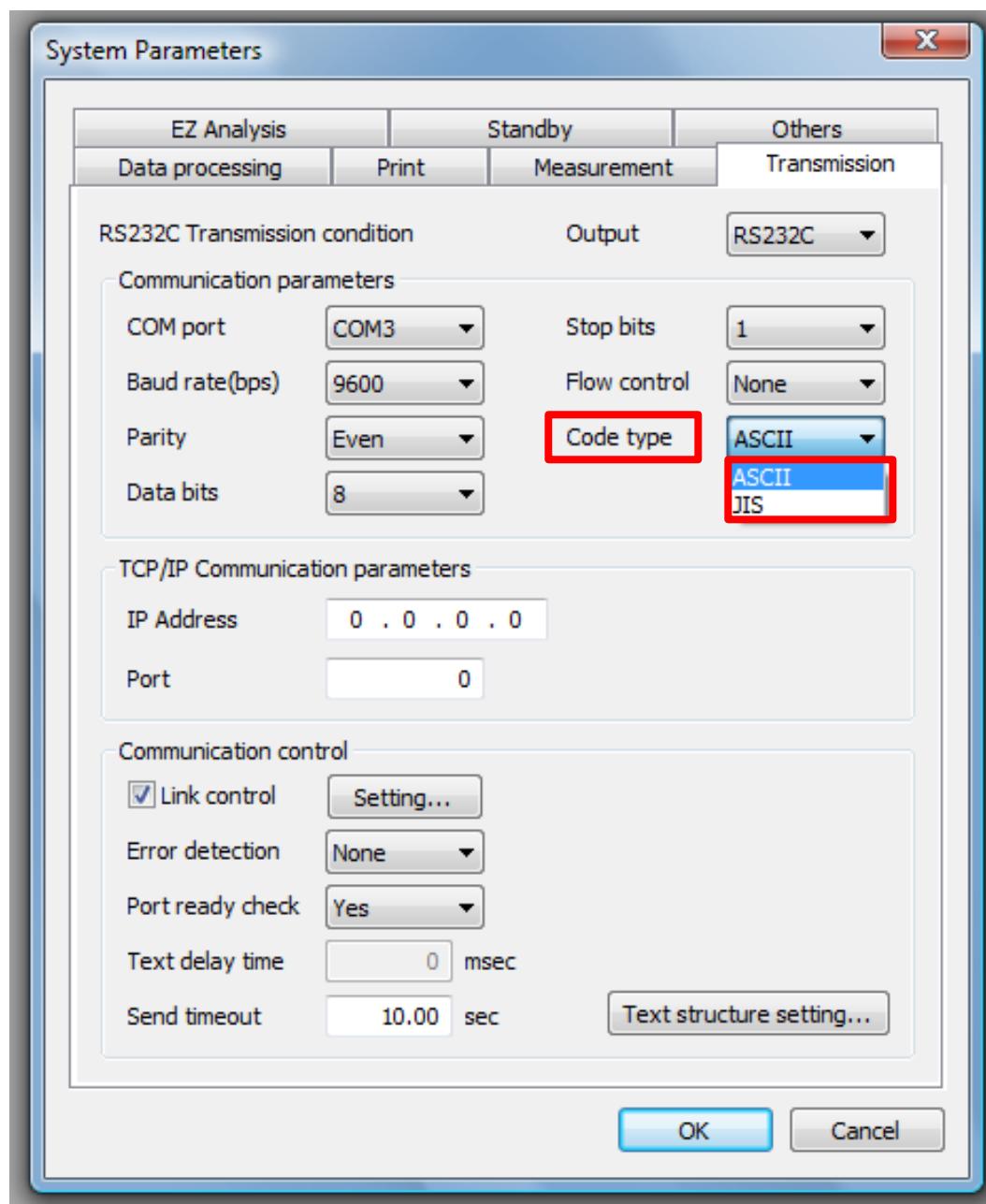


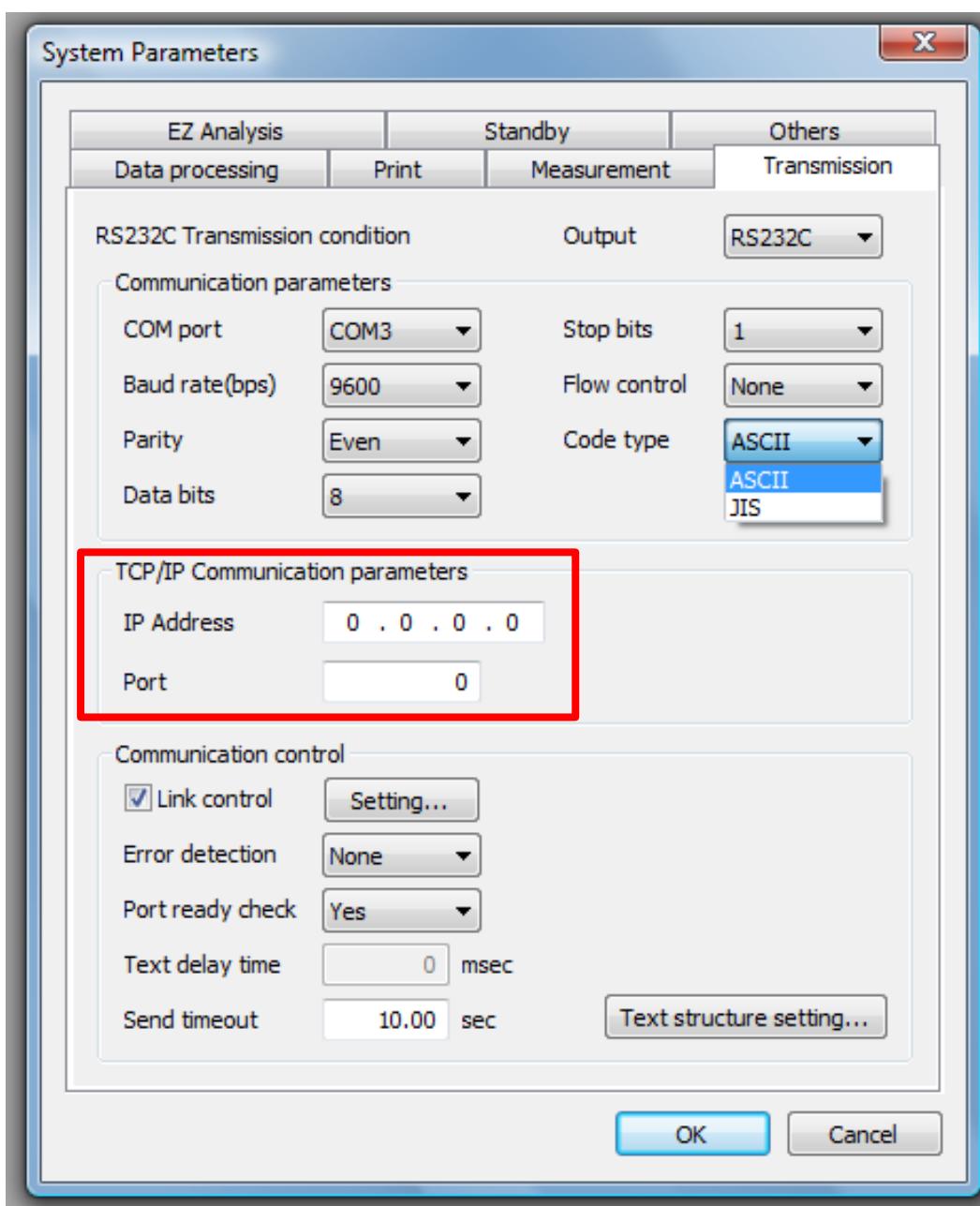




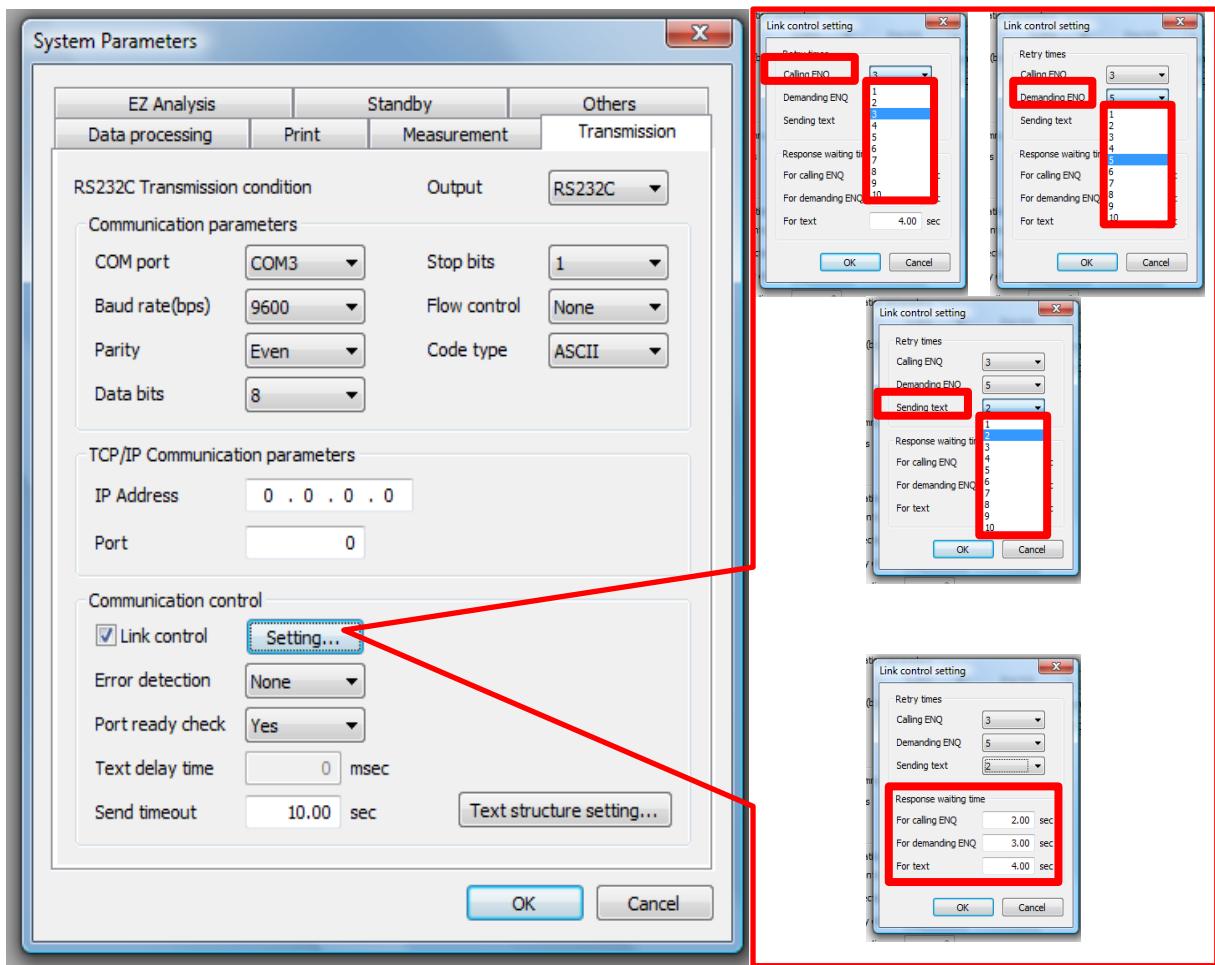


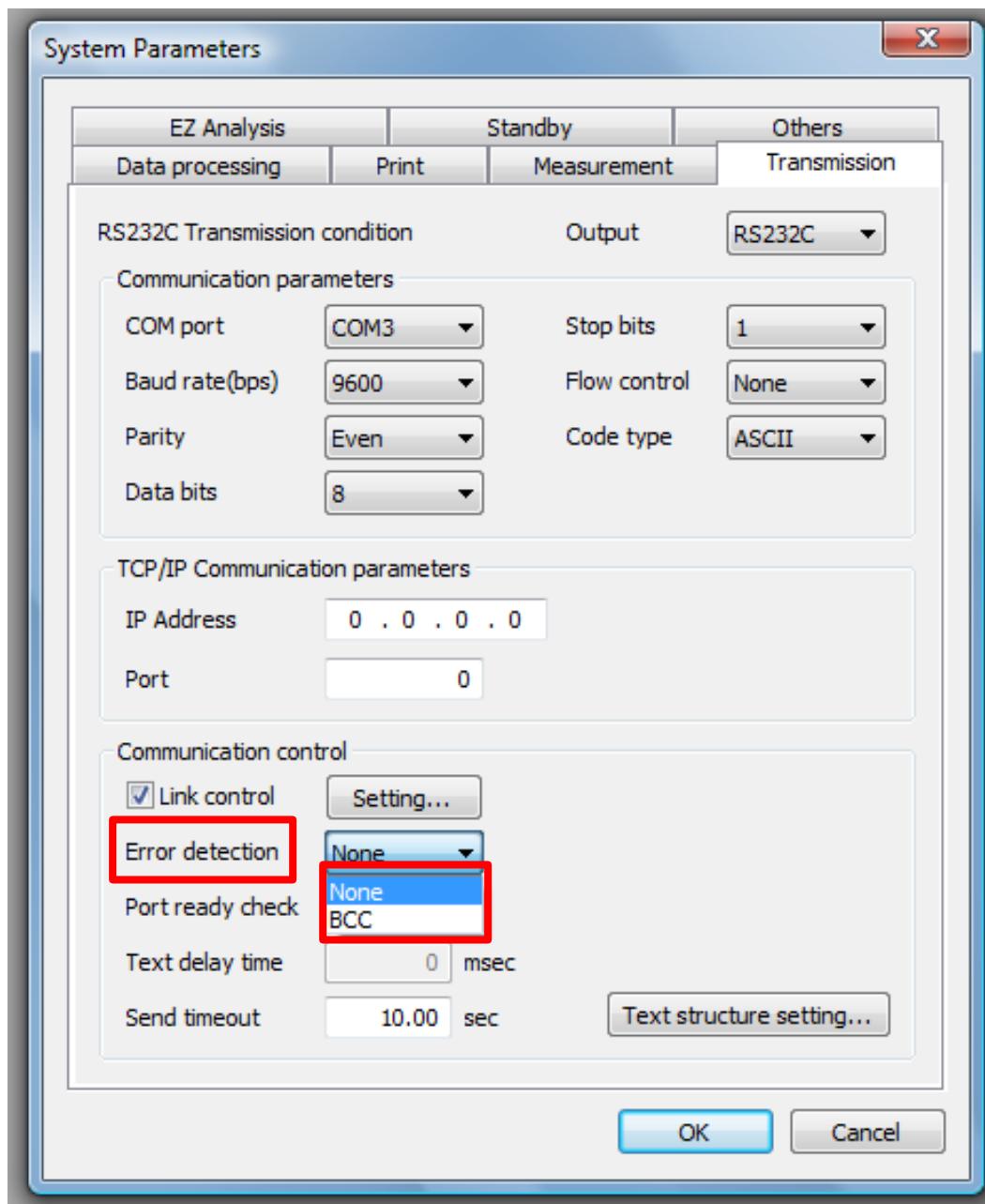


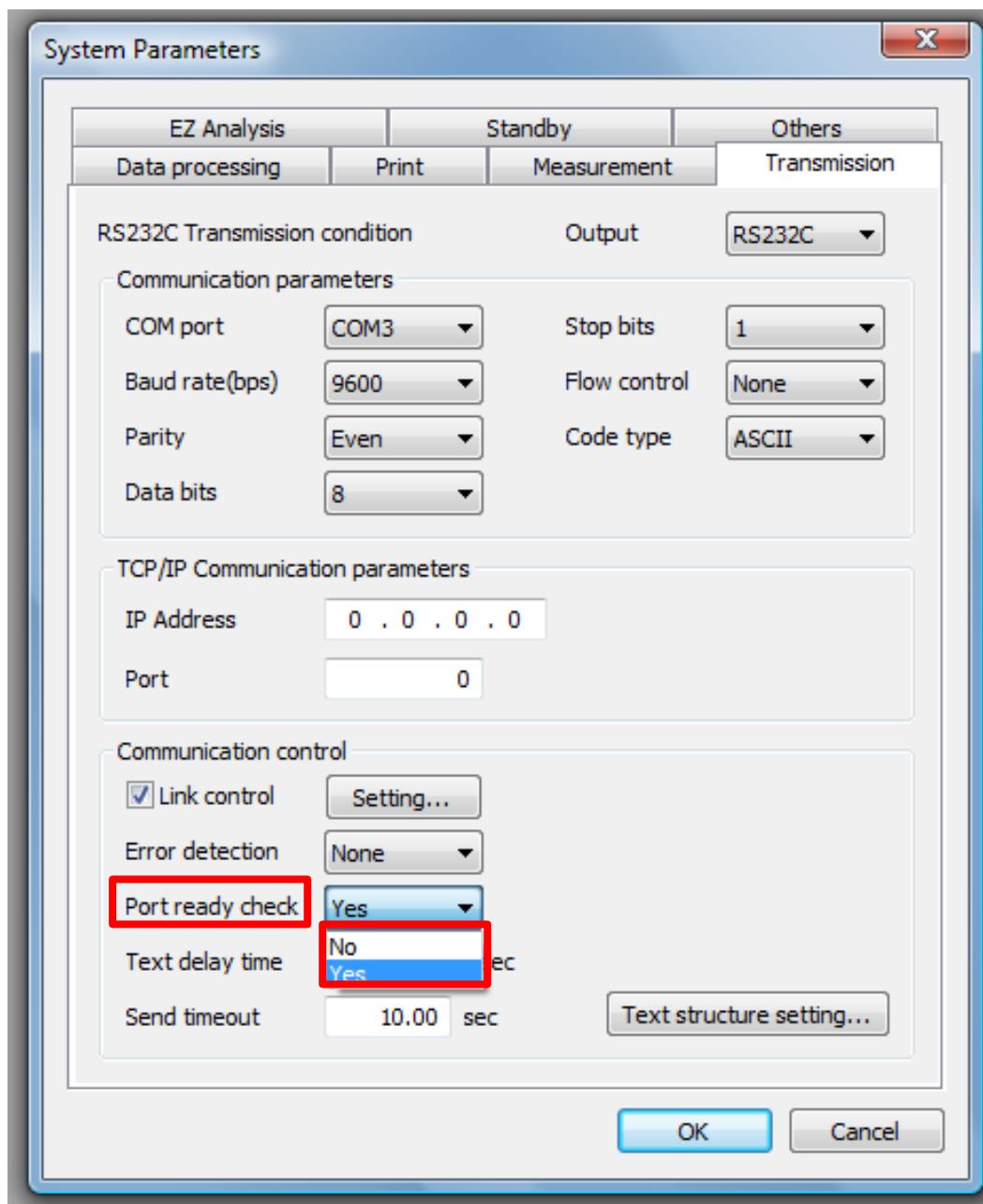


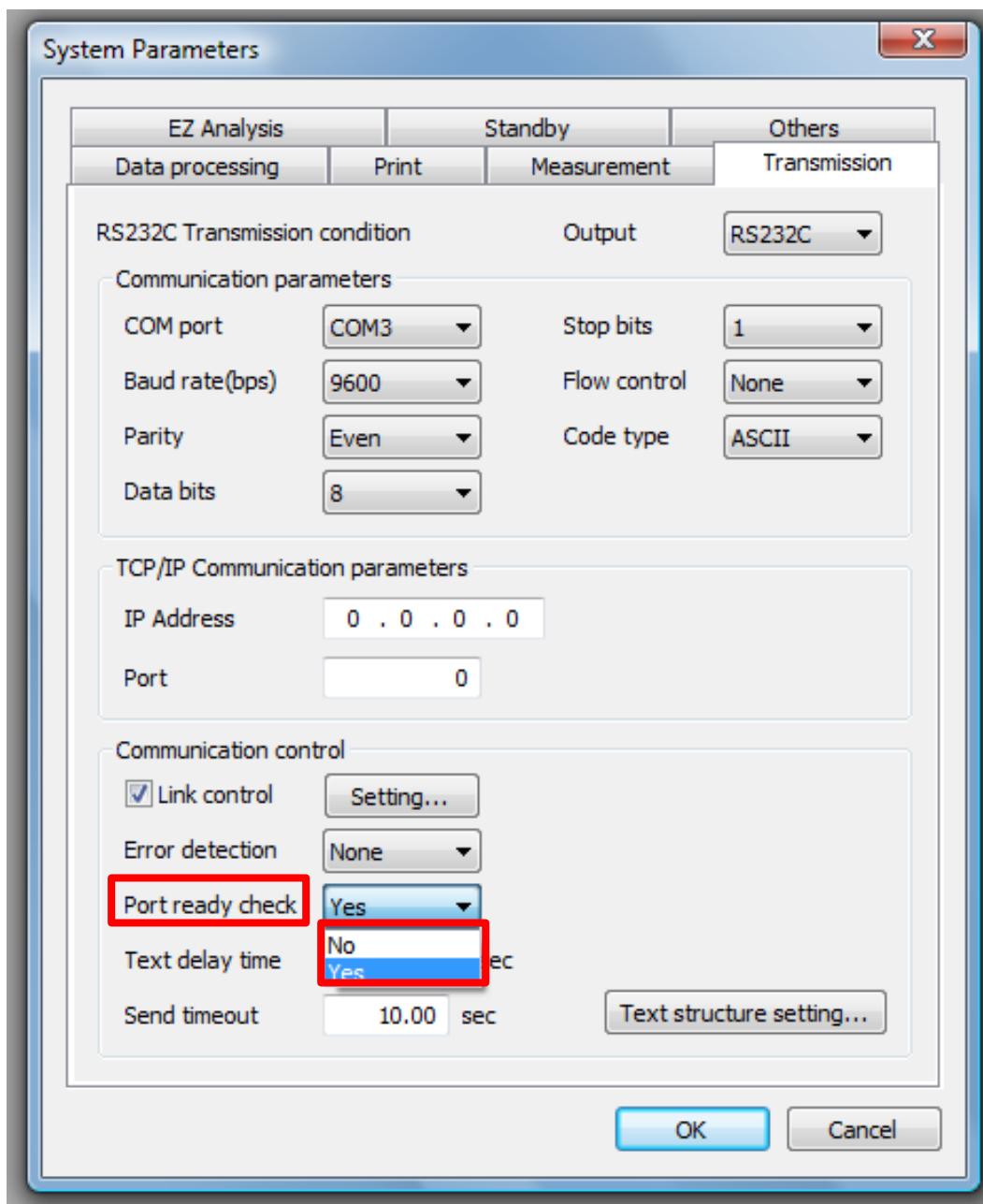


Appendix II

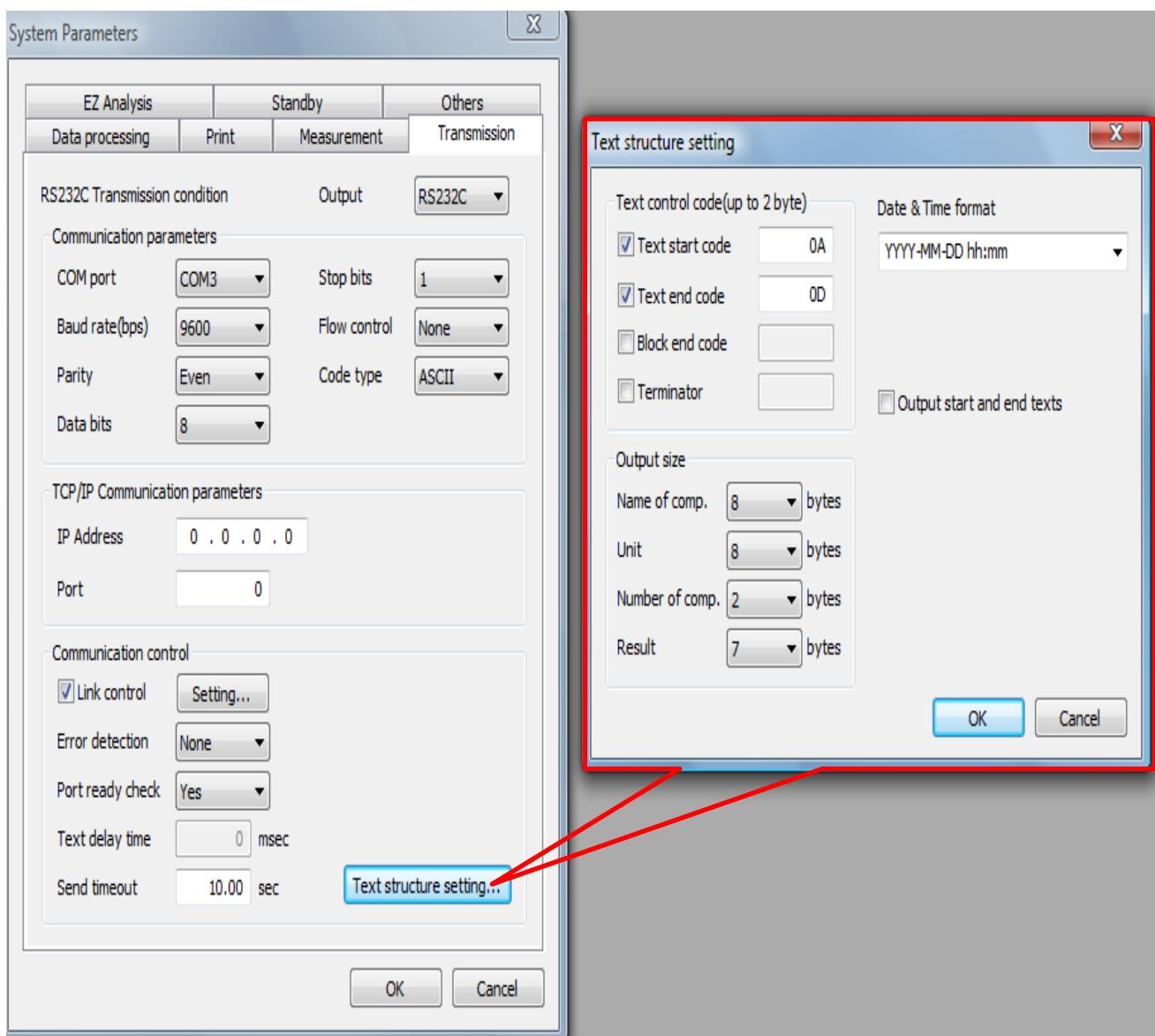


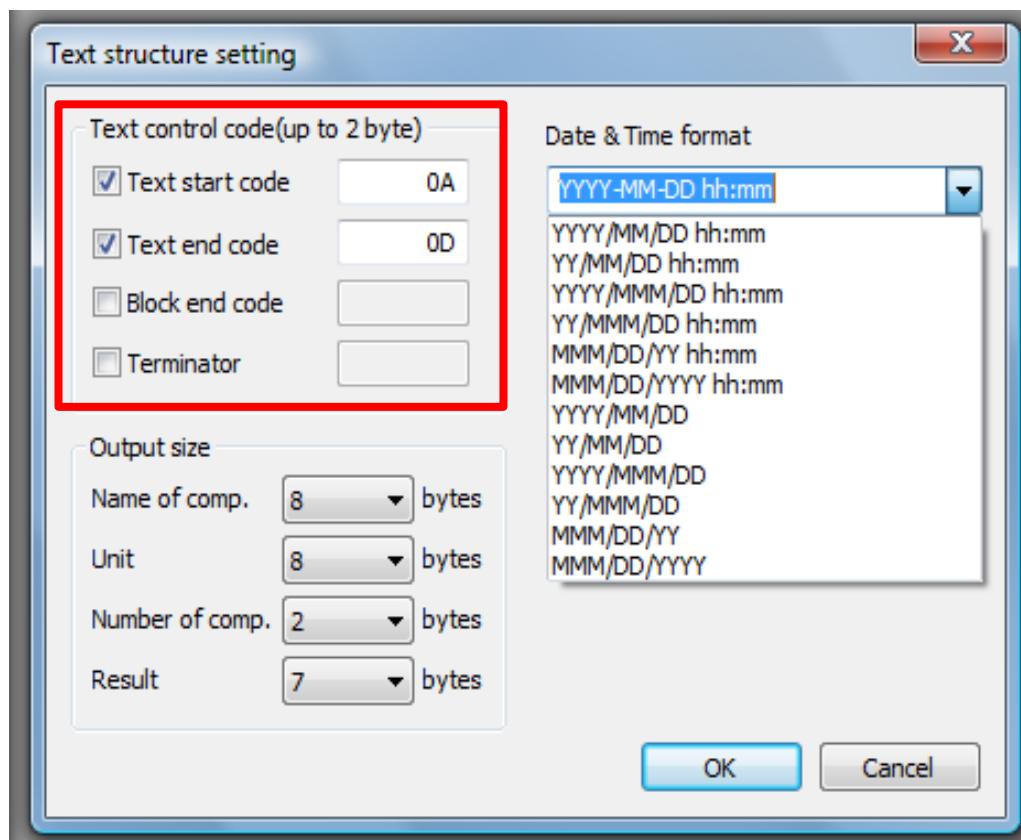
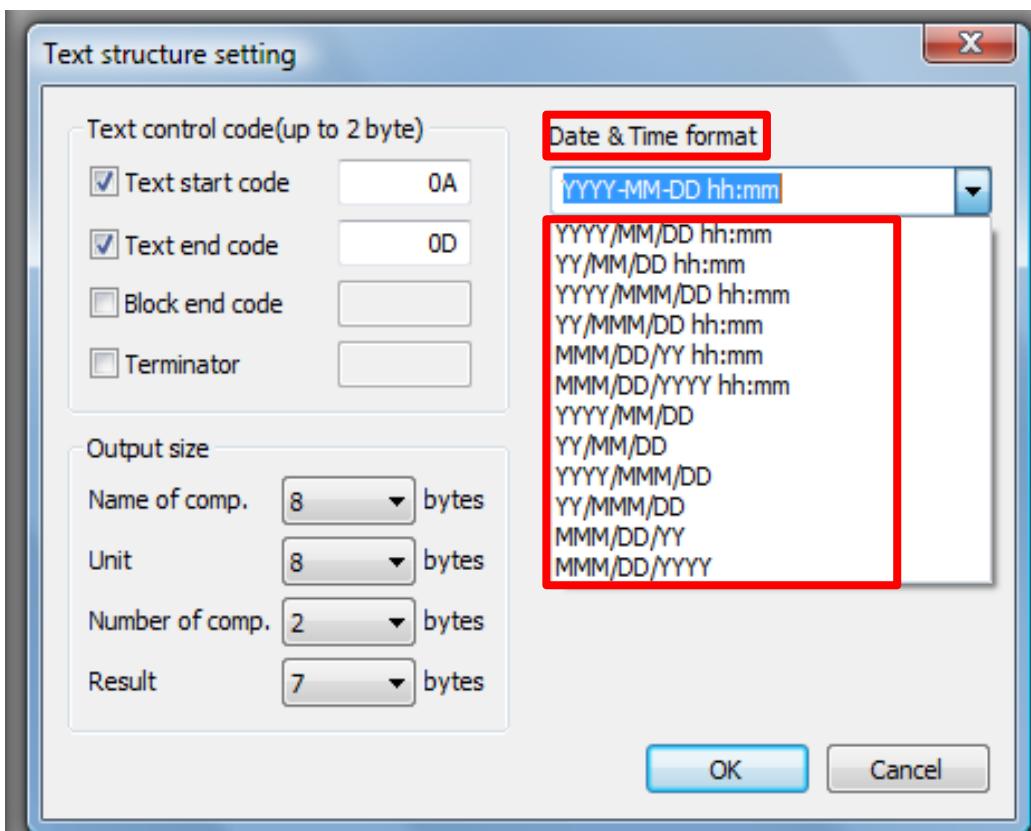


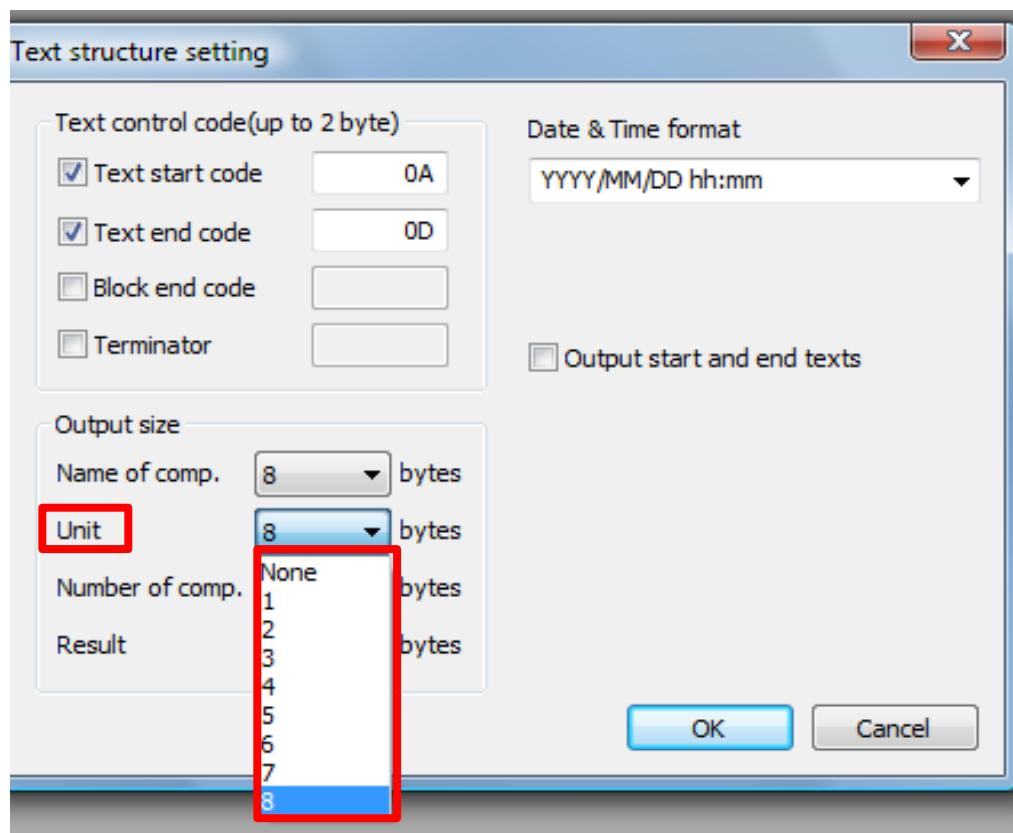
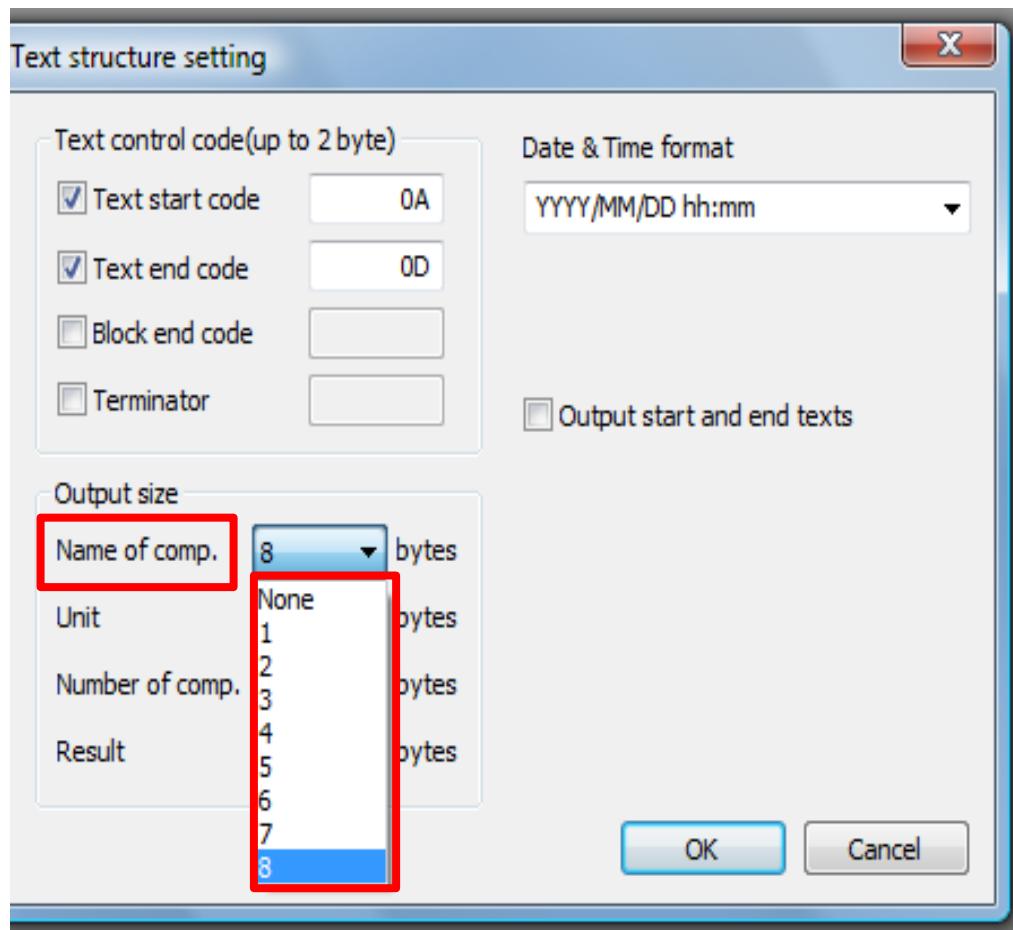


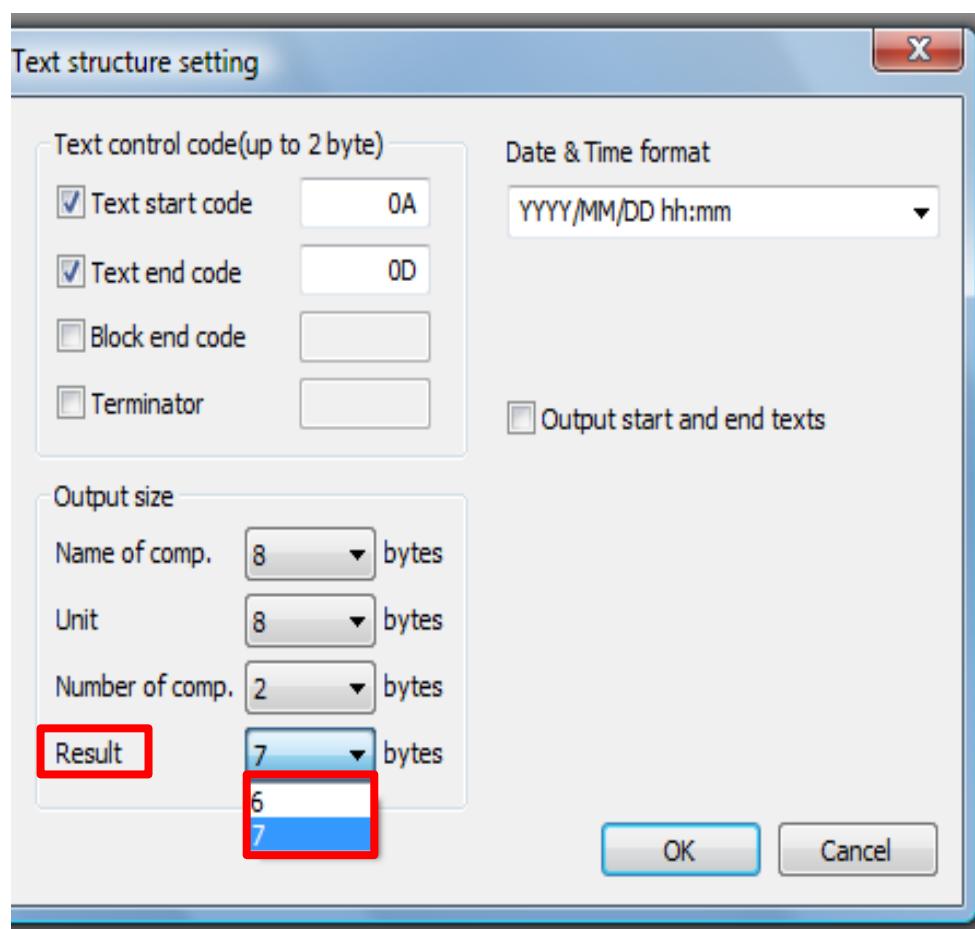
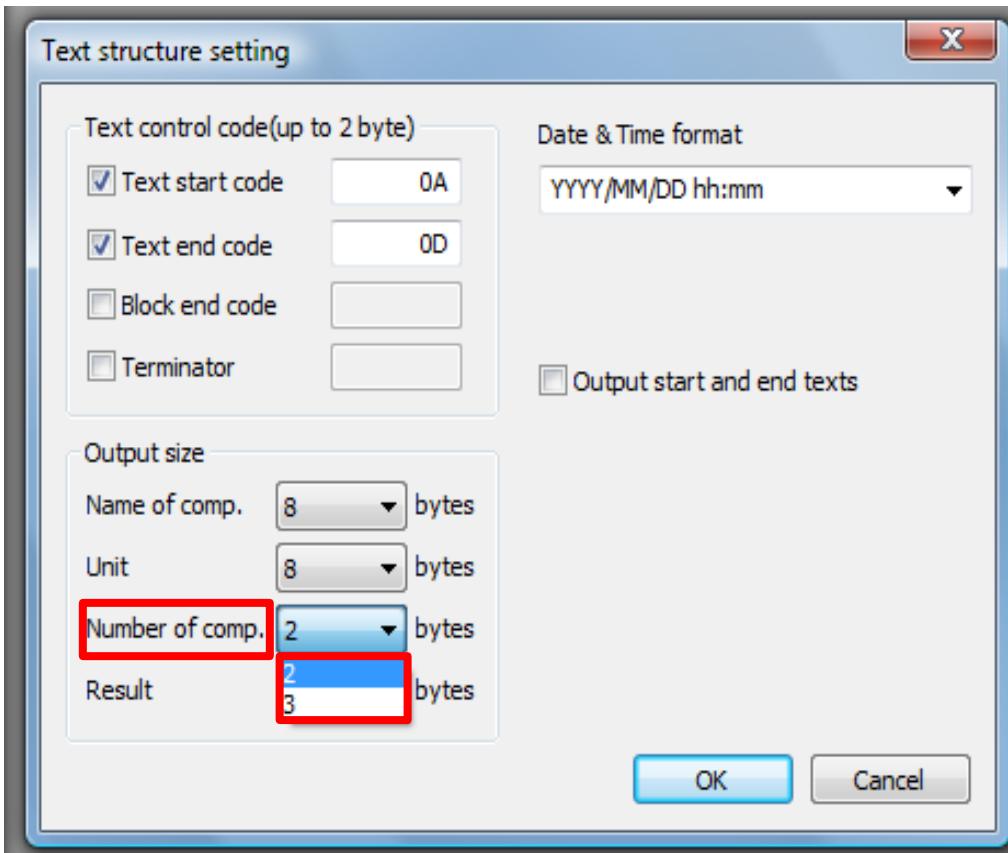


Appendix II









Appendix III. -- Advanced Drift Correction for Software Version NEX 6.16 and Later

NEX 6.16 includes the following new functions:

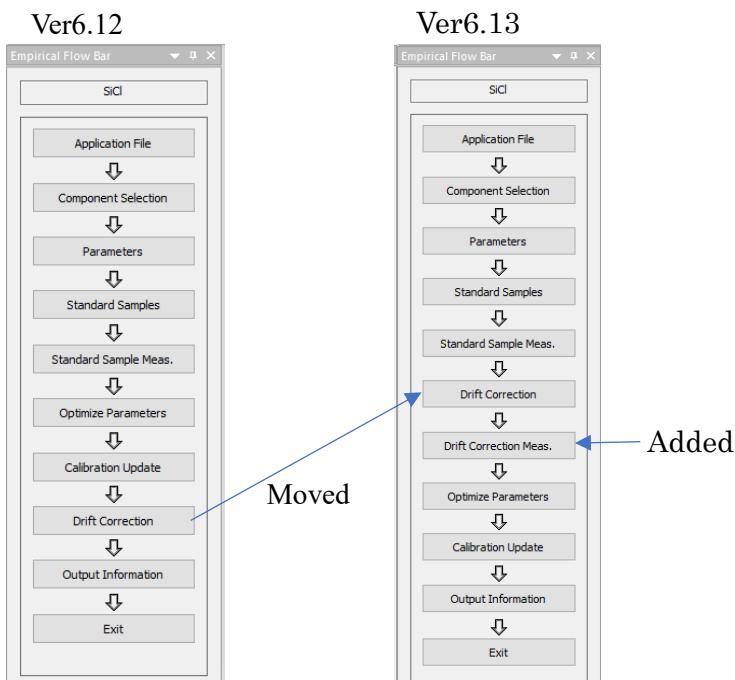
- A) Drift correction sample measurement in the EMP flow bar
- B) Drift correction line
- C) Drift correction record

A) Drift correction sample measurement in the EMP flow bar

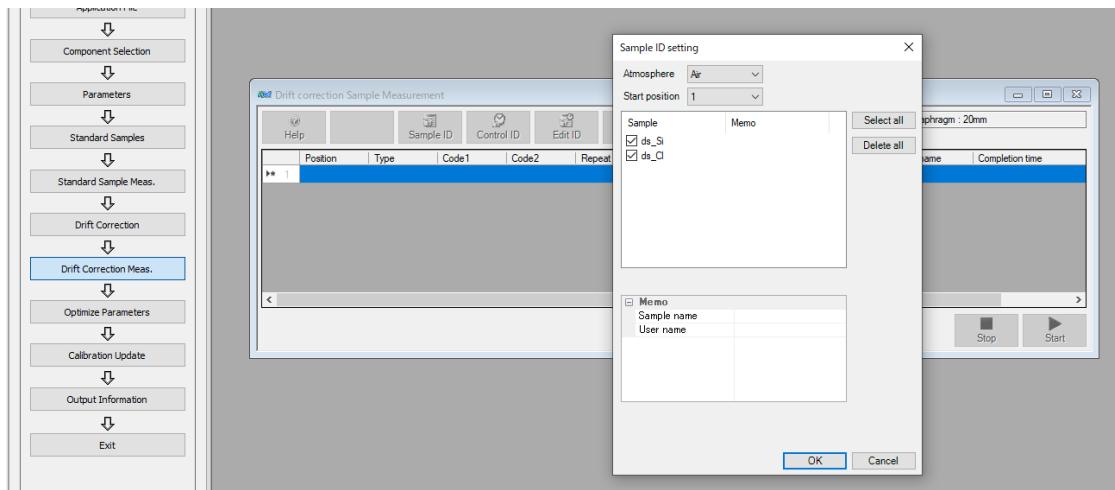
We changed an order of buttons in the EMP application flow bar as follows:

- Moved [Drift Correction] to after [Standard Sample Meas.].
- Added [Drift Correction Meas.] after [Drift Correction].

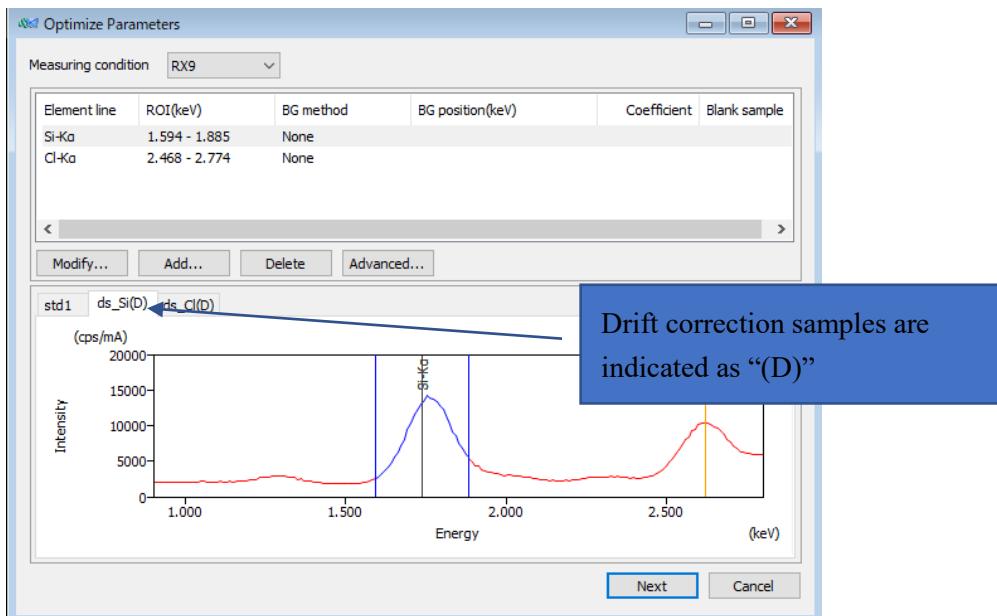
This feature enables users to measure the drift correction samples in the flow bar operation, while you need to separately run the analysis window to measure the drift correction samples in 6.12 or older version.



The Drift Correction Sample Measurement window is similar to the Standard sample measurement window.



After measuring the drift correction samples, you can check the spectrum of those samples in addition to that of standard samples in the [Optimize Parameters] window.

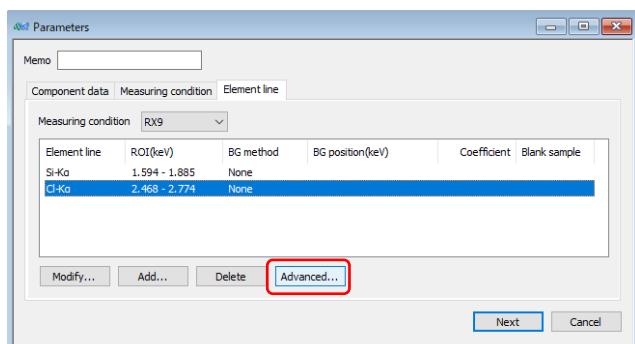


B) Drift correction line

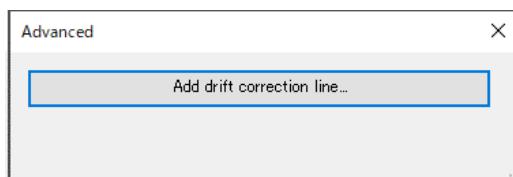
In NEX software prior to 6.16, ROI setting of the drift correction is always the same as that of the EMP element line. In 6.16 and later, you can specify ROI of the drift correction as “Drift correction line” that is different from ROI of the EMP element line. For example, you can specify ROI of Mo-LA (2.143 - 2.443keV) for the drift correction of the EMP element line Cl-KA (2.468 - 2.774keV).

You can specify the drift correction lines in the [Parameters] and [Optimize Parameters] in the EMP application flow bar. The procedure is as follows:

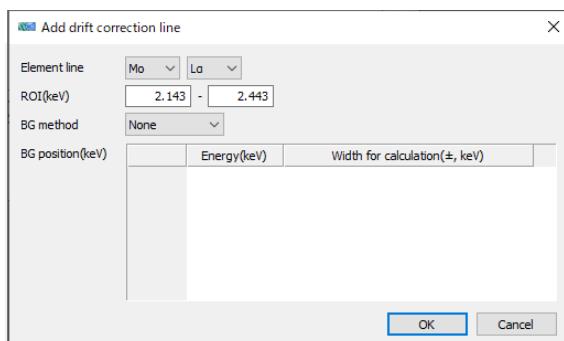
1. Select the element line in the [Element line] tab, and click [Advanced...] in the [Parameters] window.



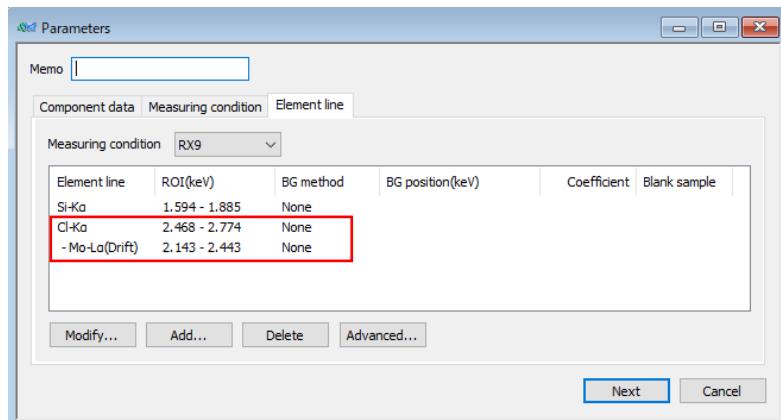
2. Click [Add drift correction line...].



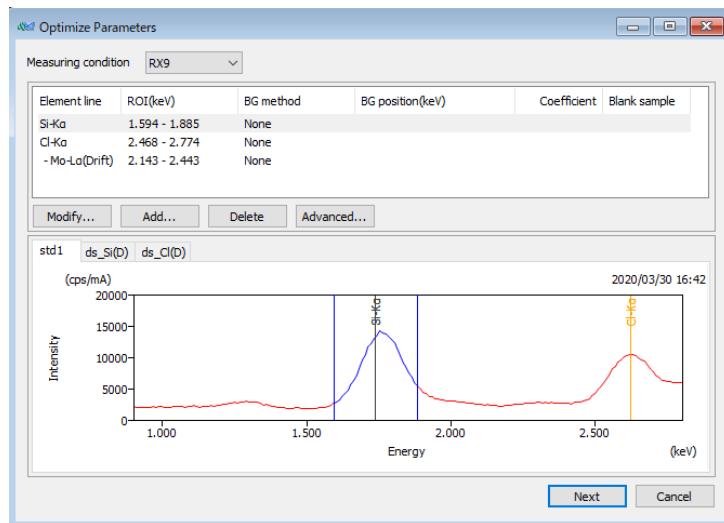
3. Specify the ROI setting for the drift correction line and press [OK].



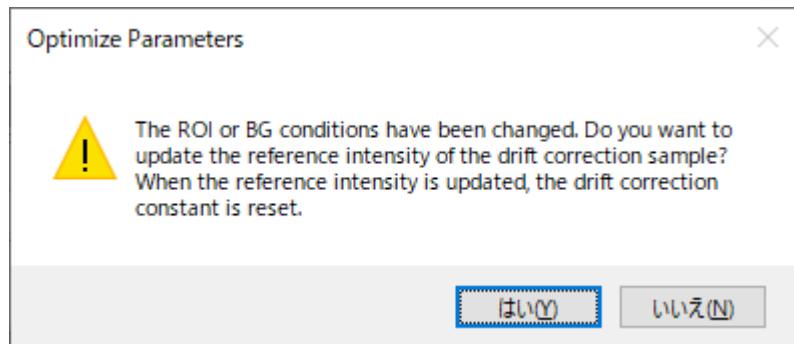
4. The specified drift correction line is indicated as “- Mo-LA(Drift)”. In the example below, the ROI of Mo-LA will be used for the drift correction of the empirical curve of Cl-KA



5. You can check the spectrum of the drift correction samples in the [Optimize parameters] window as already mentioned in “A) Drift correction sample measurement”. You can specify the drift correction line also in this window via [Advanced...] button.

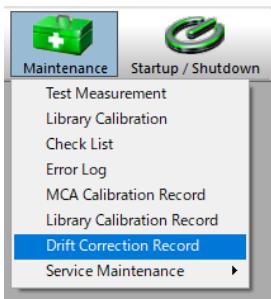


6. If you modified ROI setting of the drift correction line in the [Parameter] or [Optimize Parameters] window, the following message will be displayed when you press [Next] button.
 If [Yes], the reference intensity of the drift correction sample will be re-calculated using modified ROI setting and the coefficient of the drift correction (Alpha) will be reset to 1.0.
 If [No], the reference intensity and the coefficient of the drift correction are left as they are.

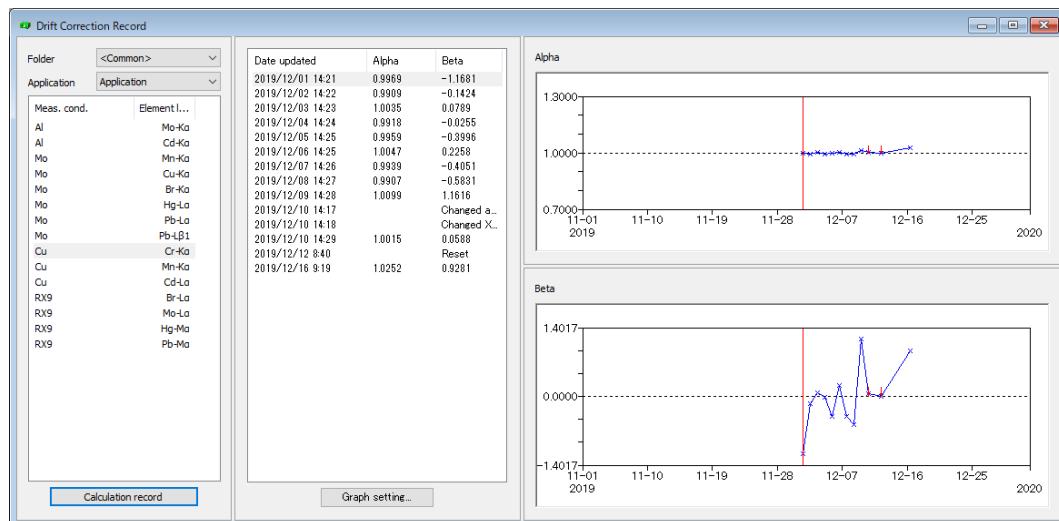


8. C) Drift Correction Record

“Drift Correction Record” is found in the Maintenance menu.



You can find the history of the drift correction coefficient for the specified element line of the specified application.



The [Calculation record] button will show the history of all drift corrections that is recorded every time you run the drift correction.

History		Detailed information				
Date updated	Result	Application	Meas. cond.	Element l...	Alpha	Beta
2019/12/11 9:07	Success	Cu	Cr-Ka		1.0035	0.0789
2019/12/11 9:07	Success					
2019/12/11 9:07	Success					
2019/12/11 9:07	Success					
2019/12/11 9:07	Success					
2019/12/01 14:21	Success					
2019/12/02 14:22	Success					
2019/12/03 14:23	Success					
2019/12/04 14:24	Success					
2019/12/05 14:25	Success					
2019/12/06 14:25	Success					
2019/12/07 14:26	Success					
2019/12/08 14:27	Success					
2019/12/09 14:28	Success					
2019/12/10 14:29	Success					
2019/12/12 8:40	Success					
2019/12/16 9:05	Success					
2019/12/16 9:19	Success					
2020/01/09 14:48	Success					
2020/01/09 14:49	Success					