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XM-17330/27330

QUALITATIVE ANALYSIS PROGRAM

IXM-17330/27330-1QL (608001)
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JEOL
Serving Advanced Technology

NOTATIONAL CONVENTIONS AND GLOSSARY

General notations

⚠ **WARNING :**	A potentially hazardous situation which, if not avoided, could result in death or serious injury.
⚠ **CAUTION :**	A potentially hazardous situation which, if not avoided, could result in minor injury or material damage. Material damage includes, but is not limited to, damage to related devices and facilities, and acquired data.
– CAUTION – :	Points where great care and attention is required when operating the device to avoid damage to the device itself.
✍ :	Additional points to be remembered regarding the operation.
☞ :	A reference to another section, chapter or manual.
1, **2**, **3** :	Numbers indicate a series of operations that achieve a task.
◆ :	A diamond indicates a single operation that achieves a task.
File:	The names of menus, or commands displayed on the screen, and those of buttons of the instrument, are denoted with **bold** letters.
File–Exit :	A command to be executed from a pulldown menu is denoted by linking the menu name and the command name with a dash (–). For example, **File–Exit** means to execute the **Exit** command by selecting it from the **File** menu.

Mouse operation

Mouse pointer:	An arrow-shaped mark displayed on the screen, which moves with the movement of the mouse. It is used to specify a menu item, command, parameter value, and other items. Its shape changes according to the situation.
Click:	To press and release the left mouse button.
Right-click:	To press and release the right mouse button.
Double-click:	To press and release the left mouse button twice quickly.
Drag:	To hold down the left mouse button while moving the mouse.

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1 GENERAL

This program enables you to measure spectra of unknown samples and to identify the constituent elements by using wavelength-dispersive X-ray spectrometers (WDS).
Up to 20 spectra can be acquired in a single measurement, and up to eight spectra can be displayed in real time on the monitor screen during spectrum acquisition. When the measurement has finished, elements are automatically identified, with element names attached to the peak positions, based upon the knowledge base of experienced analysis specialists. The program permits displaying the KLM markers, manual identification of peaks, semi-quantitative analysis for designated elements, and printing the results of identifications.

2 SPECIFICATIONS

Spectrum measurement:	Up to 20 spectra by asynchronous concurrent spectrometer driving/ measurement
Number of measurement points per spectrum:	10 to 10,000
Spectrometer measurement step interval:	1 to 1,000 μm
X-ray counting time:	1 to 100,000 ms per spectrometer step
Number of preset measurement points per sample:	1 to 10,000
Number of accumulations:	1 to 100
Realtime display during measurement:	Possible
Automatic element identification during measurement:	Possible
Spectrum display functions:	Display of up to 8 spectra Spectrum expansion/reduction Selectable forms of spectrum display (Vector, Dot and Bar) Selectable units of spectrum display (mm, Å and keV)
KLM markers:	Can be displayed
Spectrum peak identification:	Possible
Off-line element identification:	Possible
Spectrum calculations:	Smoothing, second differentiation, background removal, addition/subtraction/ multiplication/division of constants, addition/subtraction of spectra, spectrum shift
Chemical shift analysis:	Possible
Trace element analysis:	Possible
Spectrum search:	Discovering similar spectra is possible.

3 PROGRAM STRUCTURE

This program has a tree structure as shown below. Clicking the mouse on a menu item executes the item or displays menu items at a lower level in the hierarchy, from which you can select the desired item.

- Analysis
  - Qualitative Analysis
    - Sample
    - Measurement
      - Spectrometer Condition
      - On-line Semi-Quant
      - EOS Condition
      - Stage Condition
      - Condition Load
      - Condition Store
      - Print-out Condition
      - Additional Function
      - Survey Measurement
      - Preset Measurement
  - Other Analysis Menus

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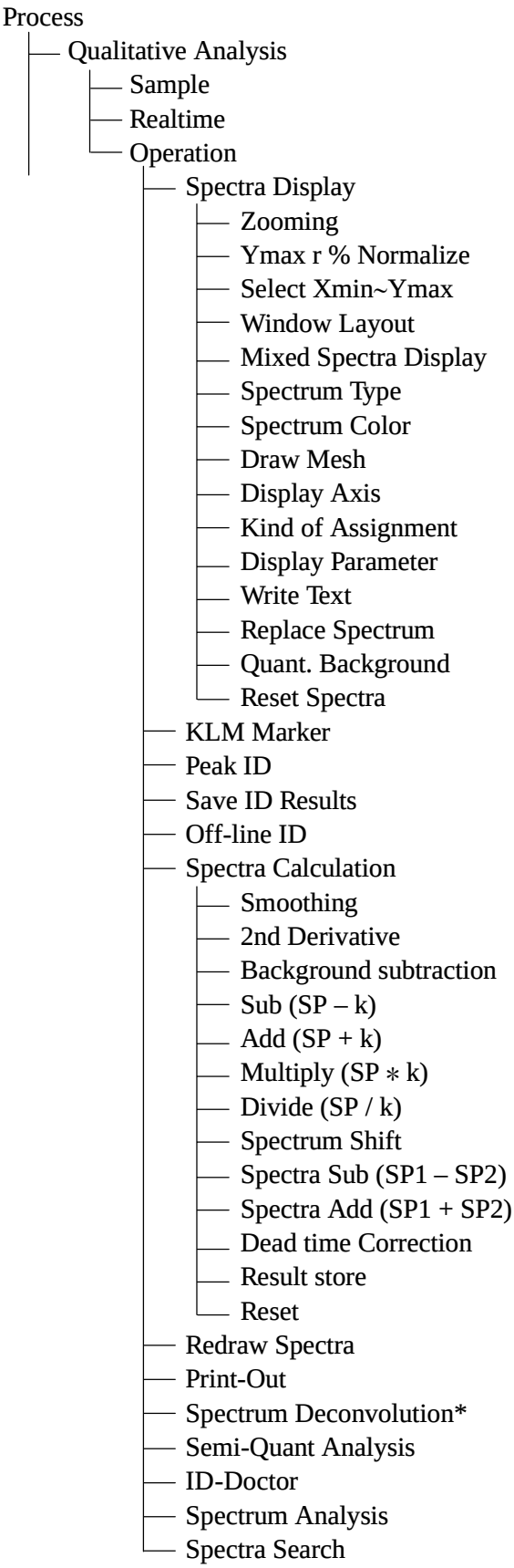
Process
Qualitative Analysis
Sample
Realtime
Operation
Spectra Display
Zooming
Ymax r % Normalize
Select Xmin~Ymax
Window Layout
Mixed Spectra Display
Spectrum Type
Spectrum Color
Draw Mesh
Display Axis
Kind of Assignment
Display Parameter
Write Text
Replace Spectrum
Quant. Background
Reset Spectra
KLM Marker
Peak ID
Save ID Results
Off-line ID
Spectra Calculation
Smoothing
2nd Derivative
Background subtraction
Sub (SP – k)
Add (SP + k)
Multiply (SP ∗ k)
Divide (SP / k)
Spectrum Shift
Spectra Sub (SP1 – SP2)
Spectra Add (SP1 + SP2)
Dead time Correction
Result store
Reset
Redraw Spectra
Print-Out
Spectrum Deconvolution*
Semi-Quant Analysis
ID-Doctor
Spectrum Analysis
Spectra Search

* Optional.

4 OPERATION

This chapter describes the procedure for qualitative analysis. The procedure is divided into three parts: measurement (analysis data acquisition), real time display (monitoring during measurement) and processing (analysis data processing).

4.1 Preparation for Measurement

This section explains the general procedure for measurement.
First, set sample names for the data to be stored. Then, set Spectrometer and EOS conditions. Set the point to be measured and execute measurement. These conditions can be saved in files, and can be recalled later for measurement. Also, element identification as well as determination of composition are possible if semi-quantitative analysis is specified at the time of measurement.

The following procedure is used for opening the main window for measurement.

1. Open the EPMA Main Menu on the computer display and then click on the **Analysis** icon to display the pull-down menu.

☞ Refer to the instruction manual of the microanalyzer main unit to learn how to open the EPMA Main Menu.

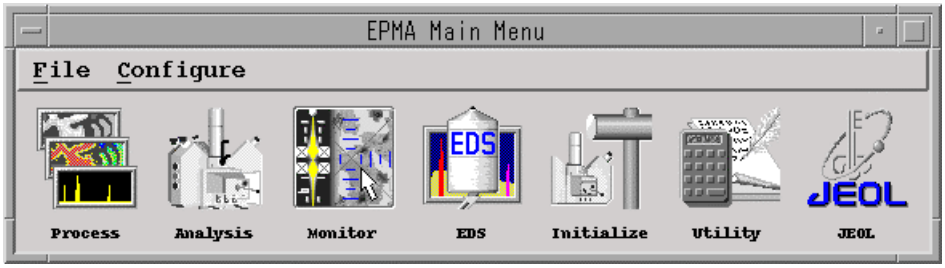

Fig. 1 EPMA Main Menu

2. Select **Qualitative Analysis**.

The Qualitative Analysis function window opens. Proceed to the following sections.

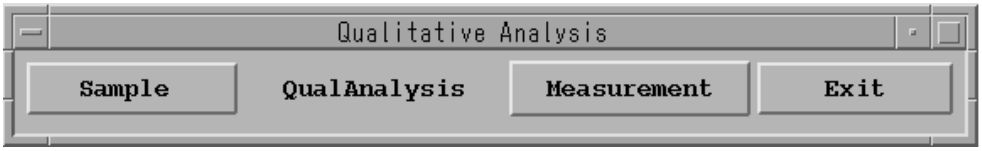

Fig. 1a Qualitative Analysis function window

4.1.1 Setting group and sample names

Measurement is carried out under the specified sample name, while the data processing and data backup take place after measurement for every sample. Up to 10,000 data can be stored for each sample name. The group name is the name containing a group of samples, and it will be convenient to name after the property of a series of samples or the operator name for easier arrangement and filing.

1. Click on the **Sample** button of the Qualitative Analysis function window.

The Select Sample window opens as shown in Fig. 2.

This window displays the list of the sample names entered previously, measurement dates and methods of analysis.

The methods of analysis are Qlw: qualitative analysis, Qnt: quantitative analysis, Lin: line analysis, Map: map analysis, and Eds: EDS analysis.

The amount of disk space (KB) in use and the amount of free space at present are shown in the window.

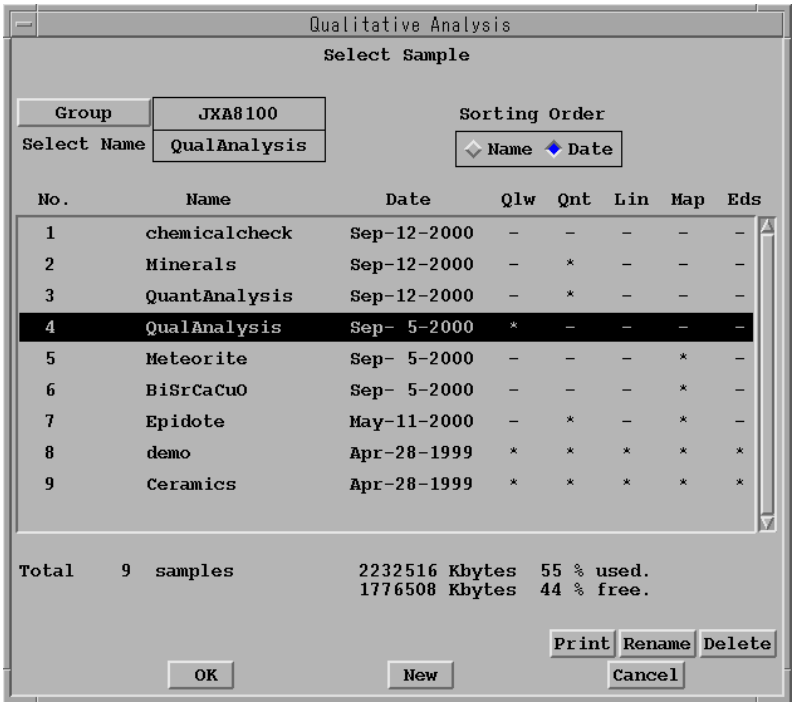

Fig. 2 Select Sample window

2. Confirm the Group name in the top left corner of the Select Sample window.

If you want to select a new group or an existing group name, click on the **Group** button to open the Select Group window; then select the desired group name, or after clicking on the **New** button, enter a new group name. The maximum length is 14 characters.

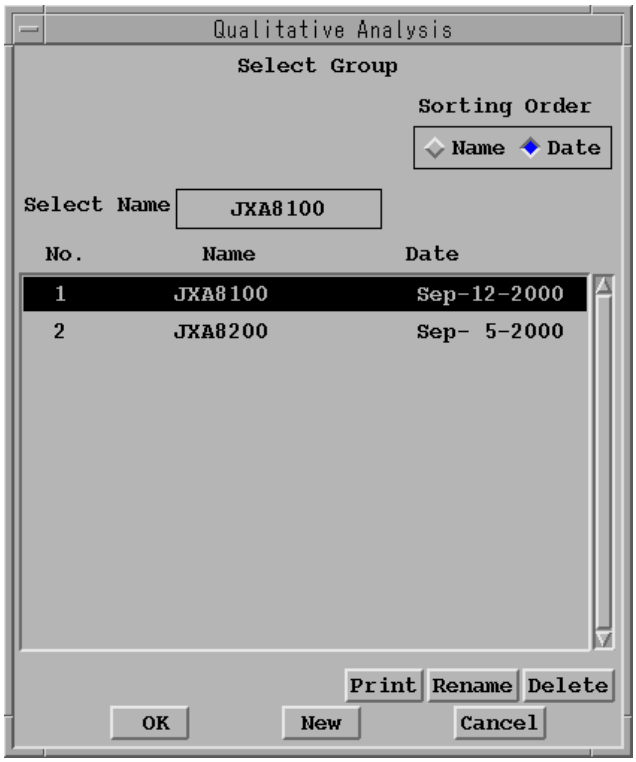

Fig. 3 Select Group window

3. To use a sample name entered previously, click on the desired sample name in the list of sample names, and then click on the **OK** button. To enter a new sample name, click on the **New** button and input the new sample name in the input box. The maximum length is 14 characters.

The remaining buttons in the Select Sample window and the Select Group window have the following functions.

Button	Function
New	After clicking on the **New** button, you can enter new Group names and Sample names. The maximum length is 14 characters. You can use alphanumerics, +, –, _, =, and . (the period cannot be the first character). When a new Group name is recorded, a Sample name also must be recorded using **New** at the same time.
Rename	After clicking on the **Rename** button, you can enter new Group names and Sample names.
Print	Click on the **Print** button in each window to print the list of Group names and Sample names.
Delete	To delete the Group names and Sample names that have been just recorded, specify them in each window and click on the **Delete** button. To delete Group names and Sample names that have been already used for measurement, delete them by selecting **Utility–File Utility** from the EPMA Main Menu.

Button	Function
Sorting Order	Clicking on the **Name** button of **Sorting Order** in each window rearranges the Sample names and Group names in alphabetical order. Clicking on the **Date** button of **Sorting Order** rearranges them in chronological order.
OK	Click on the **OK** button in each window to finalize the Sample name and close the window.
Cancel	Click on the **Cancel** button in each window if you want to cancel the Sample name that was input and close the window.

4.1.2 Setting measurement conditions

You set the measurement conditions that you want for the spectrometers.

◆ Click on the **Measurement** button of the Qualitative Analysis function window.

The Measurement menu opens.

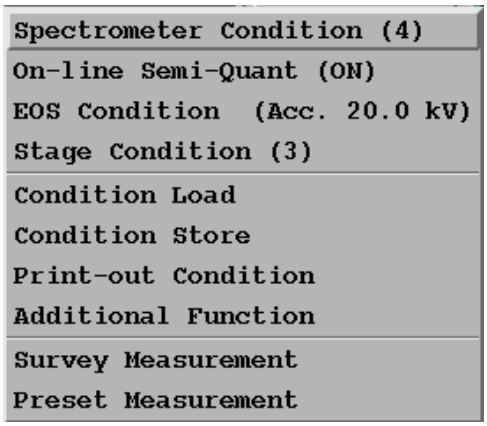

Fig. 4 Measurement menu for Qualitative Analysis

■ Spectrometer Condition

◆ To set measurement conditions, click on **Spectrometer Condition**.

The Spectrometer Condition window opens as shown in Fig. 5.

Here, you can set measurement conditions for the spectrometers. You set the number of spectra to collect by using the **No. of Spectra** button. This window displays the present settings for each spectrometer. You can change the desired settings displayed in the window.

The measurement conditions are given below.

The unit “mm” is used in the following explanation. You can choose the unit of wavelength from mm, A, and nm.

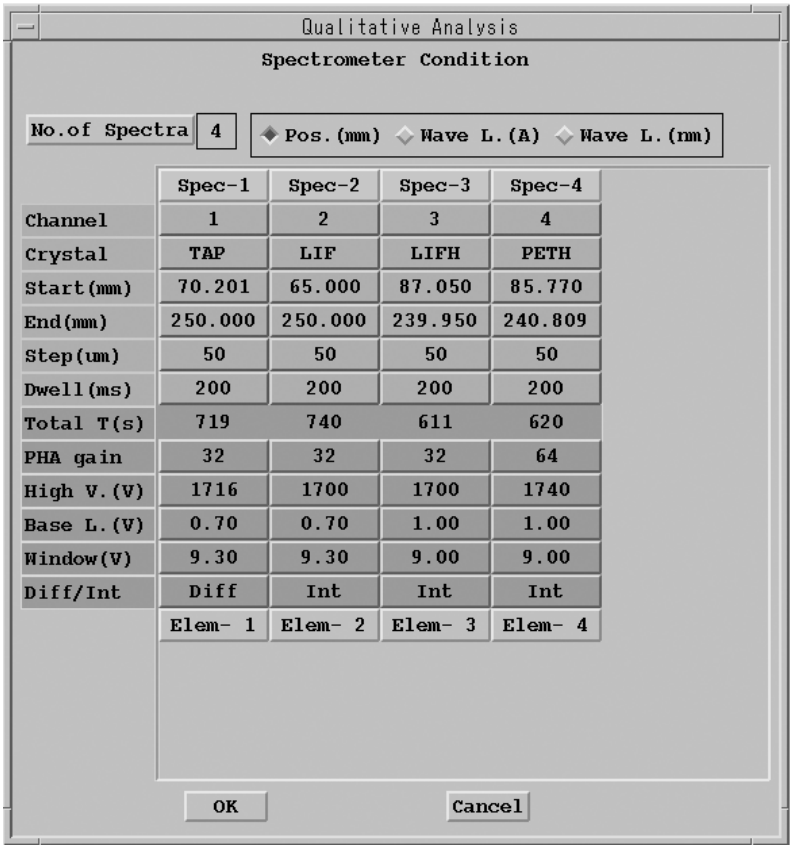

Fig. 5 Spectrometer Condition window

Buttons	Function
No. of Spectra	Input the number of spectra to be collected (up to 20 spectra possible).
Channel	Select the channel number of the spectrometer.
Crysrtal	Specify the analyzing crystal to be used.
Start	Specify the measurement start position. Setting is also possible in element-designation mode. (Refer to the following section.)
End	Specify the measurement end position. Setting is also possible in element-designation mode. (Refer to the following section.)
Step	Interval between neasurement points (μm). It is normally set to the following values: Approx. 50 µm for general analyzing crystals such as TAP, PET and LIF Approx. 100 µm for analyzing crystals such as STE and LDE2 (If **Step** is set to an excessively large value, the accuracy in element identification may deteriorate.) The number of measurement points for a single spectral line is given by the equation below. $n = (en - st) \times 1{,}000/in + 1$ where en: End (mm) value st: Start (mm) value in: Step (µm) value
Dwell	Measurement time per measurement (ms), which is to be decided, taking into account the total measurement time.

Buttons	Function
Total T	Total measurement time (s).
PHA gain **Hifh V.** **Base L.** **Window**	The values set in the SCA Configuration window are displayed. In normal use, it is not necessary to set these values.
Diff/Int	Pulse-height analysis mode setting (Diff*: differential mode, Int: Integral mode).

* With analyzing crystals for light elements, such as those of the LDE series, data with a high P/B ratio, which is less affected by high-order X-rays, can be obtained in the Diff mode. However, set the SCA conditions so that the primary ray will not be cut.

● Setting measurement start/end positions with element-designation mode

There are two methods for setting the measurement start and end positions.
One is to directly enter values of **Start (mm)** and **End (mm)** in the aforementioned Spectrometer Condition window. The other is to set the values using the element-designation mode in the WDS Elements window. The second method consists of the following steps.

1. Click on any one of the **Elem-1, -2, ...** buttons in the Spectrometer Condition window to display the WDS Elements window (Fig. 6).

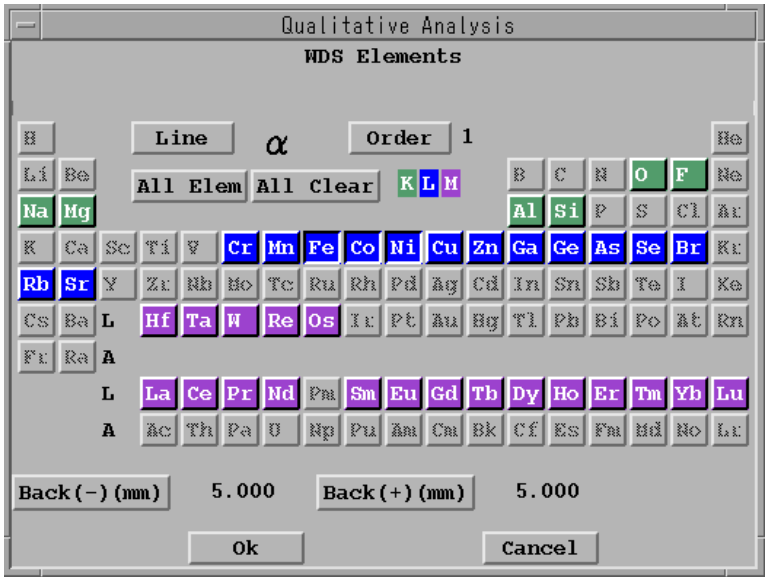

Fig. 6 WDS Elements window

2. Click on the **Order** button in the WDS Elements window to enter the order of lines, as necessary.
3. Click on the **Line** button to specify the type of lines (α, β or both α and β for K, L and M lines).
   The colored symbols of the elements to be measured are displayed in the periodic table of the elements.

4. Position the pointer on the desired element symbols and click on them.

✍ Clicking on only one element symbol is used in trace-element analysis for checking for the presence of X-ray peaks from trace elements.

✍ When multiple elements are specified, the measurement range will include all X-ray and background analysis positions for those elements.

5. To specify all measurable elements, click on the **All Elem** button.

The steps so far set the **Start** and **End** values in the Spectrometer Condition window so that they include the measurement ranges for all the elements specified.

✍ Clicking on the **Back (–) (mm)** or **Back (+) (mm)** button lets you change the distance from the peak position at which to measure the background. The default value is ±5 mm from the peak position.

■ On-line Semi-Quant

You can perform semi-quantitative analysis of the identified elements after qualitative measurement.

● Select Measurement–On-line Semi-Quant from the Qualitative Analysis function window.

The On-line Semi-Quant window opens.

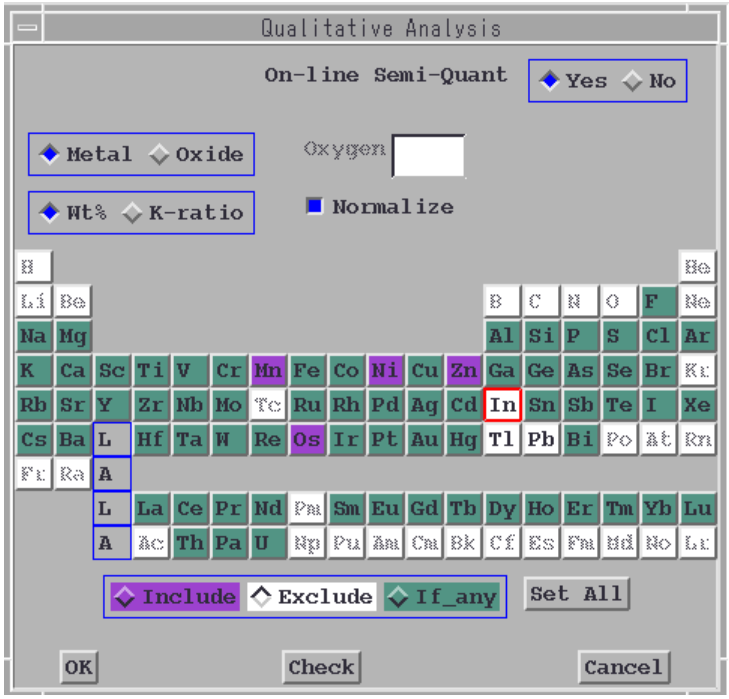

Fig. 7 On-line Semi-Quant window

◆ To carry out automatic semi-quantitative analysis, click on the **Yes** button of the On-line Semi-Quant window.

If you have selected the **Yes** button, proceed to the following Step 1.

✍ If you want to perform only qualitative analysis, click on the **No** button.

1. Click on **Metal** or **Oxide.**

- If you select **Oxide**, type the number of **Oxygen** in the input box for the calculation of chemical formula.
- Select **Wt%** or **K-ratio** for printing.
- If you select **Wt%** for printing, decide whether to normalize the total of mass concentrations to 100%.

2. To specify whether each element is to be included in the results, select **Include**, **Exclude**, or **If any**.

- **Include**: The element selected is included in the result in qualitative analysis, even if it is not identified.
- **Exclude**: The element selected is excluded from the result in qualitative analysis, even if it is identified.

If you want to set all the elements to **Exclude**, click on the **Set All** button.

- **If any**: The element selected is included in the result in qualitative analysis, if it is identified as A-rank.

If you want to set all the elements to **If any**, click on the **Set All** button.

3. If you want to specify the spectrum to be used for each element, click on the **Check** button.

The Check window appears.

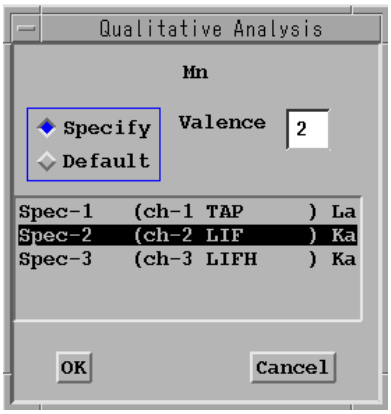

Fig. 7a Check window

If you select **Default**, the most intense spectrum will be used for calculation. When you set valence for an oxide, use the **Valence** input box of the Check window.

Qualitative analysis is performed in a normal way, and then element identification is also performed. If the **Yes** button is selected in the On-line Semi-Quant window, the results of element identification and the specified element are checked, and the results of the semi-quantitative analysis are calculated and displayed on the Listing window.

EOS Condition

The EOS Condition window allows you to set the conditions of the electron optical system (EOS). Clicking on the **Read** button reads present conditions for the EOS and displays them on the EOS Condition window in which you can input and alter items such as Probe Scan.

◆ Select **Measurement–EOS Condition** from the Qualitative Analysis function window.

The EOS Condition window opens.

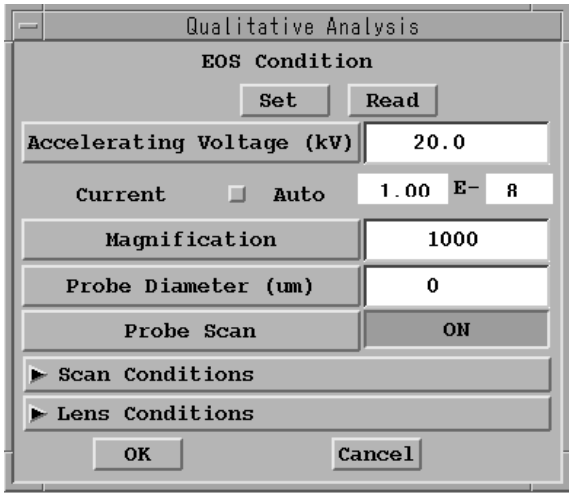

Fig. 8 EOS Condition window

Button	Function
Set	Sets the present conditions for the EOS.
Read	Reads the present conditions for the electron optical system (EOS) and displays them on the EOS Condition window.
Accelerating Voltage	Sets accelerating voltage at measurement.
Current	Displays beam current. To select the automatic current mode, in which a specified current is obtained before measurement, click on the **Auto** button; then specify beam current.
Magnification	Sets magnification for scanning image (active only when **Probe Scan** is ON).
Probe Diameter	Sets probe diameter (in µm) at measurement.
Probe Scan	Specifies whether probe scan will be ON or OFF during measurement.
Scan Conditions	Clicking on the arrowhead of this button opens the window for the following four items.
Scan Mode	Specifies scan mode (Picture, Bup, Line, Spot, or Area) for measurement.
Scan Speed	Selects scan speed from S1 to S12. The larger the number, the slower the speed.
Focus	Specifies automatic or manual focus.

Button	Function
Stabilizer	Specifies whether the beam stabilizer (CL&Tilt, CL, or Tilt) is to be used or not (OFF).
Lens Conditions	Clicking on the arrowhead of this button opens the window for the following two items.
Condenser Lens	Specifies condenser lense settings (Coarse/Fine) for measurement.
Object Lens	Specifies objective lense settings (Coarse/Fine) for measurement.
OK	Enters measurement conditions and closes the EOS Condition window.
Cancel	Cancels the conditions that have been input in the EOS Condition window and closes the window.

4.1.3 Setting analysis positions

You have to specify analysis positions before measurement. There are two modes of specifying the analysis positions. One is the Stage mode, and the other is the Beam mode.

◆ Select **Measurement–Stage Condition** from the Qualitative Analysis function window.

The Stage Condition window opens.

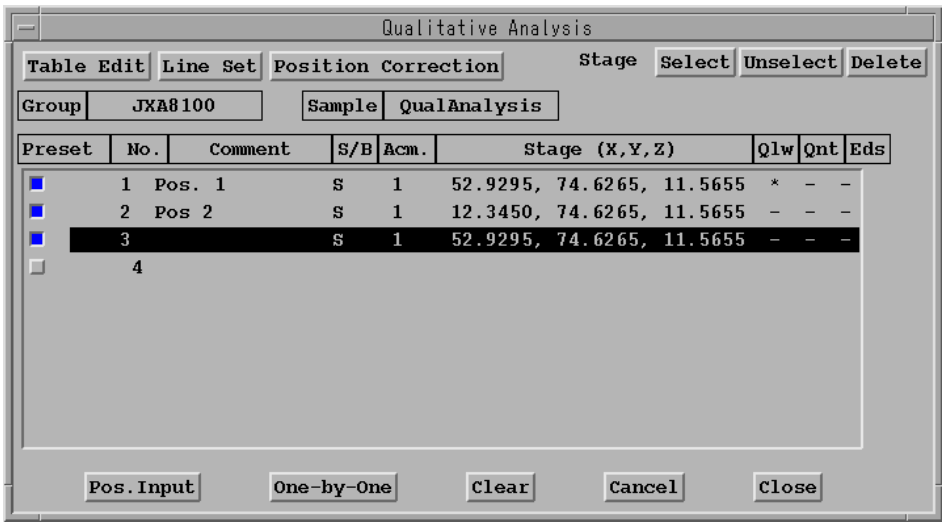

Fig. 9 Stage Condition window

When the Stage Condition window is opened, the list of coordinates that were recorded previously is displayed, and you can use it in common for qualitative analysis, quantitative analysis, and EDS qualitative analysis. Therefore, even when you perform qualitative analysis for the first time, coordinates exist provided you have carried out some other analysis. The results of analysis, however, are recorded separately even when you use the same coordinates.

The columns of the list are **Preset** for analysis execution, **No.** for analysis position number, **Comment**, **S/B** for Stage mode or Beam mode, **Acm.** for the number of accumulation times, **Stage (X, Y, Z)** for coordinates of stage position, and **Qlw** (qualitative analysis) /**Qnt** (quantitative analysis)/ **Eds** (EDS qualitative analysis) marked with asterisks when they are applied. One line of the list is always selected, and entries made using **Pos. Input** (for coordinate input) and **One-by-One** (for one-by-one analysis) affect this line.

- When you want to enter a new analysis point, select the blank line at the bottom of the list.
- When you want to alter one of the analysis points in the list, select a set of coordinates from the list, and then click on the **Pos. Input** button.
- When you want to perform an analysis at one specified point, click on **One-by-One**.

Button	Function
Pos. Input	To specify an analysis position, click on this button. The Stage Condition window is displayed, allowing you to specify analysis positions. Refer to the next section, “Stage Control Input window”.
One-by-One	Performs one specified analysis at the highlighted stage position.
Clear	Clears the highlighted line. If there are any lines below the highlighted line, they move up.
Cancel	Cancels the newly input values without entering them in the Stage Condition file. The Confirmation window appears before the input line is cleared.
Select/Unselect	To measure at the recorded coordinates with the Preset mode (☞ refer to Section 4.2.1), the **Preset** button must be on. To turn **Preset** on for all or some coordinates points, click on **Select**. To turn **Preset** off, click on **Unselect**.
Delete	Deletes some or all sets of recorded coordinates. If there are any lines below the deleted lines, they move up.

✍ When you have deleted the analysis point values by using **Clear** or **Delete**, if there are any lines below the deleted lines, they move up. If you have executed the analysis at the recorded analysis point, its results will remain unchanged.

☞ Table Edit, Line Set and Position Correction are available in this Stage Condition window. Refer to the separate instruction manual, “Quantitative Analysis Program”.

Stage Condition Input window

You set the analysis position of the stage or beam in this window that opens when you click on the **Pos. Input** button of the Stage Condition window.

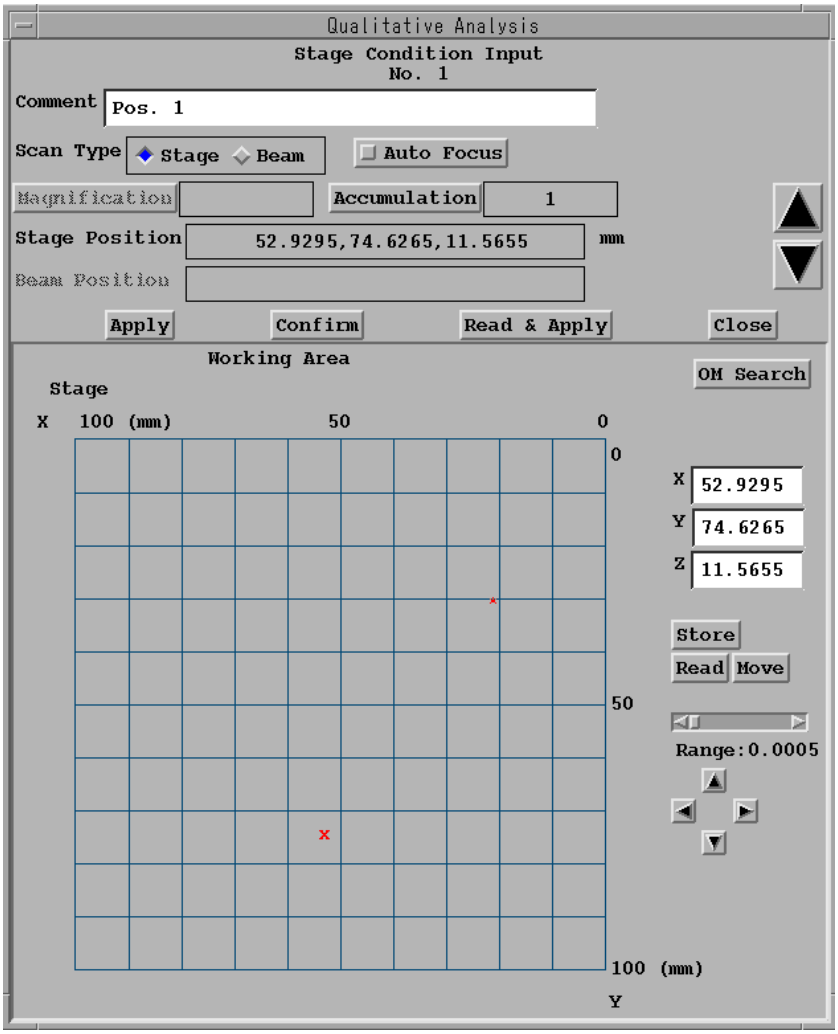

Fig. 10 Stage Condition Input window with Working Area for Stage

Entering the analysis position in the Stage mode

1. Confirm that Scan Type is **Stage**.
   If it is not, click on the **Stage** button.
2. Move the stage to the analysis position that you want to analyze, observing the OM image by using the joystick of the Joystick Controller of the EPMA main unit; then after focusing on the position, eliminate backlash by using the TEST button of the Joystick Controller.
   It is especially necessary to eliminate backlash before you perform continuous analysis in the Preset mode.
3. Click on the **Read** button to display the present stage position; then click on the **Store** button to enter the coordinates of the position.

Alternatively, click on the **Read & Apply** button, and then this step will be executed automatically. The same result will be obtained by pressing the STORE button of the Joystick Controller. In this case, after storing the position, the coordinates of the next position will be indicated. If the last character of the comment is a number, it will be incremented automatically.

4. To confirm and edit already-specified coordinates, first select the corresponding analysis position in the Stage Condition window; then move the stage using the **Move** button. After confirming the coordinates of the point by using the joystick, record the coordinates by carrying out Step 3.

- **Entering the analysis position in the Beam mode**

1. Confirm that Scan Type is **Beam**.
   If it is not, click on the **Beam** button.
2. Display an image of the analysis position on the Viewing Display.
   ☞ Refer to the instruction book of the EPMA main unit.
3. Once you have decided on the analysis position, set the image on the Viewing Display to the analysis mode; change the cross cursors to green, and then select analysis points.
4. Click on the **Read** button.
   Stage Position (**X**, **Y**, **Z**), **Magnification**, and **Beam Position** (**X**, **Y**) will be read.
5. To enter the analysis position, click on the **Store** button.

The items of the Stage Condition Input window are explained in the following table.

Object	Function
Comment	You can input up to 40 characters as an explanation of the sample.
Scan Type	Specify the Stage mode or Beam mode.
Auto Focus*	Click on this button to perform automatic stage focusing before measurement, if the optional automatic focusing device has been installed.
Magnification	Specify the magnification of the EOS by clicking on the **Read** button. This function will be effective only in the Beam mode.
Accumulation	You can specify up to 100 accumulations. Enter values, and the Coordinate Accumulation Setting window opens. Select Joystick, Line, Grid, or Fix. If you select Line or Grid, enter the number of steps and the scan width. When you specify **Accumulation**, confirm each coordinate point by clicking on the **Confirm** button.
Stage Position	Displays the present recorded position of the stage.
Beam Position	Displays the present recorded position of the beam (only in the Beam mode).
Apply	You can enter analysis points in the list of coordinates by clicking on this button.
Confirm	Be sure to click on this button when you have specified the number of accumulations. Move to the accumulation point by using the Joystick Controller; then after confirming the focus, press the STORE button of the Joystick. Repeat this operation as many times as the accumulation number. If you click on the **Cancel** button of the window, the remaining accumulation points are neglected, and the number of accumulations is reset to the number that you did prior to cancellation.

Object	Function
Read & Apply	Reads the position of the stage, and also that of the beam if necessary, and records them in the list of coordinates. The same result can be obtained by using the STORE button of the Joystick Controller.
Close	Closes the Stage Condition Input window. If the analysis position has been changed, the Confirmation window opens.
Upward and downward arrow buttons	Move to the previous or following coordinates. If the analysis position has been changed, the Confirmation window opens.
OM Search*	Executes the automatic stage focusing at the present position of the stage, if the optional automatic focusing device has been installed.
X, Y, Z	Displays the coordinates of the recorded stage position. If you have selected **Read**, the present position of the stage is displayed.
Read	Reads the present position of the stage and displays it in the the **X**, **Y**and **Z** boxes.
Store	Copies the values of **X**, **Y** and **Z** to the **Stage Position** box.
Move	Moves the stage to the position having the coordinates **X**, **Y** and **Z**.
Arrow buttons	Moves the stage by the specified step width in the X or Y direction.
Range	Drag the scroll bar to specify the amount that the stage will move when you click on the Arrow buttons.

* These items are optional.

4.1.4 Loading measurement conditions

If you load preset measurement conditions, you can perform measurements by simply selecting samples and inputting stage positions.

◆ Select **Measurement–Condition Load** from the Qualitative Analysis function window.

The Condition File Load window opens.

This window has the list of recorded measurement conditions such as file names, dates recorded and comments.

- To call up the recorded conditions, select the desired file from the list of recorded measurement conditions, and click on the **Load** button.

  The loaded conditions are settings of **Spectrometer Condition**, **On-line Semi-Quant**, **EOS Condition**, **Print-out Condition**, and **Additional Function**.
- If you click on the **Check** button before loading, you can check the stored conditions.

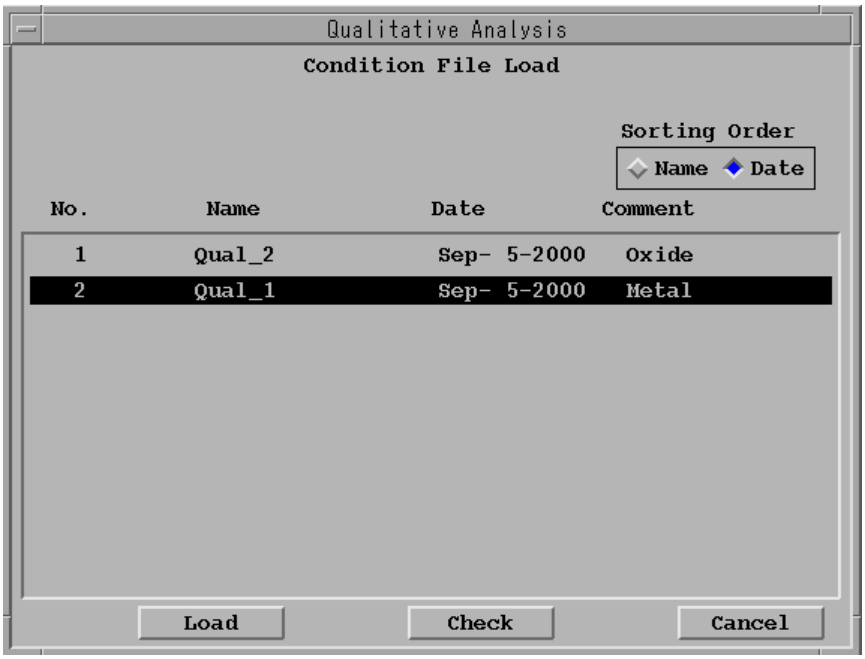

Fig. 11 Condition File Load window

4.1.5 Storing measurement conditions

If you set conditions in the Spectrometer Condition, On-line Semi-Quant, EOS Condition, Print-out Condition, and Additional Function windows, you can store them in a file and give a new name to the file.

◆ Select **Measurement–Condition Store** from the Qualitative Analysis function window.

The Condition File Store window opens.

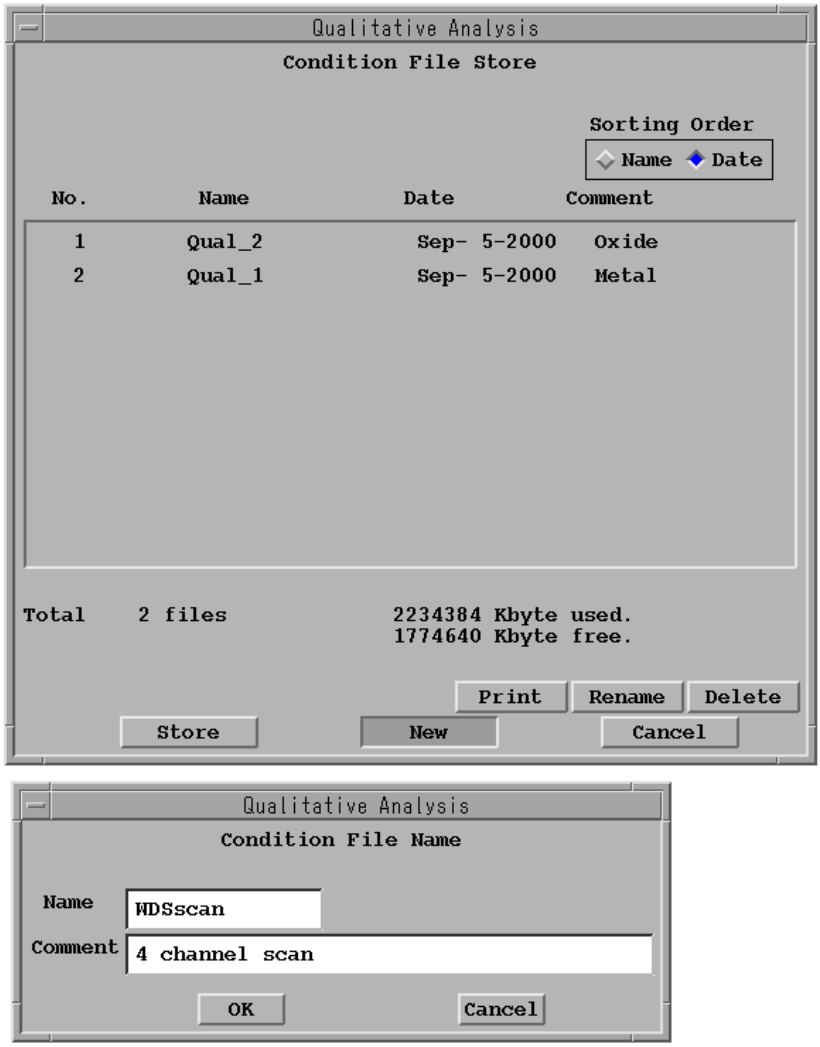

Fig. 12 Condition File Store window with its sub-window

- When you want to store new measurement conditions, click on the **New** button, and input a file name (14 characters maximum) and a comment (40 characters maximum); then click on the **Store** button.
- When you want to write new measurement conditions in place of the existing measurement conditions recorded in a file, select the desired file from the list of recorded measurement conditions, and then click on the **Store** button.
- You can carry out file operations such as printings (**Print**), changing the name (**Rename**) and deleting (**Delete**) in the Condition File Store window.

- When you want to back up recorded data to other media, select **Utility–File Utility** from the EPMA Main Menu.

4.1.6 Printing measurement conditions and results

◆ Select **Measurement–Print-out Condition** from the Qualitative Analysis function window.

The Print-out Condition window opens, and you can select items that you want to print.

☞ Refer to Fig. 13.

Button	Function
Measurement Condition	Prints measurement conditions
Summary of Identified Elements	Prints names of identified elements in the A and B ranks.
Peak Positions with Identified Elements	Prints list of peak positions and intensities
Identified Elements with Peak Positions	Prints peak position for each identified element
Semi-Quant Result	Prints results of semi-quantitative analysis

```
                >>> Qualitative Measurement condition. <<<

    Group    : JXA8100            Sample : Ceramics
    Comment  :
    Date     : Oct 28 13:26 1999
    Stage No.1     Position mm  X : 15.3685   Y : 87.5315   Z : 11.1775

    Accelerating Voltage          20.0  kV
    Probe Current             2.148E-07  A
    Probe Scan Off,  Probe          50  um

           |  Spec- 1  |  Spec- 2  |  Spec- 3  |  Spec- 4  |
-------------------------------------------------------------
Channel    |     1     |     2     |     3     |     4     |
Crystal    |   TAP     |   LIF     |   PETH    |   LDE2    |
Start  (mm)|   75.000  |   75.000  |   86.025  |   61.000  |
End    (mm)|  229.980  |  230.000  |  229.945  |  230.000  |
Step   (um)|       70  |       50  |       70  |      200  |
End-St (um)|  154.980  |  155.000  |  143.920  |  169.000  |
Points     |     2215  |     3101  |     2057  |      846  |
Dwell  (ms)|       75  |       55  |       80  |      180  |
PHA gain   |       32  |       32  |       64  |       64  |
High V  (V)|     1736  |     1726  |     1756  |     1780  |
Base L  (V)|     0.69  |     0.68  |     0.68  |     0.69  |
Window  (V)|     9.30  |     9.30  |     9.00  |     9.30  |
Diff/Int   |   Diff    |   Int     |   Int     |   Diff    |
Clb.Fctr(0)| -0.591565 | -1.666040 | -1.991093 | 0.000000  |
        (1)|  0.146720 |  2.608430 |  0.968443 | 0.000000  |
        (2)| -0.011457 | -1.257531 | -0.168091 | 0.000000  |
        (3)|  0.000258 |  0.197070 |  0.010351 | 0.000000  |
Max. data  |     4109  |     1060  |     1940  |     7675  |
Min. data  |        0  |        0  |        2  |        2  |
Ave. data  |       40  |       10  |       57  |      321  |
Accum.     |        1  |        1  |        1  |        1  |
-------------------------------------------------------------

                >>> Summary of identified elements. <<<

    Group    : JXA8100            Sample : Ceramics
    Comment  :

    *ID-Doctor*
    A-Rank : C  O  Mg Al Si P  K  Ca Ti Cr Mn Fe Zr Sn
    B-Rank : Na Ga As
```

Fig. 13 Printout example

4.1.7 Additional Function

◆ Select **Measurement–Additional Function** from the Qualitative Analysis function window.

You can specify whether **ID-Doctor** will function or not for element identification at measurement. By default, **ID-Doctor** will operate.

4.2 Measurement

Two methods of measurement are available: one is the **Preset** mode and the other is the **Survey** mode.

Preset mode: Method of performing measurement by using stored measurement conditions.

Survey mode: Method of performing measurement without changing the present instrument conditions (for the EOS and stage). Use this mode to perform analysis without specifying detailed measurement conditions.

✍ Real-time spectrum display is possible during measurement in both modes.

☞ Refer to Sect. 4.2.3 “Realtime display”.

4.2.1 Measurement under stored conditions (Preset mode)

This section describes how to perform measurement under stored measurement conditions and positions in the Preset mode.

1. Select **Measurement–Preset Measurement** from the Qualitative Analysis function window.

The Preset Measurement window opens.

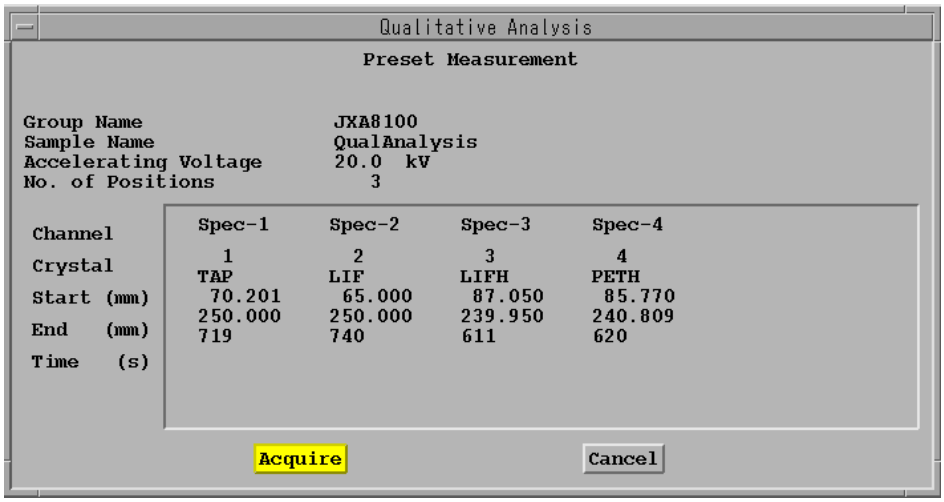

Fig. 14 Preset Measurement window

2. Click on the **Acquire** button in the Preset Measurement window.

The preset measurement conditions and the analysis positions in the list of the Stage Condition window whose **Preset** check boxes are turned on will be loaded, and then the measurement will be carried out at the analysis positions.

4.2.2 Measurement under present instrument conditions (Survey mode)

This section describes how to perform measurement under present EOS conditions and at the stage position in the Survey mode.

1. Select **Measurement–Survey Measurement** from the Qualitative Analysis function window.

Survey Measurement will be performed. The data obtained will be stored always at the stage number 99999. The data will be overwritten every time the measurement is performed.

2. When you wish to store the measurement results in a file after **Survey Measurement**, click on the **Save** button; then enter Position No. and Comment.

The limit of stage number is one more than the number of positions that are already set.

During measurement, the Measurement Control Window appears as shown in Fig. 15, allowing you to interrupt measurement (**Measurement Stop**) and stop accumulation (**Accum. Stop**). If you have selected **Accv off**, the accelerating voltage will be turned off automatically after measurement.

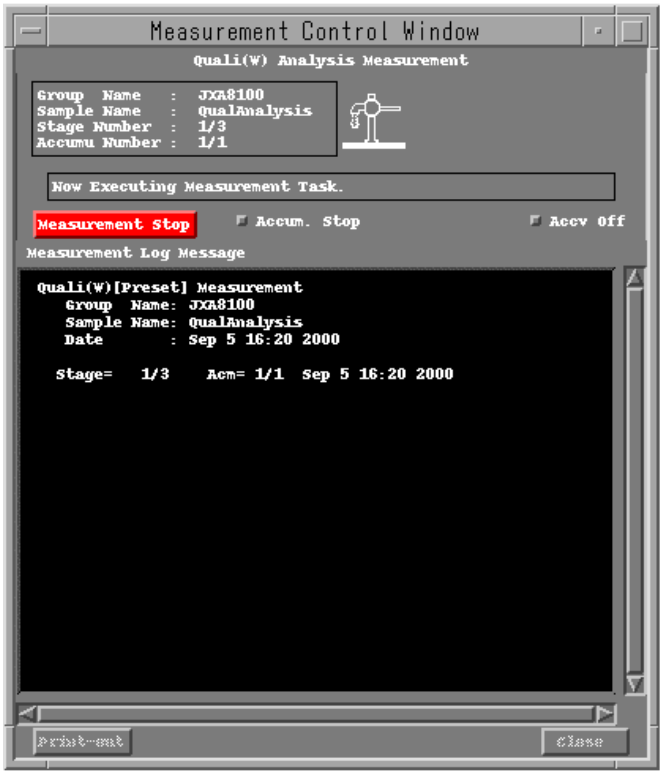

Fig. 15 Measurement Control Window

4.2.3 Realtime display

1. Select **Process–Qualitative Analysis** from the EPMA Main Menu during measurement.

The Data display window opens.

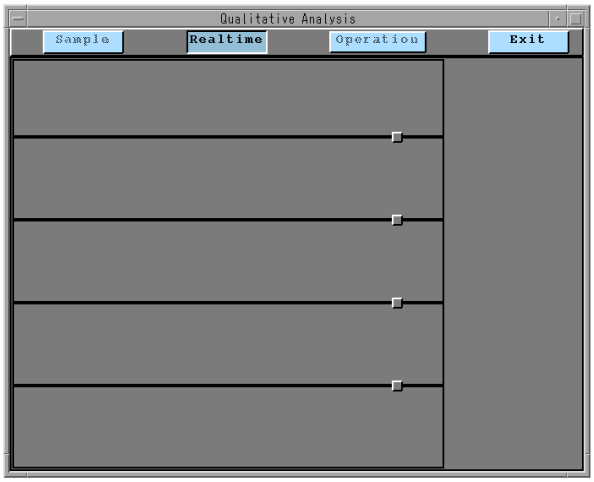

Fig. 16 Data display window

2. Click on the **Realtime** button.

The Realtime Display window opens. You can perform **Spectra Display** during measurement. If you want to display **KLM Marker**, confirm the peak position using **Peak ID** during realtime display, or enlarge a part of spectrum, click on the **Stop** button to stop the realtime display; then you can perform these operations.

☞ For details, refer to Sect. 4.3 “Processing”.

Fig. 16a Realtime Display window

Button	Function
Sample	Specifies the sample and displays the measured spectra. Do not click on **Sample** during realtime display. When you want to display spectra that have already been measured, stop the realtime display; then click on the **Sample** button and select the sample that you want to display.
Realtime	Executes realtime display. Click on the **Realtime** button. The Realtime Display window of Fig. 16a opens. Click on the **Start** button; then the spectra being measured are displayed. When the analysis point has moved to other coordinates, the message "Next Display?" appears. If you want the next realtime display to begin immediately, select **OK**. After apause, the realtime display of the next sample begins automatically. The sequence of spectrum display can be changed as follows: **Channel No. Order**: Spectra are displayed in real time in the order of the spectrometer channel, beginning with channel 1 at the top. **Spectrum No. Order**: Spectra are displayed in real time in the order of measurement. To select the number of spectra (1 to 8) to be displayed, click on the **Max Cell Number** button.
Operation	Performs operations described below for spectra to be displayed in real time.

■ Operation menu during realtime display

◆ Select **Operation** during realtime display.

The Operation menu opens. During realtime display, some selections are unavailable.

Spectra Display, **KLM Marker** and **Peak ID** can be selected.

☞ Refer to Sect. 4.3 "Processing" for a description of each operation.

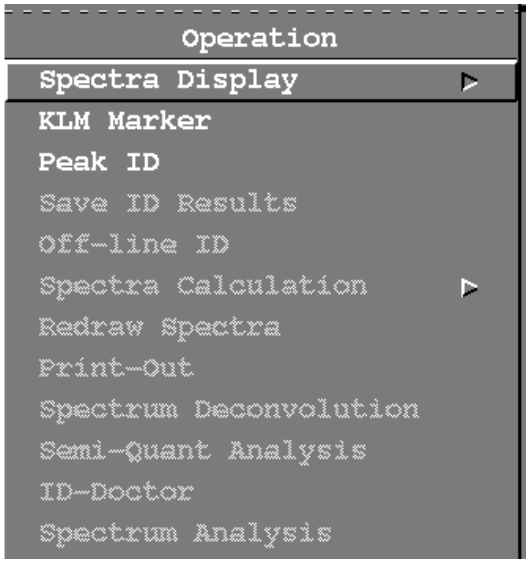

Fig. 17 Operation menu (during realtime display)

4.3 Processing

This section describes how to process the data obtained from the measurements performed so far.

4.3.1 Selecting sample names and processing methods

1. Select **Process–Qualitative Analysis** from the EPMA Main Menu shown on the Computer Display.

The Data display window opens.

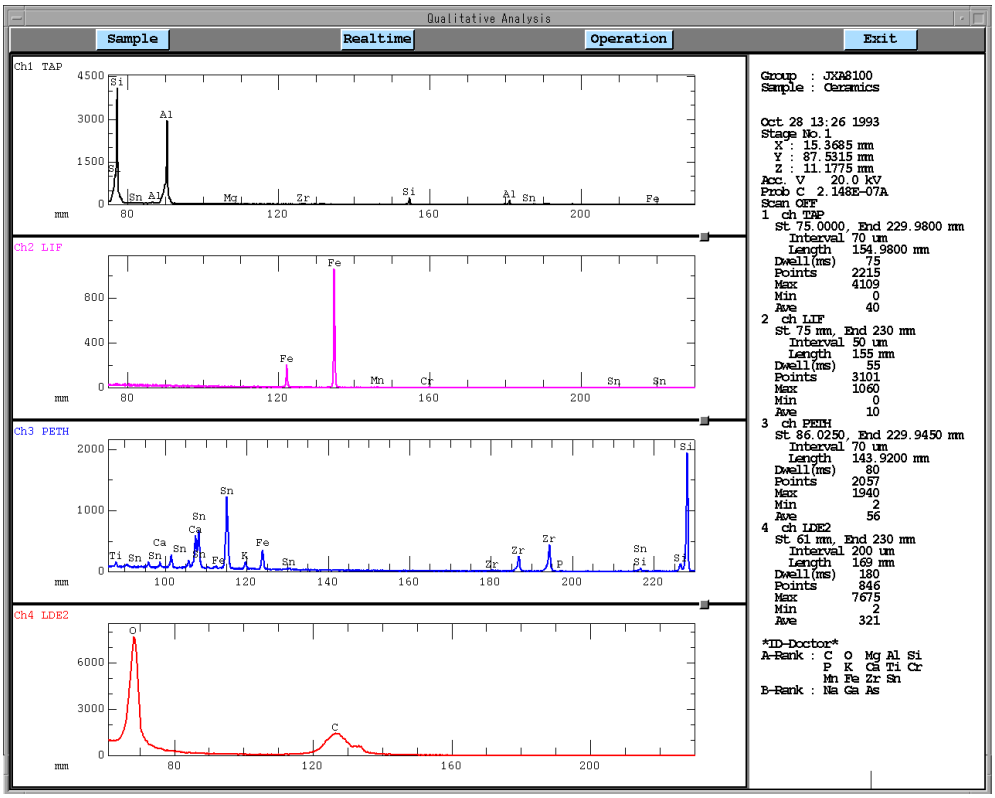

Fig. 18 Data display window

2. Click on the **Sample** button in the Data display window.

The Sample window (for processing) opens.

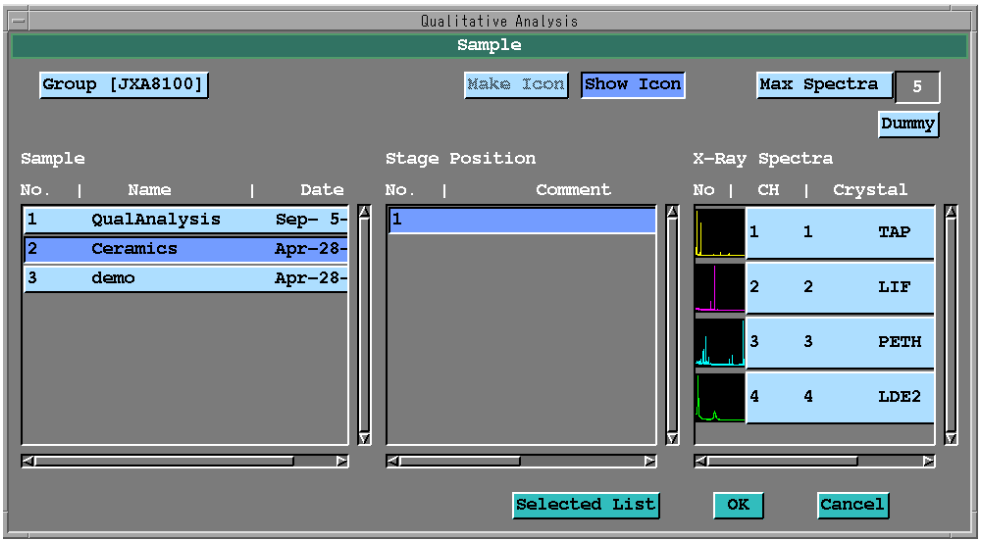

Fig. 19 Sample window (for processing)

3. Select the spectra to be displayed from the Sample window.

There are two display modes. One is the single-sample mode, in which the spectra of one analysis point are displayed. The other is the multiple-sample mode, in which the spectra of different samples are displayed at the same time. The latter mode is used when you perform the chemical shift and the optional waveform separation. However, you cannot perform the off-line element identification, nor the semi-quantitative analysis, using the multiple-sample mode. To specify the number of spectra to be displayed, click on the **Max Spectra** button.

In the Sample window, the lists of Sample Name, Stage Position, and X-Ray Spectra are displayed. In the X-Ray Spectra list, thumbnail spectra are displayed. If they are not displayed, click on **Show Icon** to show them.

Button	Function
Make Icon	Makes thumbnail spectra. Normally, the spectra are made automatically at measurement.
Show Icon	Switches on and off the display of thumbnail spectra.
Dummy	Displays blank spaces in place of spectra.
Max Spectra	Specifies the number of spectra to be displayed.
Selected List	Displays the list of spectra that have been selected. To deselect spectra, click on their names in the list.

4. If you want to display and process data of another group, click on the **Group** button. The Group window that lists existing group names opens as shown in Fig. 20. Click on the desired group name and then click on the **OK** button. Repeat Step 3.

5. In the single sample mode, select **Group⇒Sample⇒Stage Position**, and then select the desired spectra in **X-Ray Spectra**.

If you do not select any spectra in **X-Ray Spectra**, all the displayed spectra will be selected automatically.

6. In the multiple sample mode, every time you select a spectrum, select group, sample and stage position.

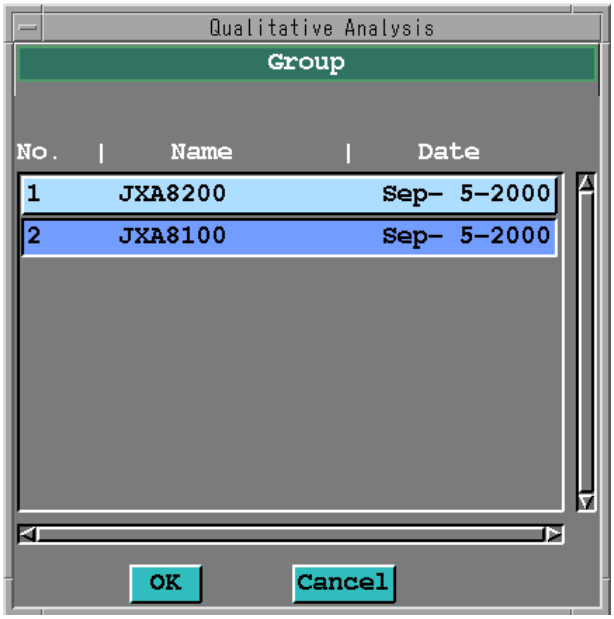

Fig. 20 Group window (for processing)

7. Click on the **OK** button.

Spectra will be displayed in the Data display window in the order in which you selected them in **X-Ray Spectra**.

The data corresponding to the item that was specified with **Display Parameter** (☞ Sect. 4.3.2) are displayed at the right of the spectra.

4.3.2 Operation menu

◆ Click on the Operation button in the Data display window of Fig. 18.

The Operation menu opens as shown in Fig. 21.

Clicking on a menu item allows the operations described below. The menu items that you use frequently can be selected from the pop-up menu that opens by right clicking on any position on a displayed spectrum.

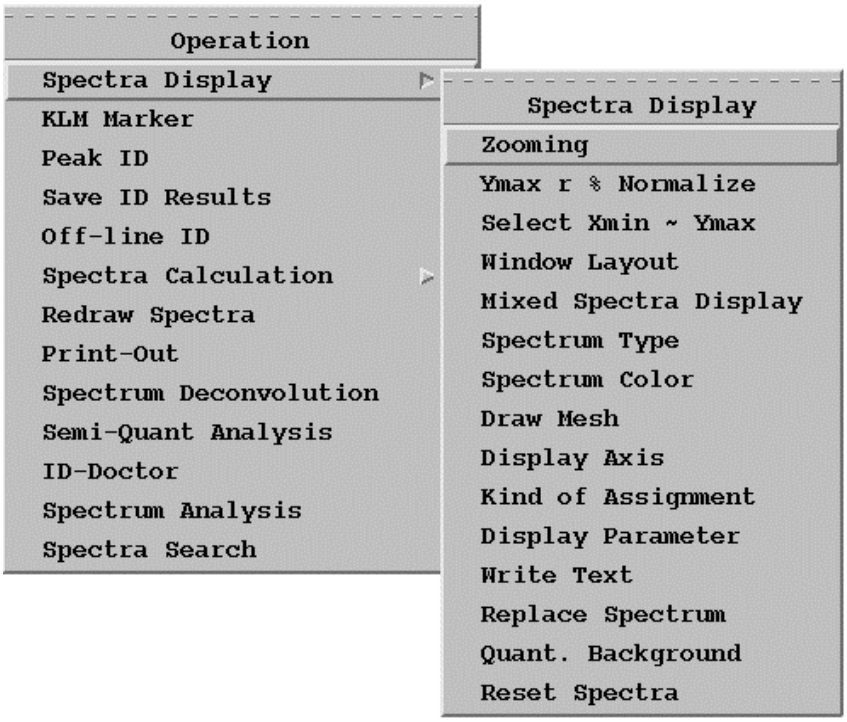

Fig. 21 Operation menu

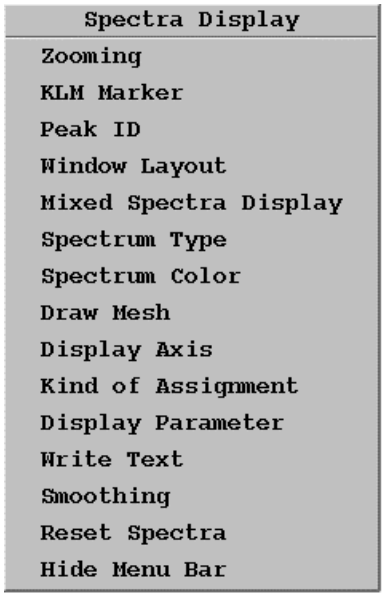

Fig. 22 Pop-up menu

Spectra Display

Selecting **Spectra Display** from the **Operation** menu shows the menu items related to the methods of displaying spectra, allowing the following operations.

Zooming

1. Select **Spectra Display**–**Zooming** from the Operation menu.
   The Zooming window opens, and also at the same time the graph window showing the whole spectrum image opens separately as shown in Fig. 23.
   To specify the spectrum to be processed, select a spectrum number, or click on any position on the spectrum that is being displayed.
2. Position the mouse cursor near the position where you want to start to zoom and drag the cursor in the diagonal direction.
   An enlarged rectangular frame is formed.
3. Once you have decided the size to be zoomed in on, release the mouse button, and then click on the **Apply** button.
   The part of the spectrum in the specified area is enlarged in the graph window. If you performed Step 2 in the graph window, you need not click on the **Apply** button.
4. To move the frame for enlargement, drag a point near the center of the frame to the desired position. If you want to change the size of the frame, drag the frame line.

   ✍ In these operations, the shape of the mouse cursor will change so that you can distinguish each operation.

   ✍ You can perform the following operations by using the arrow and other keys of the keyboard.

   [→] : Moves the frame to the right.
   [←] : Moves the frame to the left.
   [↑] : Enlarges in the vertical direction.
   [↓] : Reduces in the vertical direction.
   [+] : Enlarges in the horizontal axis direction.
   [−] : Reduces in the horizontal axis direction.
   [Shift] : If you press one of the above keys while holding down this key, the amount of movement will increase.

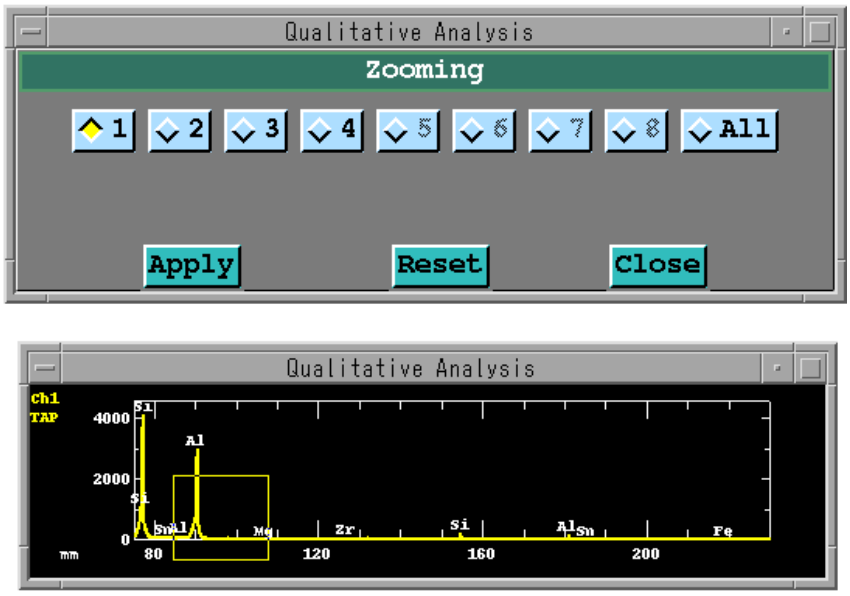

Fig. 23 Zooming window and its graph window

- **Ymax r % Normalize**

  You can redisplay the maximum intensity of a spectrum on display at the desired full-scale percentage by using this function.

  1. Select **Spectra Display–Ymax r % Normalize** from the Operation menu.
  The Ymax r % Normalize window opens.

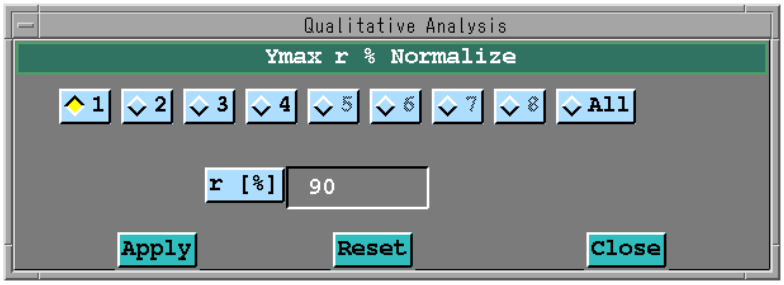

Fig. 24 Ymax r % Normalize window

2. Click on the **r [%]** button in the Ymax r % Normalize window.
The r window opens as shown.

Fig. 24a r window

3. Enter the desired percentage in the input box of the r window by clicking on the number buttons in the r window; then click on the **Apply** button of the Ymax r % Normalize window.
The maximum intensity of spectrum on display will be redisplayed as the percentage of the full-scale value that you entered.

If the **All** button is selected in the Ymax r % Normalize window, the full-scale percentage of all the spectra is set to the same value.

● Select Xmin – Ymax

You can set the horizontal and vertical scales for spectra to the desired values.
The procedure is the same as for the Ymax r % Normalize window.

1. Select **Spectra Display–Xmin – Ymax** from the Operation menu.
The Select Xmin – Ymax window opens.

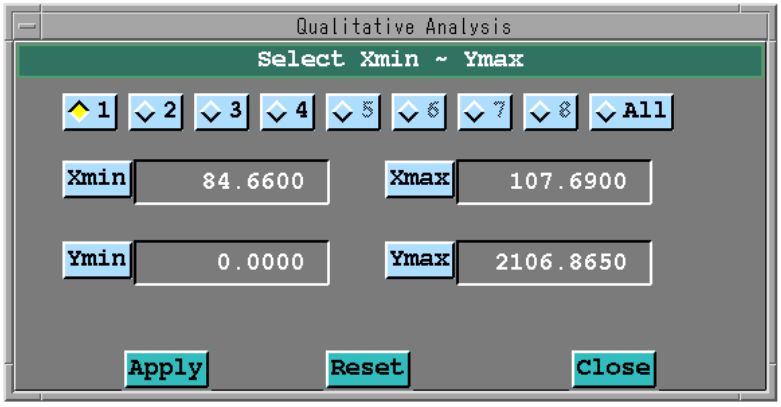

Fig. 25 Select Xmin – Ymax window

2. Click on the **Xmin**, **Ymin**, **Xmax**, or **Ymax** button in the Select Xmin – Ymax window.

   The **Xmin**, **Ymin**, **Xmax**, or **Ymax** window opens.

3. Enter the desired values in the input box of the window by clicking on the number buttons in the window; then click on the **Apply** button of the Select Xmin – Ymax window.

   The horizontal and vertical scales for spectra are set to the specified values.

   If the **All** button is selected in the Select Xmin – Ymax window, the scales of all the spectra are set to the same values.

Window Layout

You can specify the layout of the Data display window by using this function.

◆ Select **Spectra Display–Window Layout** from the Operation menu.

The Window Layout window opens.

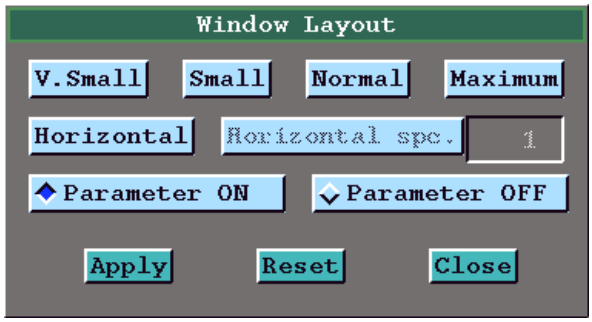

Fig. 26 Window Layout window

Window size:	Select the desired size from **V. Small**, **Small**, **Normal**, and **Maximum**.
Horizontal layout:	Usually, spectra are displayed vertically in a column. If you want to display the spectra horizontally in lines, click on the **Horizontal** button, and then enter the number of spectra to be shown horizontally in the **Horizontal spc.** input box.
Parameter display:	If you do not want to display parameters on the right side, select **Parameter OFF**. If you want to display them, select **Parameter ON**.

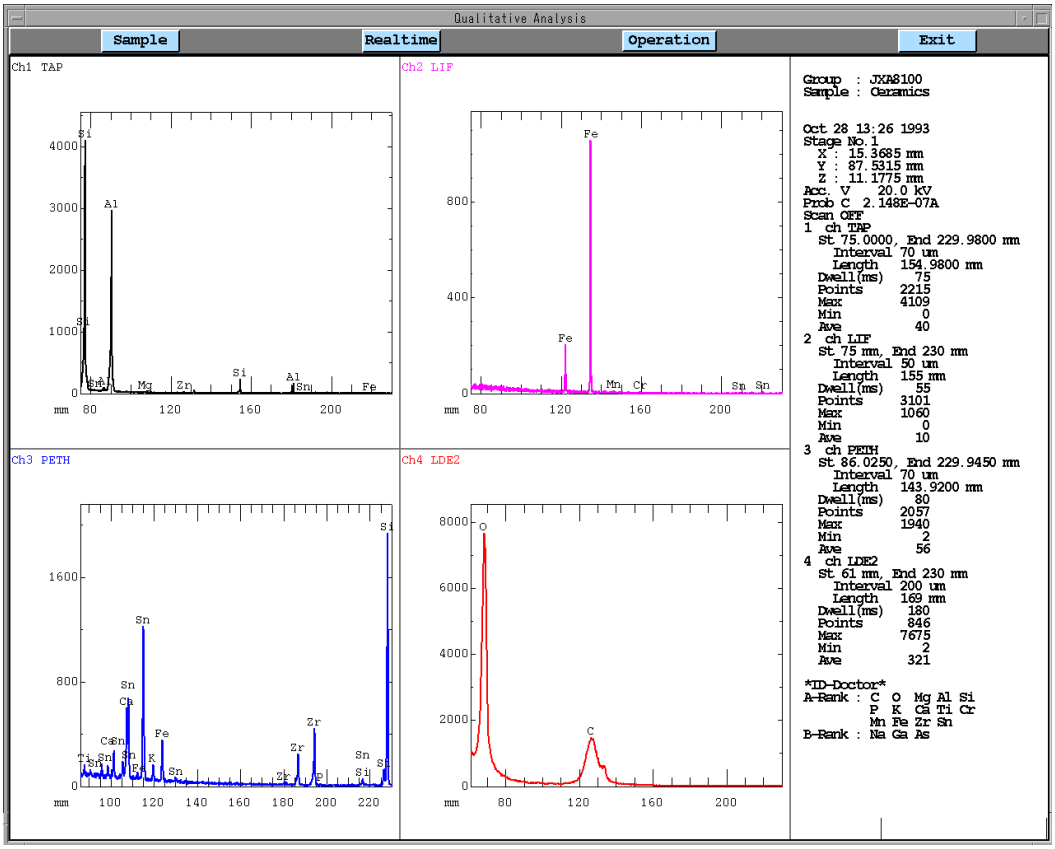

Fig. 27 An example of the horizontal display of spectra in lines

● Mixed Spectra Display

You can display spectra individually on different parts of the display or all together in one area.

◆ Select **Spectra Display–Window Layout** from the Operation menu.
The Mixed Spectra Display window opens.

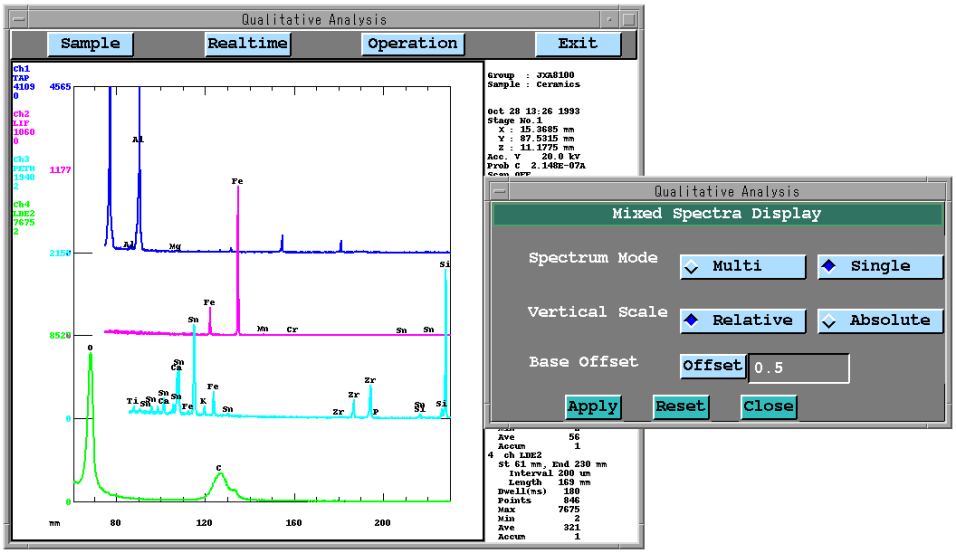

Fig. 28 Mixed Spectra Display window

If **Spectrum Mode** is **Single**, spectra are displayed all together in one display area. In the **Single** mode, if you have selected **Relative** for **Vertical Scale**, the full-scale value of the vertical axis becomes 100; the relative intensity of each spectrum is shown with the maximum value of each spectrum taken as 100. If you have selected **Absolute** for **Vertical Scale**, spectra will be shown with the maximum value of all spectra taken as 100.

The offset value of the Y-axis is displayed as the value of **Base Offset** when the maximum value of each spectrum is taken as 1. In the **Single** mode, each spectrum is displayed on the same base line when the value of **Base Offset** is 0.0. However, each spectrum is displayed on a different base line with the same base level when the value of **Base Offset** is 1.0.

● Spectrum Type

You can specify the style for displaying spectrum lines.

1. Select **Spectra Display–Spectrum Type** from the Operation menu.

The Spectrum Type window opens.

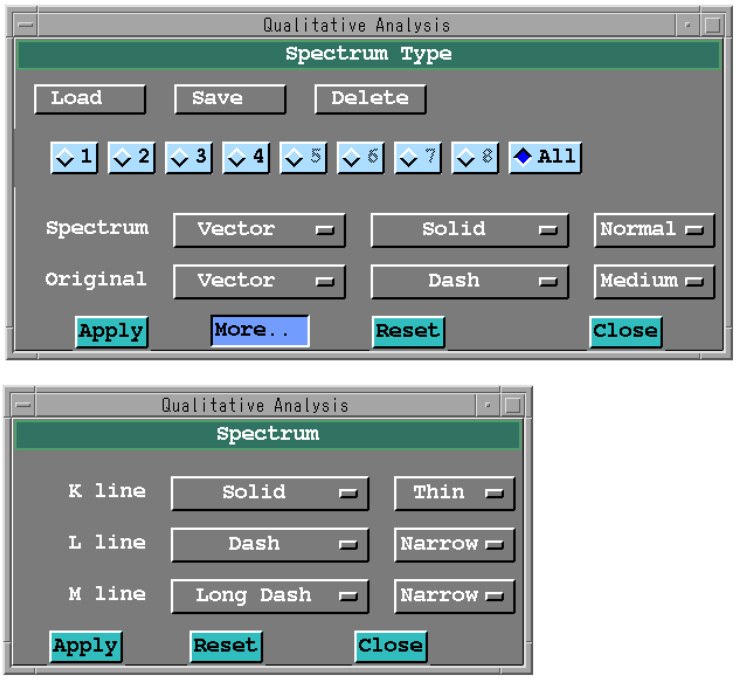

Fig. 29 Spectrum Type and Spectrum windows

2. Select a spectrum number from the Spectrum Type window.

3. To specify the style for displaying spectrum lines, select the desired items from the following list; then click on the **Apply** button to display the spectra in the specified style.

- Style for displaying the line

Vector:	Line
Marker:	Symbols
Bar:	Vertical bars
Marker + Vector:	Symbols and line
Vector + Bar:	Lines and vertical bars

- Line type for Vector

Solid:	Solid line
Dash:	Dashed line
Dot:	Dotted line
Dash Dot:	Dashed line with a dot
Dash Dot Dot:	Dashed line with two dots
Long Dash:	Long dashed line

- Thickness of the line

Thin:	Thin line
Normal:	Normal line
Thick:	Thick line

You can store, recall and delete using the **Load**, **Save** and **Delete** buttons.

Spectrum Color

You can change the color of the displayed spectra, background and parameter area by using this function.

1. Select **Spectra Display**–**Spectrum Color** from the Operation menu.
The Spectrum Color window opens.

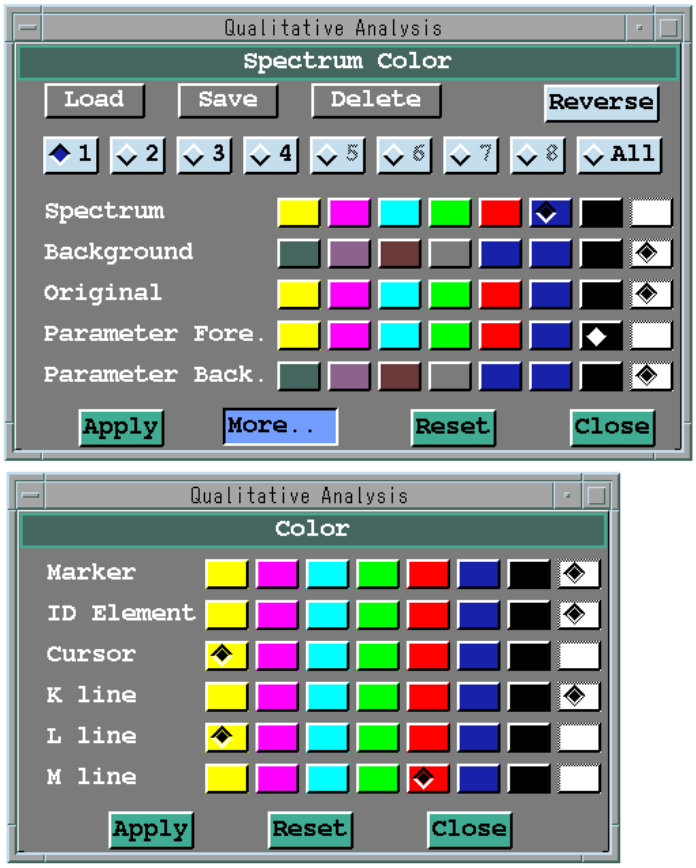

Fig. 30 Spectrum Color and Color windows

2. Select a spectrum number from the Spectrum Color window.
3. To specify the color for displaying spectra, background and parameter areas, select the desired items from the following list; then click on the **Apply** button to display the color as desired.

- Items available for color display

Spectrum:	Spectra
Background:	Background
Original:	Original spectra prior to calculation
Parameter Fore.:	Parameter characters
Parameter Back.:	Parameter background

- Buttons for operations

More..:	Lets you to specify the colors of **Marker**, **ID Element**, **Cursor** and **KLM lines**

You can store, recall and delete by using **Load**, **Save** and **Delete** buttons.

Draw Mesh

You can draw a grid as the background of spectra by using this function.

1. Select **Spectra Display–Draw Mesh** from the Operation menu.
   The Draw Mesh window opens.

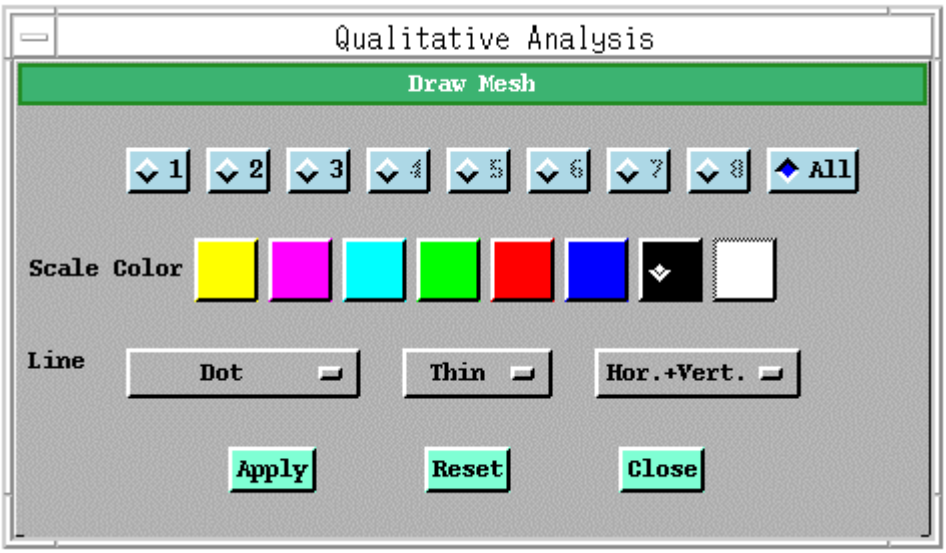

Fig. 31 Draw Mesh window

2. Select a spectrum number from the Draw Mesh window.
3. To specify **Scale Color** and **Line** for the grid, select the desired items from the following lists; then click on the **Apply** button to display the grid as desired.

- Line type

Solid:	Solid line
Dash:	Dashed line
Dot:	Dotted line
Dash Dot:	Dashed line with a dot
Dash Dot Dot:	Dashed line with two dots

- Thickness of the line

Thin:	Thin line
Normal:	Normal line
Thick:	Thick line

- Lines to draw

Hor. + Vert.:	Horizontal and vertical lines
Horizontal:	Horizontal lines
Vertical:	Vertical lines
None:	No lines

● Display Axis

You can set the unit for the abscissa of the specified spectra by using this function. The unit for the abscissa is always the same for all the spectra.

1. Select **Spectra Display**–**Display Axis** from the Operation menu.
The Display Axis window opens.

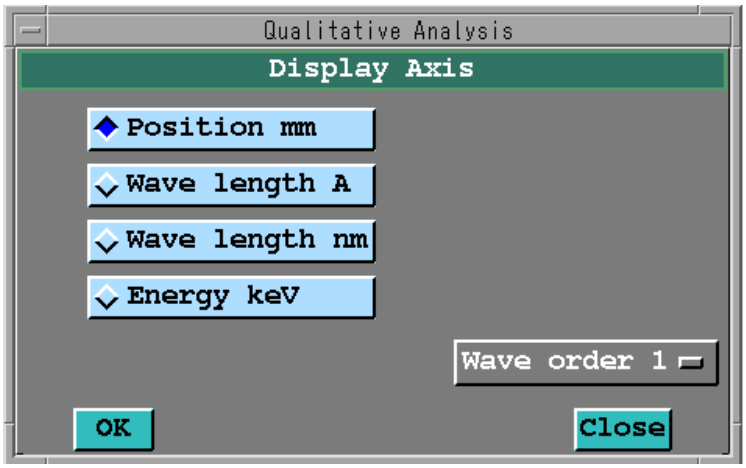

Fig. 32 Display Axis window

2. To specify the desired item in the Display Axis window, select **Position mm**, **Wave length A**, **Wave length nm** or **Energy keV**; then click on the **OK** button.
The abscissa for the spectra on the display changes as specified. **Wave order** is useful for displaying the spectra in the original wavelength when high-order lines are displayed in such narrow range as chemical shift. The function is effective only when the wavelength is specified as the abscissa.

● Kind of Assignment

You can select the style for displaying element labels by using this function.

1. Select **Spectra Display**–**Kind of Assignment** from the Operation menu.
The Kind of Assignment window opens.

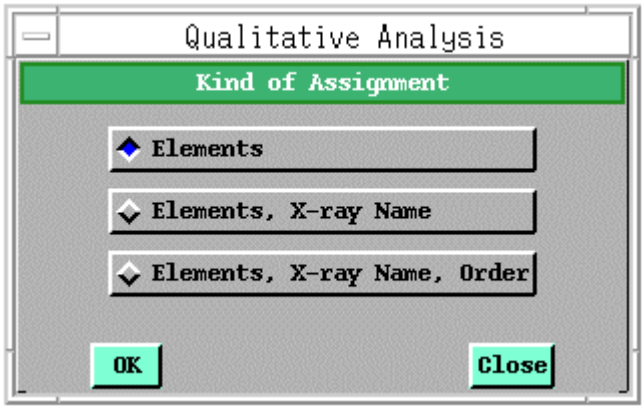

Fig. 33 Kind of Assignment window

2. Select **Elements**; **Elements, X-ray Name;** or **Elements, X-ray Name, Order**; then click on the **OK** button.

Elements: Only element names are displayed.

Elements, X-ray Name: Element names and X-ray names are displayed.

Elements, X-ray Name, **Order**: Element names, X-ray names and order are displayed.

● Display Parameter

You can specify which parameters to display in the parameter-display area of the Data display window, by using this function.

1. Select **Spectra Display–Display Parameter** from the Operation menu.
The Display Parameter window opens.

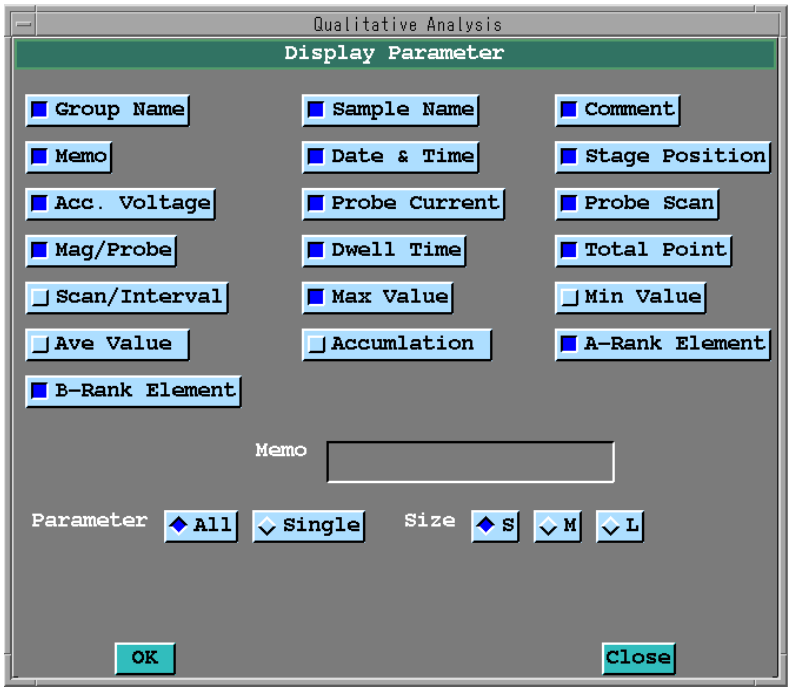

Fig. 34 Display Parameter window

2. Select the desired parameters; then click on the **OK** button.

The specified parameters are displayed in the parameter-display area of the Data display window.

- If you select **All**, the information of all the spectra is shown, while if you select **Single**, only the information of the specified spectrum is shown.
- To change the size of the displayed characters, select **S**, **M** or **L**.

An **A-Rank Element** is an element that is judged certain to exist, and a **B-Rank Element** is an element that is judged possible, though not certain, to exist.

For the method of element identification, refer to Chapter 5 “Appendix” of this instruction manual.

● Write Text

You can write text in the display area of spectra by using this function.

1. Select **Spectra Display–Write Text** from the Operation menu.

The Write Text window opens.

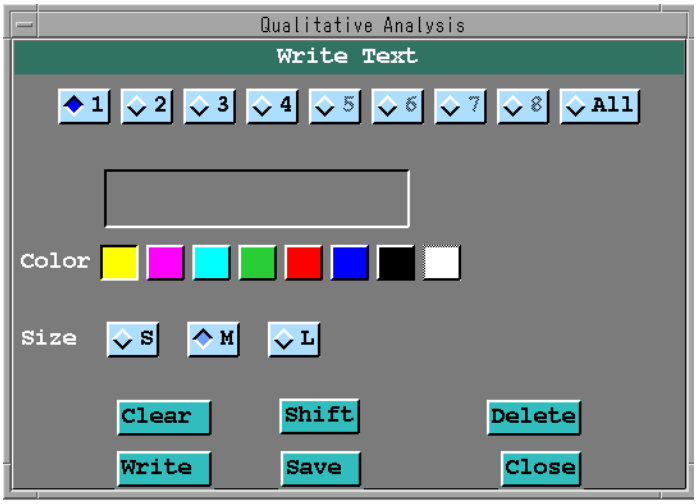

Fig. 35 Write Text window

2. Click on the spectrum on which you want to write text, and then input the desired text in the input box of the Write Text window; then click on the **Write** button.

The text is displayed on the selected spectrum of the Data display window.

Color: Selects the color of text.
Size: Selects the character size of text from S, M and L.
Clear: Clears the input box.
Shift: Adjusts the position of the input text when you drag it.
Delete: Deletes the text. Click on this button and then select the text that you want to delete from the list of texts.
Write: Writes text on the selected spectrum.
Save: Saves the texts that you have written on spectra. The texts will be displayed when you next open the spectra.

Replace Spectrum

You can replace the spectra that are being displayed by using this function.

1. Select **Spectra Display–Replace Spectrum** from the Operation menu.
   The Replace spectrum window opens.

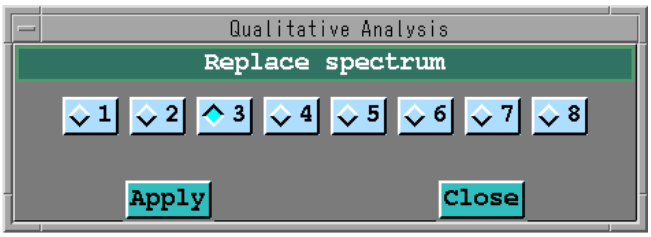

Fig. 36 Replace spectrum window

2. Select the number of the spectrum to replace from the Replace spectrum window, and then click on the **Apply** button.
   The Sample window opens.
   ☞ Refer to Fig. 19 Sample window (for processing).
3. Select the spectrum to replace from the Sample window.

Reset Spectra

You can reset all the changes that you have made using all the items of the **Spectra Display** menus to their initial states if you use this function.

1. Select **Spectra Display–Reset Spectra** from the Operation menu.
   The Reset Spectra window opens.

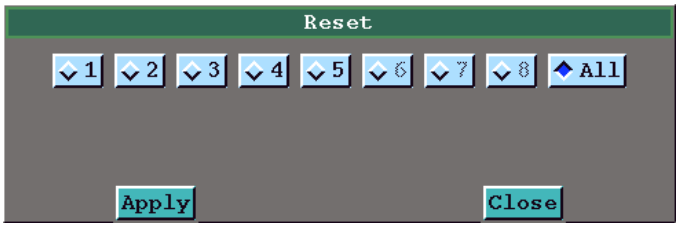

Fig. 37 Reset window

2. Select the number of the spectrum for resetting from the Reset window and then click on the **Apply** button.
   All the changes made for the selected spectrum will be cancelled.

KLM Marker

You can display identified elements in colors in the KLM Marker window.

1. Select **KLM Marker** from the **Operation** menu.

The KLM Marker window opens.

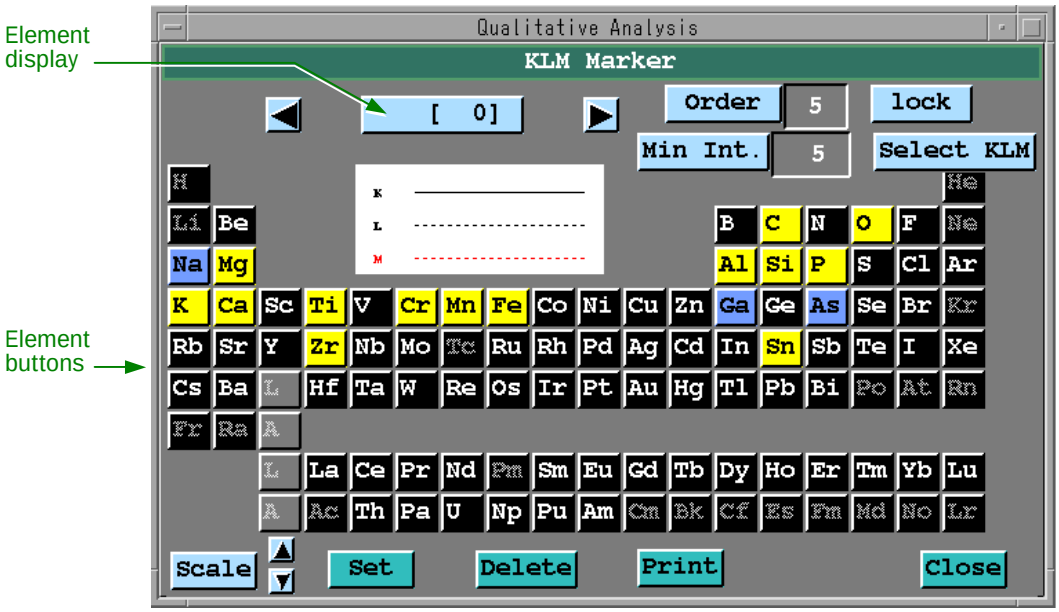

Fig. 38 KLM Marker window

2. Click on the buttons of the desired element in the KLM Marker window.

The positions and element names of the characteristic X-rays (Kα, Kβ, etc.) for specified elements are displayed on the spectrum.

The following functions are available.

Button	Function
Element buttons	Clicking on an element button displays KLM markers. When you right-click another element button, its KLM markers appear together with the previous ones. You can specify the line type for the KLM markers in the Spectrum Type window, and the color in the Spectrum Color window.
Element display	The name of the selected element is displayed. Right-clicking on the display shows the name of the element to the right of the present one, while left-clicking on the display shows the name of the element to the left of the present one.
Arrow buttons 	Clicking on the right arrow button selects the right element to the present one, while clicking on the left arrow button selects the left element to the present one.
Order	Specifies the order of the X-rays. You can specify from the first to the tenth.
Min Int.	Specifies the minimum intensity of the KLM Markers. The intensity to be specified is the theoretical intensity shown in the wavelength table. It means the relative intensity when the intensity of the alpha line is taken as 100. When you specify 0, all the X-rays are shown.

Button	Function
Select KLM	Selects the X-rays to be displayed from the K, L, and M lines. Only highlighted X-rays are displayed. It is useful for displaying specified X-rays.
Lock	To display the KLM Markers of multiple elements, specify elements by right-clickings (☞ refer to the above Element buttons). You can lock the KLM Markers if you click on the **Lock** button; then if you click on an element, you can display its KLM Markers together with the locked ones. To unlock the locked KLM Markers, deselect **Lock**.
Scale	The scale for each X-ray height of KLM Markers is based on the theoretical intensity. When an X-ray marker cannot be seen easily, you can adjust the height of the KLM Markers by using the arrow-buttons to the right of the **Scale** button. The same result can be obtained by dragging a point on the spectrum up or down.
Set	Adds the selected elements to the list of identification elements, and displays element names at the position of each KLM Marker. To label peaks, use the Peak ID window.
Delete	Deletes the selected elements from the list of identification elements as well as all the element names. When you want to delete element names one by one, use the Peak ID window.
Print	Prints the information on the KLM Markers.
Close	Closes the KLM window.

Peak ID

The wavelength and intensity at the specified position of the spectrometer are displayed. At the same time, the possible names of characteristic X-rays near the specified position are listed.

1. Select **Peak ID** from the **Operation** menu.

The Peak ID window opens.

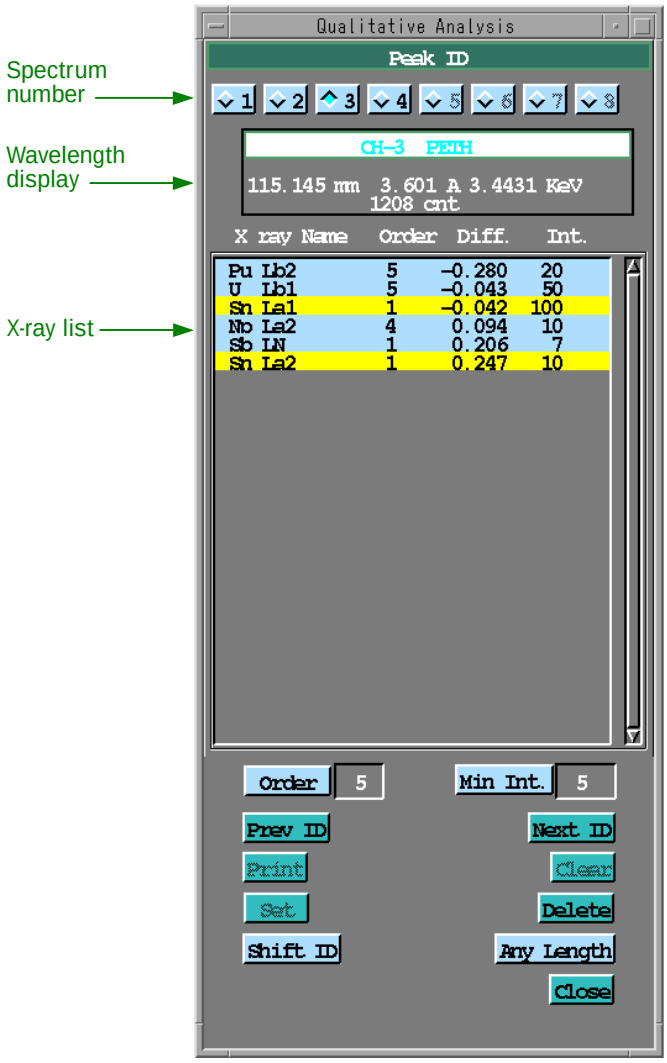

Fig. 39 Peak ID window

2. Select any position from a spectrum in the Data display window.

The wavelength and intensity at the specified position of the spectrometer are displayed in the X-ray list of the Peak ID window. At the same time, the possible names of characteristic X-rays near the specified position are listed. The identified elements and the elements corresponding to the displayed KLM Markers are displayed in different colors.

The following functions are available.

Object	Function
Spectrum number	Specifies the desired spectrum. You can also specify it by clicking on it.
Wavelength display	Displays the wavelength and X-ray intensity at a position specified by clicking. You can perform a fine adjustment of the cursor position by using the ← and → keys on the keyboard.
X-ray list	Displays the X-rays near the wavelength selected by clicking. The information displayed is Element Name, X ray Name, Order, Diff. (distance from the selected position) and Int. (theoretical X-ray intensity).
Order	Specifies the order of the X-rays to be detected.
Min Int.	Specifies the minimum theoretical intensity of the X-rays to be displayed.
Prev/Next ID	Move the cursor to the peak positions whose elements were already identified. The movement direction of **Prev ID** is toward lower wavelengths, and that of **Next ID** is toward higer wavelengths. The cursor jumps to the other end when it is moved beyond an end.
Print	Opens the Listing window and displays the data of the item selected from the X-ray list.
Clear	Clears the items that were specified for printing.
Set	After you have selected a line of an element in the X-ray list, clicking on the **Set** button identifies the element and displays its peak label.
Delete	After you have selected an identified element in the X-ray list, clicking on the **Delete** button deletes the peak label of the element.
Shift ID	Moves peak labels. After you have clicked on the **Shift ID** button, clicking near the label changes the shape of the cursor. Then, drag the label to move it. To deselect this mode, click on the **Shift ID** button once again.
Any Length	Measures the distance between two arbitrary points. After you have clicked on the **Any Length** button, click on two arbitrary points on a spectrum; then the marker and distance between the points are displayed. To deselect this mode, click on the **Any Length** button once again.
Close	Closes the Peak ID window.

■ Save ID Results

After you have changed the element name display on the spectrum by using **Peak ID**, for example, the element name can be saved as an A rank element.

1. Select **Save ID Results** from the Operation menu.
The Save ID Results window opens.

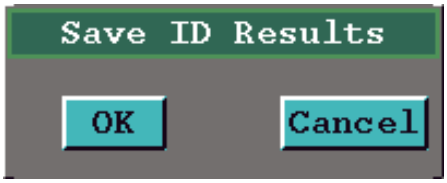

Fig. 40 Save ID Results window

2. Click on the **OK** button in the Save ID Results window, after you have changed the element name display on the spectrum by using **Peak ID**, for example.

The element name displayed on the spectrum will be saved as an A rank element in a file. These data will be displayed automatically beginning with the next operation.

■ Off-line ID

You can perform element identification once again.

1. Select **Off-line ID** from the **Operation** menu.

The Off-line ID window opens.

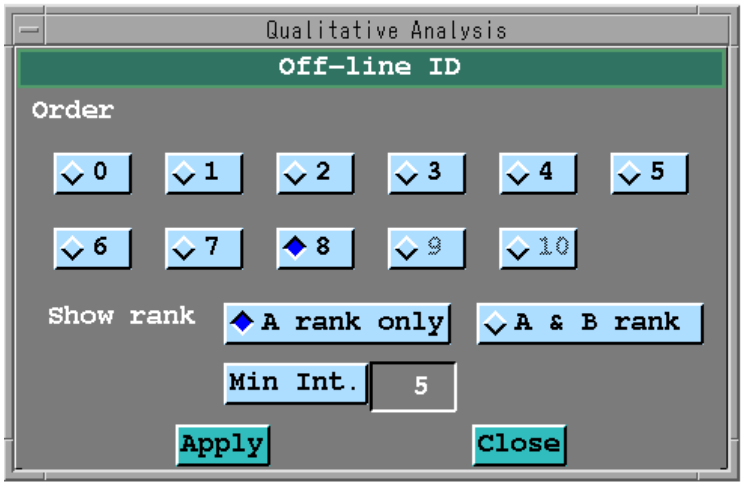

Fig. 41 Off-line ID window

2. Specify the necessary conditions; then click on the **Apply** button to perform element identification once again, after.

- The ID-Doctor does not operate during off-line identification of element.
- In element identification, the specified order of X-rays is taken into account, and if **A rank only** is selected, only the labels for A rank elements are displayed after the identification.
- If **A & B rank** is selected, all the labels are displayed after identification.
- If a number other than zero is specified in the **Min Int.** input box, the labels for only the X-rays having the specified intensity or more are displayed after identification.

When the **Close** button is clicked on with the **Order**–**0** button selected, with **Apply** not selected, the identified element names will not be displayed on the spectra in subsequent measurements.

Spectra Calculation

◆ Select **Spectra Calculation** from the **Operation** menu.

The Spectra Calculation window opens.

You can perform the arithmetic operations that are shown in the Spectra Calculation window.

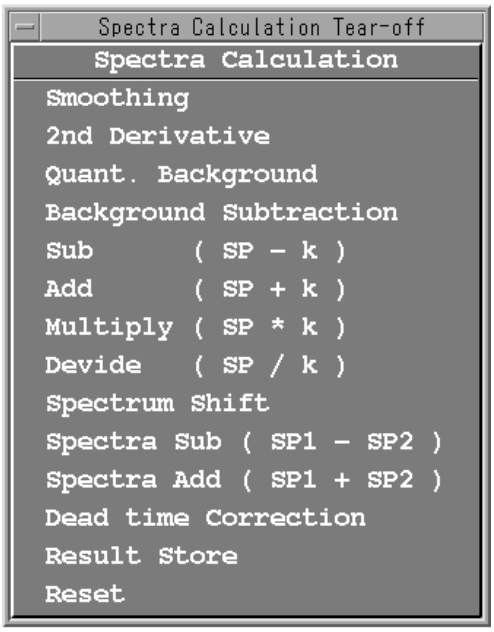

Fig. 42 Spectra Calculation window

Smoothing

◆ Select **Spectra Calculation–Smoothing** from the **Operation** menu.

The Smoothing window opens.

You can smooth the specified spectra using the Savitzky-Golay method.

- If you selected **Manual**, you can assign a number of points to use in smoothing.
- If you selected **Result Only**, only the smoothed spectra are displayed, whereas if you selected **Original & Result**, the spectra before smoothing are also displayed at the same time.

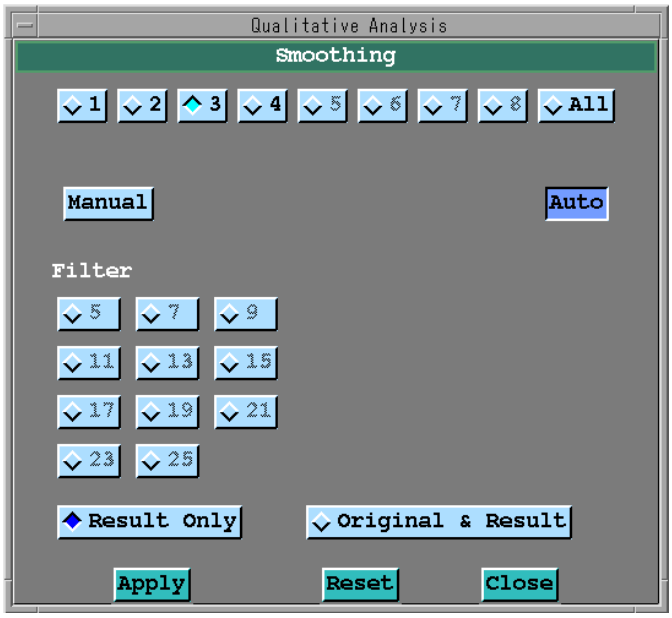

Fig. 43 Smoothing window

2nd Derivative

◆ Select **Spectra Calculation–2nd Derivative** from the **Operation** menu.

The 2nd Derivative window opens.

You can find the second derivatives of the specified spectra using the Savitzky-Golay method.

Quant. Background

You can display the background position that is used in the quantitative analysis, and correct the position while watching the corresponding spectra.

◆ Select **Spectra Calculation–Quant. Background** from the **Operation** menu.

The Quant. Background window opens.

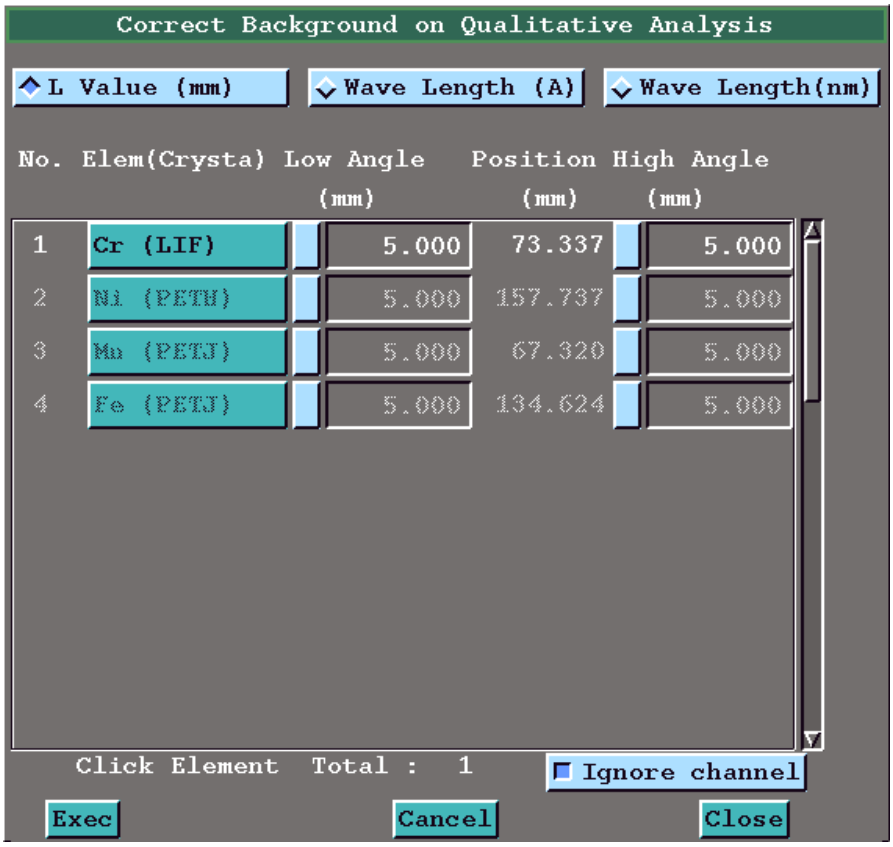

Fig. 44 Quant. Background window

- The information displayed is **No.**, **Elem (Crysta)**, **Low Angle**, **Position** and **High Angle**.
- You can select mm, angstrom, or nm as the unit.
- If an element name is dimmed, the corresponding spectra do not exist.
- If you have selected **Ignore channel**, you can search the spectra only by using crystal names. If you have deselected the **Ignore channel** button, you can search for spectra that match both crystal names and measuring channels.
- If you select any of the displayed elements, the background position will be shown on the spectra by markers. To correct the position, drag the markers to the desired positions.
- If you want to enter the corrected items, click on the **Exec** button, and if you do not want to, click on the **Cancel** button.

Background Subtraction

You can eliminate background components from the specified spectrum.

◆ Select **Spectra Calculation–Background Subtraction** from the **Operation** menu.

The Background Subtraction window opens.

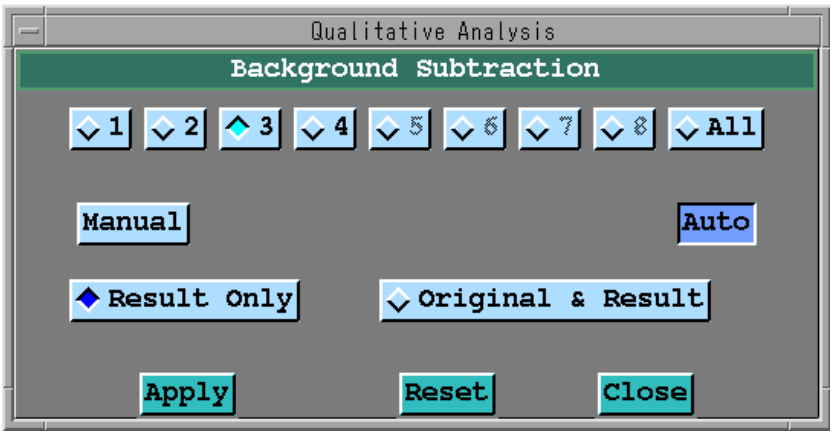

Fig. 45 Background Subtraction window

- Clicking on the **Manual** button performs spline interpolation for the specified points (normally, multiple points) and forms a background curve.
- Clicking on the **Auto** button performs background subtraction using the Sonneveld method.

Sub (SP – k)

You can subtract a constant (k) from the specified spectrum and display the spectra.

◆ Select **Spectra Calculation–Sub (SP – k)** from the **Operation** menu.

The Sub (SP – k) window opens.

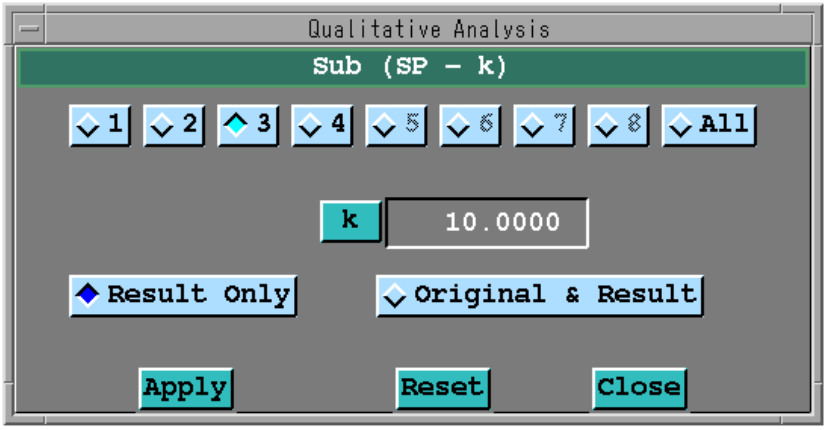

Fig. 46 Sub (SP – k) window

- **Add (SP + k)/Multiply (SP ∗ k)/Divide (SP / k)**

  You can add, multiply, or divide by a constant (k) in the specified spectrum and display the spectra.

  ◆ Select **Spectra Calculation**–**Add (SP + k)** or **Multiply (SP ∗ k)** or **Divide (SP / k)** from the **Operation** menu.

  The Add (SP + k)/Multiply (SP ∗ k)/Divide (SP / k) window opens.

- **Spectrum Shift**

  You can shift spectra to the left or right.

  ◆ Select **Spectra Calculation**–**Spectrum Shift** from the **Operation** menu.

  The Spectrum Shift window opens.

- **Spectra Sub (SP1 – SP2)**

  You can subtract two spectra.
  The result of the calculation can be displayed in another display area.

  ◆ Select **Spectra Calculation**–**Spectra Sub (SP1 – SP2)** from the **Operation** menu.

  The Spectra Sub (SP1 – SP2) window opens.

- **Spectra Add (SP1 + SP2)**

  You can add two spectra.
  The result of the calculation can be displayed in another display area.

  ◆ Select **Spectra Calculation**–**Spectra Add (SP1 + SP2)** from the **Operation** menu.

  The Spectra add (SP1 + SP2) window opens.

- **Dead-time Correction**

  You can perform dead-time correction.

  ◆ Select **Spectra Calculation**–**Dead-time Correction** from the **Operation** menu.

  The Dead-time Correction window opens.

  ☞ For the formulas for calculation, refer to the separate instruction manual of the Quantitative Analysis Program.

● Result Store

You can store displayed spectrum, such as the results of a calculation, in a file.

1. Select **Spectra Calculation**–**Result Store** from the **Operation** menu.
The Result Store window opens.

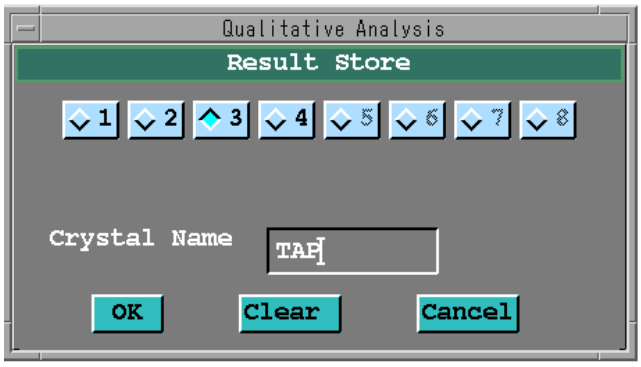

Fig. 47 Result Store window

2. Type a crystal name in the **Crystal Name** input box, and then click on the **OK** button.
The spectrum that you have selected will be added to after the spectrum that was stored last. At most twenty spectra can be stored.
If you want to delete a spectrum, select it, and type the slash (/) 8 times; the selected spectrum will be deleted.

● Reset

You can undo arithmetic operations executed in **Spectra Calculation**.

◆ Select **Spectra Calculation**–**Reset** from the **Operation** menu.
The settings will returned to their initial states.

■ Redraw Spectra

You can display spectra again.

◆ Select **Redraw Spectra** from the **Operation** menu.

Print-out

You can print conditions and results.

1. Select **Print-out** from the **Operation** menu.
   The Print-Out window opens.
2. Select the items that you want to print, and then click on the **OK** button.
   The Listing window appears.
3. Click on the **Print** button in the Listing window.
   The output will be sent to the printer.

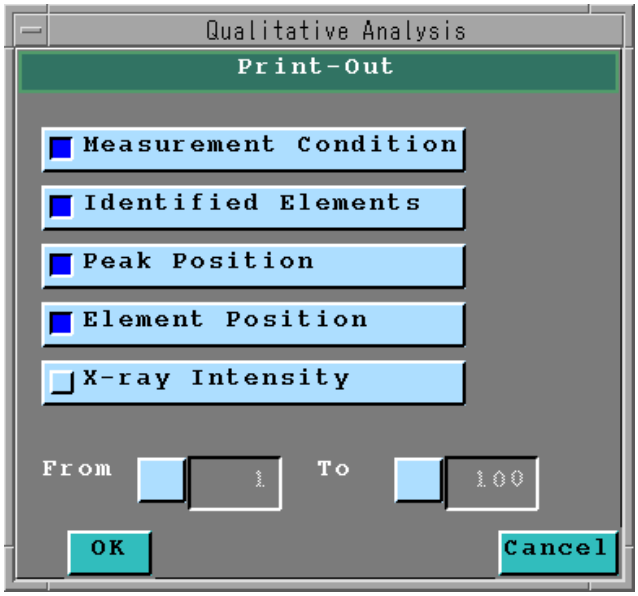

Fig. 48 Print-out window

Semi-Quant Analysis

After you have obtained background intensity from an identified element and a qualitative spectrum, you perform a calculation for correction based on the ratio to the X-ray intensity of a pure element (the K ratio) so as to obtain the result of a quantitative analysis. Below is the method for calibrating the X-ray intensity of a pure element.

◆ Select **Semi-Quant Analysis** from the **Operation** menu.

The Semi-Quant Analysis window opens.

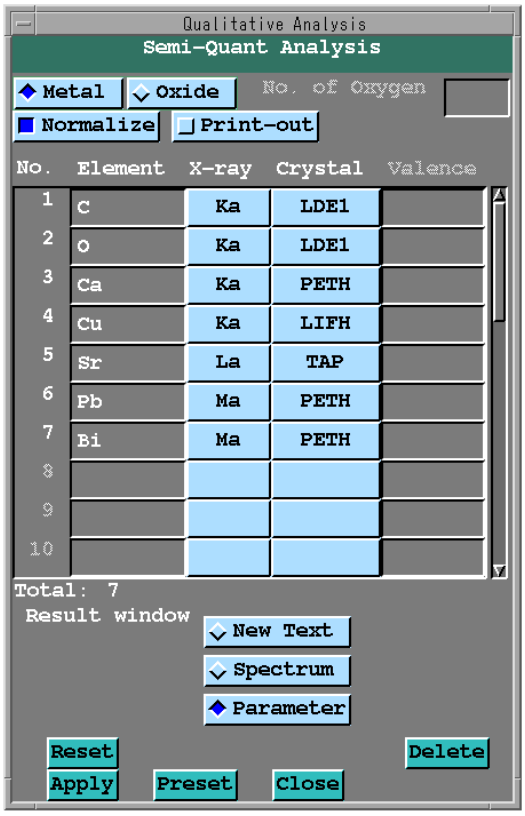

Fig. 49 Semi-Quant Analysis window

This window displays a list of the element names (in **Element**), X-ray names (in **X-ray**), and analyzing crystals (in **Crystal**) for the A-rank identified elements whose spectra show the strongest X-ray intensities.

● Metal/Oxide

When the sample to be analyzed is a metal, click on the **Metal** button. When the sample is an oxide, click on the **Oxide** button; then enter values into the **No. of Oxygen** and **Valence** input boxes.

● No. of Oxygen input box

You can input a value when **Oxide** is selected. Type the number of oxygen atoms in the input box. The number is to be used as the standard when the molecular formula is calculated after correction.

Normalize

Clicking on this button normalizes the mass concentration after correction; the total mass concentration becomes 100%. If you do not click on the **Normalize** button, the current values are displayed as they are.

Element, X-ray, Crystal and Valence in the list

Button	Function
Element	Adds and deletes arbitrary elements.
X-ray	Specifies the type (Kα, Kβ, Lα, Lβ, Mα, and Mβ) of the X-rays in the scan range.
Crystal	Specifies one of the analyzing crystals displayed in the column when the characteristic X-rays span multiple spectra.
Valence	Specifies the valence of oxides. The mass concentration of oxygen can be obtained by using the valence.

Result window

You can specify where to display the results that you obtain.

Button	Function
New Text	Opens a text window, in which the results are displayed.
Spectrum	Displays the results in the spectrum display area if it is open.
Parameter	Displays the results in the parameter-display area.

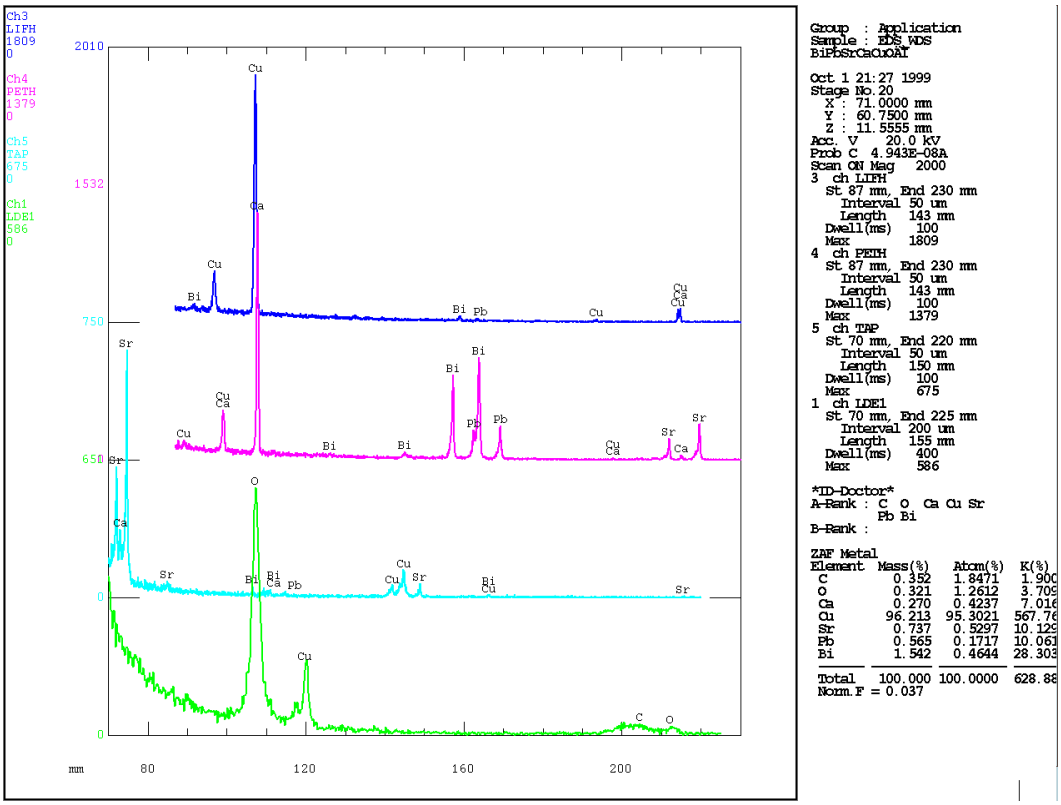

Fig. 50 An example of Semi-Quant Analysis results

■ ID-Doctor

You can perform element identification based upon the knowledge base of experienced analysis specialists. If **ID-Doctor** is selected, the element identification under **ID-Doctor** will be executed automatically after measurement.

1. Select **ID-Doctor** from the **Operation** menu.

The ID-Doctor window opens.

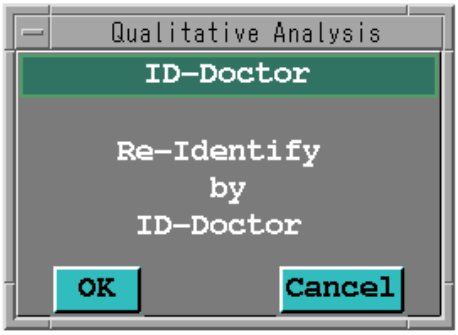

Fig. 51 ID-Doctor window

2. Click on the **OK** button.

☞ For details, refer to Chapter 5, “Appendix”.

Spectrum Analysis

You can display on a spectrum the position, height, area, FWHM (full width at half maximum) and height from the baseline, of the specified spectrum peak. You can print the spectrum, after the spectrum analysis, by using the **Print** button.

1. Select **Spectrum Analysis** from the **Operation** menu.
The Spectrum Analysis window opens.

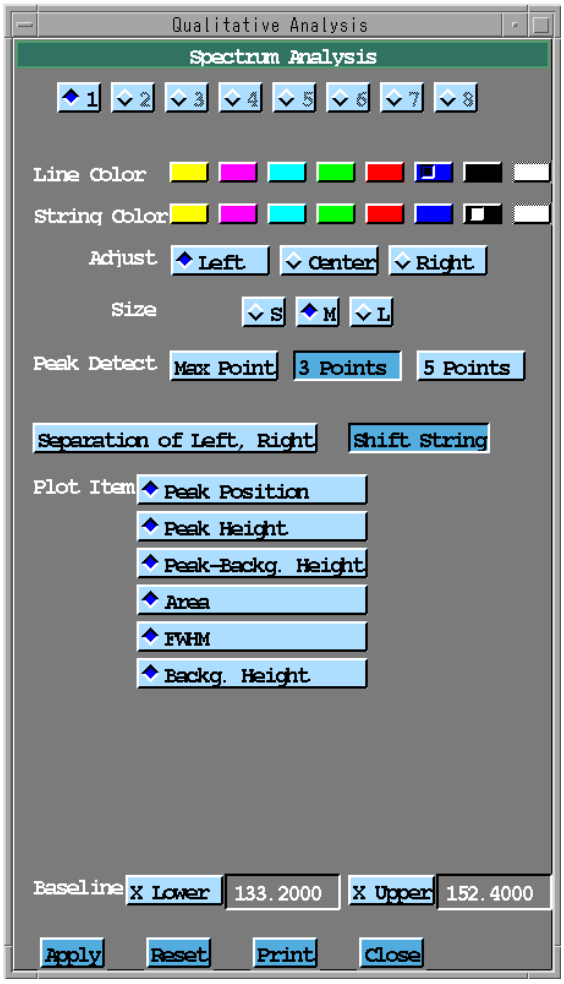

Fig. 52 Spectrum Analysis window

2. Specify the baseline between any two desired points by dragging the mouse from the start point and dropping it at the finish point.

3. Click on the **Apply** button.
The results of Spectrum Analysis will be displayed on the spectrum.

- If you enlarge a desired part of a spectrum by using **Spectra Display–Zooming** and specify the desired baseline between two points, you can obtain the baseline with greater precision.
- You can also specify the baseline by inputting values in **Baseline X Lower** and **X Upper** input boxes.
- You can select the following items.
  Line color: Color of lines

String Color:	Color of text
Adjust:	Place in which text is to be printed, selected from **Left**, **Center** and **Right**
Size:	Size of text, selected from **S**, **M** and **L**
Peak Detect:	Peak position selected from **Max Point**, **3 Points** and **5 Points**
Plot Item:	Items to be displayed
Shift String:	Lets you shift the text already written by dragging it.

4. Turn on the **Separation of Left, Right** button, and then click on the **Apply** button.

You will obtain different results on the left side and right side of the peak position.

■ Spectra Search

Spectra Search has two important functions. One is a search function. The other is a load function. Using the search function, you can display average correlation and other information about spectra judge to be similar in the Search result scroll window by using correlation coefficients from the start position to the finish position for the same crystal. Using the load function, you can also display the spectra that you have selected from the Search result scroll window.

The correlation coefficient between a spectrum and another one is 1.0 if the two are the same, while the closer the coefficient is to 0.0, the less similar the two are. The correlation coefficient obtained by calculation will be the same even if one or both are multiplied by a constant. If the correlation coefficient is about 0.9 or more, two spectra are similar experimentally. However, it is recommended that you judge similarity from the display by using the load function.

1. Select **Spectra Search** from the **Operation** menu.

The Spectra Search window opens.

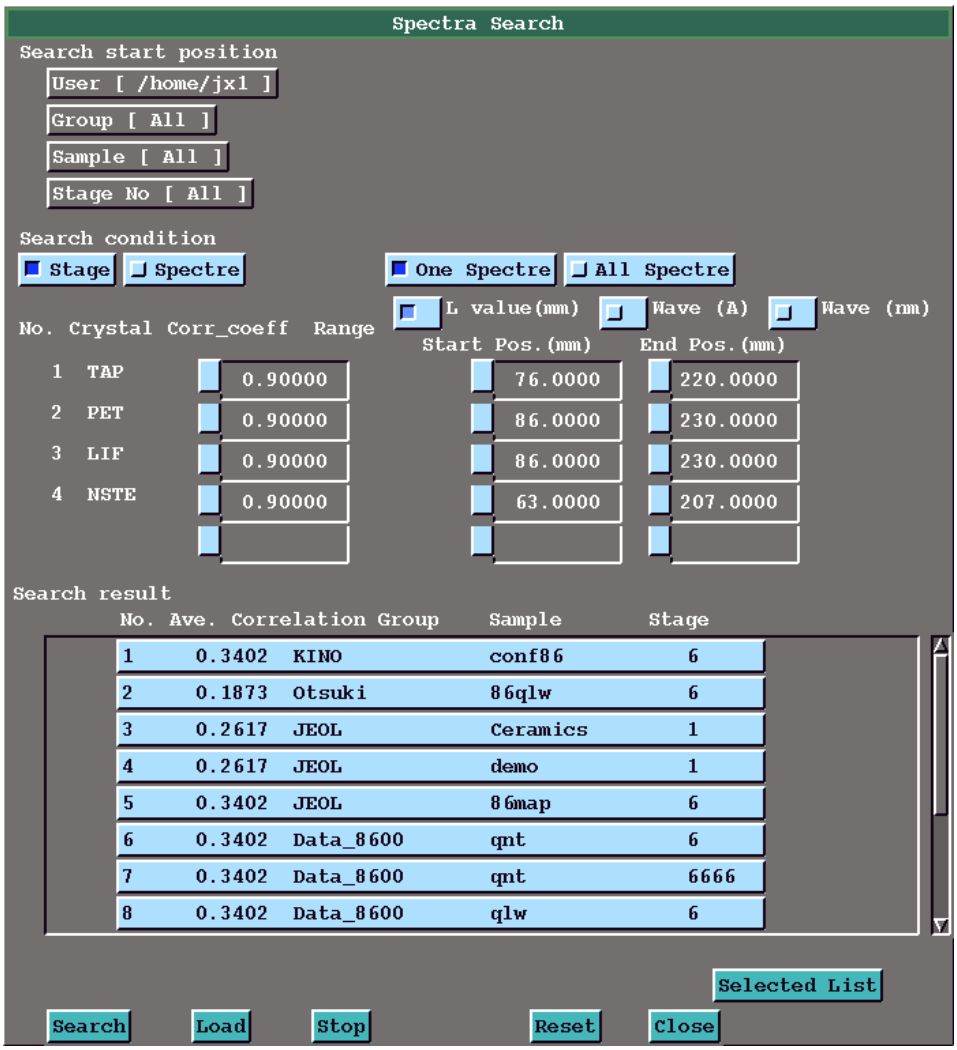

Fig. 53 Spectra Search window

Button	Function
Search start position– **User** **Group** **Sample** **Stage No**	Specifies search start directory. If there are many spectra for qualitative analysis, specifythe start position by using the **Group** button to save search time. Clicking on the **Group** button opens the Group window, where you can specify group names. In the same way, clicking on the **Sample** button opens the Sample window, where you can specify sample names.

Fig. 54 Group window

Button	Function
Search condition –Stage/Spectra	Selecting **Stage** performs searches through samples. Selecting **Spectra** performs searches through spectra.
One Spectrum/ All spectra	When **Stage** is selected, this function is available.
L value (mm)/ Wave (A)/Wave (nm)	Used when you specify the search range.
Corr_coeff	Correlation coefficient from 0.0 to 1.0.
Start Pos./ End Pos.	Start position for calculation of correlation coefficient/ Finish position for calculation of correlation coefficient. You can change the positions.
Search result	Scroll window to display the results of search.
Search	Starts search.
Load	Displays the spectrum selected from the Search result scroll window.
Stop	Stops search.
Reset	Resets correlation coefficient, and the values of **Start Pos.** and **End Pos**. Clicking on this button also resets the spectrum presently displayed to the previous one.
Selected List	Click on this button to print the results of the search. You can print only the spectra that you have selected from the Search result scroll window.
Close	Resets the spectra that are presently displayed, and closes the Spectra Search window.

4.4 Semi-Quantitative (Semi-Quant) Analysis Program

In ordinary quantitative analyses, quantitation results are corrected by measuring the intensities of peaks and backgrounds for each element. Differing from the ordinary quantitative analyses, the semi-quantitative analysis (Semi-Quant Analysis) program corrects quantitation results by using the spectral data. Namely, the intensities of the peaks and backgrounds for the elements identified in the qualitative analysis are obtained from the qualitative-analysis spectral data.
The ratio of the spectral peak intensity for each element to that of the characteristic X-rays for the pure element is calculated as the K-ratio.

K-ratio = Iunk/Istd

where

Iunk: Intensity of the characteristic X-rays from an unknown sample
Istd: Intensity of the characteristic X-rays of a pure element

Based on this K-ratio, ZAF correction is performed, and the mass concentration of each element is printed together with the K-ratio.
This result can be used later to perform an off-line quantitative analysis (Summary), or it can also be recorrected by using the measured standard sample data.
The characteristic X-ray intensities of pure elements are calculated from the standard sensitivity curve obtained in advance. At that time, since their dependence on the accelerating voltage is also calculated, the K-ratio for the qualitative analysis data measured from 5 kV to 30 kV (in 5 kV steps) can be calculated.
The standard sensitivity curves have been measured in our company for a large number of standard samples. However, there may exist slight differences in the measured curves, depending on the instruments used. Therefore, using some standard samples, measure and correct the standard sensitivity curves of some elements for each analyzing crystal and for each characteristic X-ray (Kα, Lα, and Mα). Once this correction has been done, you need not repeat it each time you perform analysis.

4.4.1 How to calculate X-ray intensities in the Semi-Quant Analysis

X-ray intensities in the Semi-Quant Analysis are calculated in the following way.
As the peak position, the position at which the X-ray intensity is the strongest is selected after searching for it in the vicinity of the aimed characteristic X-rays.
As the background position, the position at which the mean X-ray intensity having the lowest moving average of three points from a position a little apart from the peak position is selected.
These results are displayed as measurement conditions. The search ranges of the peaks and backgrounds are defined for each analyzing crystal in the **sqt.cond** file in the **/opt/epma/conf/anal** directory.
The results of the Semi-Quant Analysis are saved in the file named **1.onq** in the directory of the quantitative analysis.

4.4.2 Using Semi-Quant Analysis results in off-line quantitative analysis

The results of the Semi-Quant Analysis can be utilized in the off-line quantitative analysis.

■ Summary

You can execute Summary in the Semi-Quant Analysis.

1. Select **Process**–**Quantitative Analysis**–**Summary** from the EPMA Main Menu.
   The Function window for process opens.
2. Click on the **Sample** button.
   The Select Sample window opens.
3. Select **Qnt** for Semi-Quant Analysis.
   The remaining steps are the same as for ordinary quantitative analysis.

✍ In the Semi-Quant Analysis, elements are different for each sample in many cases. If you print the results of many samples, it will be difficult to distinguish them, since there are many elements. To solve this problem, use **Mass order** for printing.

✍ If you select **Qlw** in place of **Qnt**, you can obtain the list of identified elements when you have printed it.

■ Off-line Correction

You can execute Off-line Correction of the analysis results obtained in the Semi-Quant Analysis.

1. Select **Process**–**Quantitative Analysis**–**Off-line Correction** from the EPMA Main Menu.
   The Function window for off-line correction opens.
2. Click on the **Sample** button.
   The Select Sample window opens.
3. Select **Qnt** for Semi-Quant Analysis.
4. Set the conditions as follows:
   - Specify the correction method and accelerating voltage used for measurement.
   - Specify the same elements as those used in the Semi-Quant Analysis.
   - Select the same X-rays, channel, and analyzing crystal as in the result of the Semi-Quant Analysis for the element to be measured.
   - Select appropriate standard samples, if any. When **Cal-STD** is selected as the standard sample for all elements, the same result as in the Semi-Quant Analysis is obtained.

☞ For procedures for entering the conditions for the quantitative analysis, refer to the separate operation manual of the Quantitative Analysis Program.

After setting the above conditions, click on **Read Intensity & Correction**, and the X-ray intensities obtained in the Semi-Quant Analysis will be displayed. Then, click on the **Apply** button, and off-line correction will be executed.

4.4.3 Correcting the standard sensitivity curve

The Semi-Quant Analysis program includes a program for correcting the standard sensitivity curve. By using the standard sample installed in the microanalyzer, the X-ray intensity coefficient (I) in the equation, in which the atomic number is used as the variable, can be obtained for each analyzing crystal and for each characteristic X-ray (Kα, Lα, and Mα) of each X-ray spectrometer. Once this program has been executed, you need not run it again.

$$I = \exp (a + bZ + cZ^2 + dZ^3$$

where

I: Intensity of characteristic X-rays (cps/100 pA)
Z: Atomic number
a, b, c, d: Coefficients

Combinations of the standard sample and characteristic X-rays to be used are defined in a file in the **/opt/epma/cali/sqt** directory. The coefficients obtained are saved in the **sqt.coef** file in the **/opt/epma/cali** directory together with the measured data. The characteristic X-ray intensities of pure elements for each analyzing crystal and for each accelerating voltage are saved in a file in the **/opt/epma/cali/std_int** directory.
Follow the procedures in the sections below to execute correction of the standard sensitivity curve.

■ Opening the Semi-Quant Calibration window

◆ Select **Initialize–Semi-Quant Calibration** from the EPMA Main Menu.

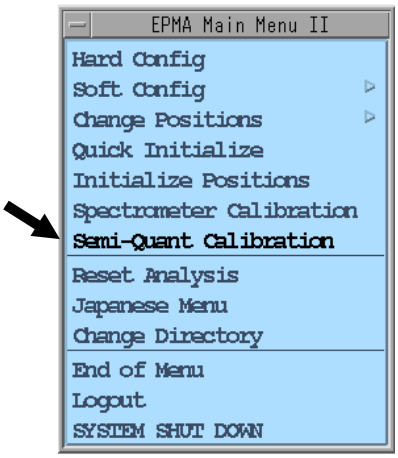

Fig. 55 Pull-down menu of the Initialize icon

The Semi-Quant Calibration window opens. You can perform corrections in this window.

Selecting analyzing crystals

Select analyzing crystals whose standard sensitivity curves are to be corrected.

◆ Click on the **Crystal** button in the Semi-Quant Calibration window.

The Crystal and Channel window opens.

If necessary, click on the **for Light elements** button in the Crystal and Channel window; then the Light Elements window opens.

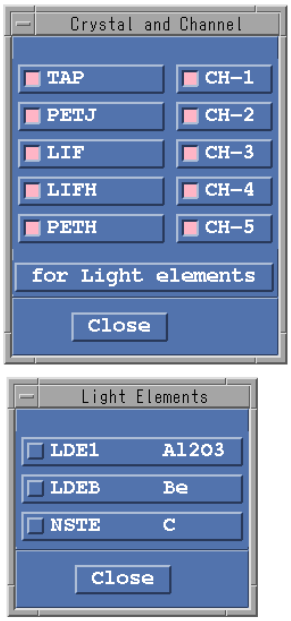

Fig. 56 Crystal and Channel and Light Elements windows

Out of the analyzing crystals installed in the instrument, the analyzing crystals whose standard sensitivity curves can be corrected will be displayed in the Crystal and Channel and Light Elements windows.

By default, all the crystals are selected. If there are some analyzing crystals whose standard sensitivity curves you do not want to correct, deselect their crystal buttons.

Setting the stage positions for standard samples

You have to enter the stage positions for standard samples prior to measurement.

1. Click on the **Stage** button in the Semi-Quant Calibration window.

The Set Stage, Stage Monitor and Memory windows open.

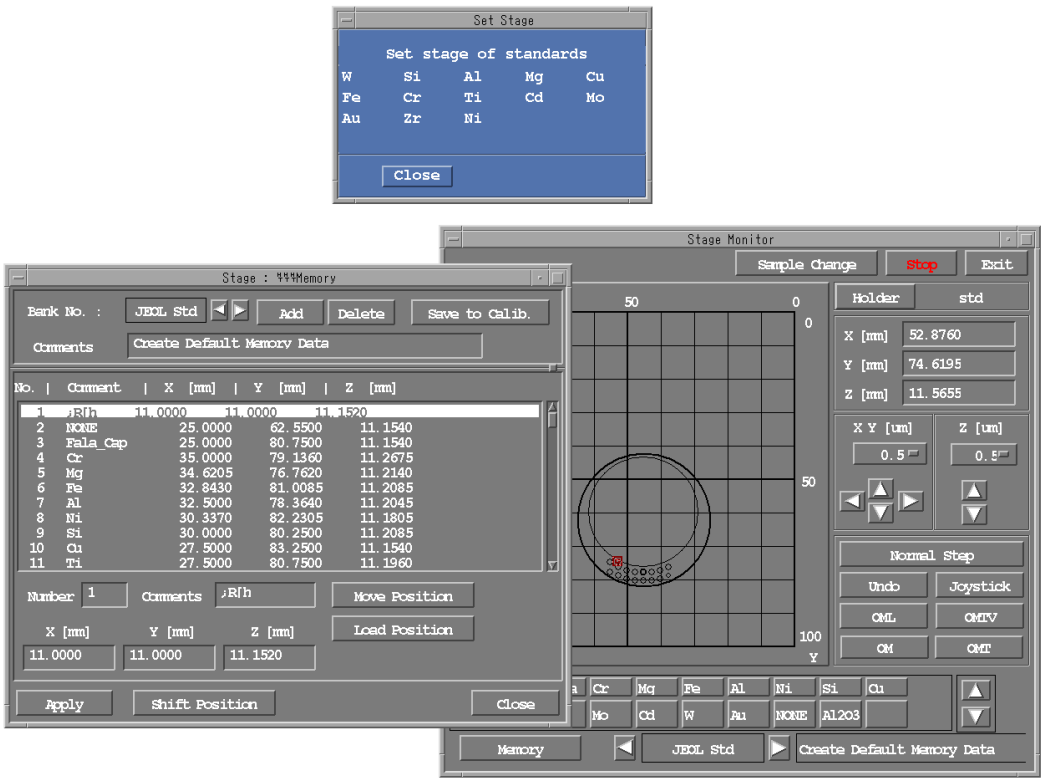

Fig. 57 Specifying the stage positions for standard samples

2. Specify the desired standard sample names and their stage positions.

If stage positions have been already set, you need not specify them.

For the operating procedure, refer to the instruction manual of the main unit.

Executing measurements

You execute measurement by using the following procedure.

1. Click on the **Measure** button in the Semi-Quant Calibration window.

The Start Calibration window opens.

2. Set the current to about 1×10^{-7} A in advance.

Although the accelerating voltage is automatically set to 20 kV, but the current is not set automatically.

3. Confirm all the conditions that you require to use; then click on the **OK** button.

Measurement begins.

If you click on the **Cancel** button, the previous window will return.

Measurement is performed for each analyzing crystal, and the progress of the measurement is displayed on the screen.

Stopping measurement

- To stop measurement for any reason, click on the **Stop** button.
- To temporarily interrupt measurement, click on the **Pause** button. On completion of peak search, measurement stops temporarily. To restart the measurement, click on the **Pause** button once again.

Displaying measurement results

On completion of measurement with an analyzing crystal, the correction coefficients and the calculated intensities of the characteristic X-rays of the pure elements are displayed. The coefficients are displayed in units of cps/100 pA. The X-ray intensities for Kα, Lα, and Mα are displayed (vertical axis: logarithmic scale in units of cps/μA, horizontal axis: atomic number).

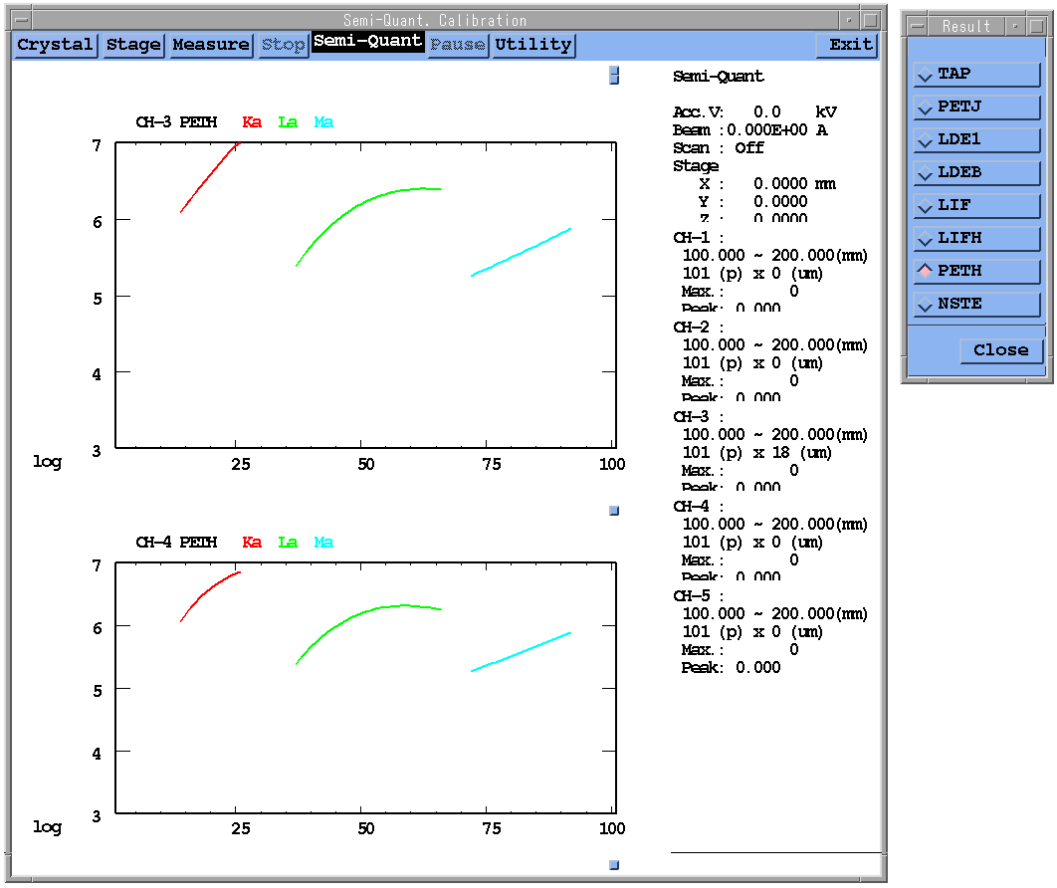

Fig. 58 Display of measurement results

Utility

◆ Click on the **Utility** button in the Semi-Quant Calibration window.

The Utility pop-up window opens.

The following functions are included in **Utility**.

Button	Function
Reset spectra	Individual spectrum windows can be enlarged or reduced by dragging the button at the lower right corner of each window. Clicking on **Reset spectra** after enlarging or reducing the window will return it to the original size.
Draw results	Clicking on this item displays results of measurement on the computer display.
Print results	Clicking on this item prints the measured data and the intensity coefficients of the standard sensitivity curve.
Check configuration	Clicking on this item enables you to check the combination of the analyzing crystal and characteristic X-rays in measurement.
Check pksk parameter	Clicking on this item enables you to check the parameters for peak search. Special care should be exercised in changing the parameters because the operation affects peak search and related operations in quantitative analysis.
Smoothing	Clicking on this item smoothes and displays the waveform for peak search.
2nd derivative	Clicking on this item displays the second derivative waveform of the peak search spectrum.
Log scale	Clicking on this item displays the peak search spectrum on a logarithmic scale.

5 APPENDIX

■ Determining peak positions and intensities, and identifying elements

Using the procedures below, this program detects the peaks of characteristic X-rays and identifies the elements for the detected peaks.

● Detecting peak positions

1. Smooth a spectrum by the Savitzky-Golay method.
2. Evaluate the second derivative of the smoothed spectrum.
3. Detect the peak positions from the second derivative waveform and determine the background positions from the waveforms obtained after second differentiation and smoothing.
4. From the smoothed spectrum, determine the peak intensity and the background intensity.
5. If the difference between the peak intensity and the background intensity is too large to attribute to statistical variation, judge that a peak of interest exists.

● Identifying elements

1. Detect peaks for each spectrum.
2. If any one of the Kα (1), Lα (1) and Mα (1) lines is detected, assign the elements of interest to the B rank.
3. Further judge the elements of B rank as follows:
   - **a.** If ZKα min ≤ Z ≤ ZKβ min, assign the elements of interest to the A rank.
     Usually, ZKα min=4, ZKβ min=17.
   - **b.** If both a Kα (1) line and a Kβ (1) line are found in the same spectrum, assign the elements of interest to the A rank.
   - **c.** If both a Kα (1) line and a Kβ (1) line are found in different spectra, assign the elements of interest to the A rank.
   - **d.** If both an Lα (1) line and an Lβ (1) line are found in the same spectrum, assign the elements of interest to the A rank. Similarly, if both an Mα (1) line and an Mβ (1) line are found in the same spectrum, assign the elements of interest to the A rank.
4. If any peaks of B-rank elements are superimposed on any peaks of characteristic X-rays of A-rank elements (usually, of the first-order), exclude the B-rank elements from the elements to be identified.
5. If no A-rank element exists but only one B-rank element exists, assign this B-rank element to the A rank.

Expert identification of elements

- This expert identification of elements is a reliable method of ratiocination based upon the knowledge base of experienced analysis specialists.
- This method enables us to evaluate a level of confidence in the result of the element identification performed by using the above-mentioned element identifying method, if it is applied for calculation.
- The knowledge base has been created under the following conditions

Accelerating voltage:	20 kV
Probe current:	10 to 500 nA
Step:	50 μm
Sample time:	50 ms or more
Number of spectra:	4
Analyzing crystal:	LIF, PET, TAP, and STE
Scanning range of spectrometer:	65 mm to 250 mm (258 mm for TAP)

  Even if you cannot observe under the above conditions to some extent for some reasons, you can obtain good results since this program is created to avoid problems. Of course, it is best to meet the requirements as much as possible. However, when you specify elements to be identified, set the spectrometer range to include the first-order X-rays (up to the second order, if possible) of α and β of K, L, and M of the elements.
- In the results after execution of **ID-Doctor**, the indication * ID-Doctor * is shown both at the area where elements are displayed in the Data Display window and just above the area where A-Rank and B-Rank elements are displayed. Therefore, you can easily distinguish the results obtained with the **ID-Doctor** from those obtained without the **ID-Doctor**.
- The element identification executed by using this program is performed using the judgment method that imitates experienced analysis specialists; the results are not absolutely correct. Please use this **ID-Doctor** as a reference and keep it in mind that you, the user of this program, decide whether to accept the results or not.