

XM-27330

EDS QUALITATIVE ANALYSIS PROGRAM

For the proper use of the instrument, be sure to read this instruction manual. Even after you read it, please keep the manual on hand so that you can consult it whenever necessary.






IXM-27330-1QL (608501)

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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 is intended for acquiring a spectrum of an unknown sample using an energy-dispersive spectrometer (EDS), identifying the elements in the spectrum, and performing semi-quantitative analysis.

Spectrum acquisition can be specified as live time or real time, or as a count in the specified ROI. When measurement is completed, element identification takes place automatically based on artificial intelligence, and element names are appended to the peak positions. Also, semi-quantitative analysis takes place automatically, enabling the composition of the unknown sample to be determined as well. After the measurement ends, the spectrum is displayed, semi-quantitative analysis in which the KLM marker, peak ID and elements are specified becomes possible, and measurement data are printed.

2 SPECIFICATIONS

EDS acquisition conditions

Energy full scale:	20 keV
Number of spectral data points:	2 kch
Measurement time:	1 to 10,000 seconds
Aperture number designation:	1 to 6 (1: open; 6: closed)

Number of measurement preset points per sample:

1 to 10,000

Number of accumulations: 1 to 100

Real-time display during measurement: Possible

Automatic element identification during measurement:

Possible based on artificial intelligence

KLM marker: Can be displayed*

Spectrum peak identification: Possible*

Off-line element identification: Possible*

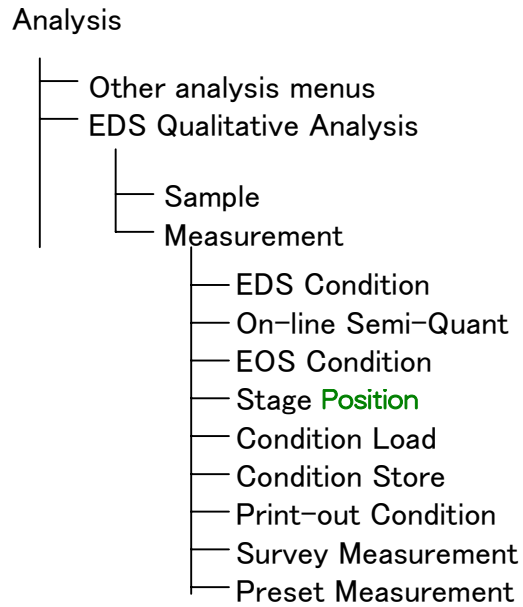
Semi-quantitative analysis: Possible, up to 30 elements

Spectrum search: Enables similar spectrum to be found.*

* These functions are included in the EDS operation program. Before executing them, refer to the separate instruction manual, "EDS Operation Program".

3 PROGRAM CONFIGURATION

As shown below, this program has a tree configuration. Each menu item can be selected using the mouse, enabling you to execute procedures and select items beneath the hierarchy.



4 OPERATION

The general measurement procedure is set out below.

Display the EDS home window from the EDS.

First, try acquiring an EDS spectrum of the sample to be measured. When doing this, set the sample and the aperture so that the dead time is between 20 and 30%.

Next, set the sample name and the measurement positions, and perform measurement.

These conditions can be saved in a file in advance, and subsequently recalled and executed. Also, when performing measurement you can specify semi-quantitative analysis, and determine the composition of the sample as well as perform element identification.

The detailed procedure for carrying out EDS qualitative analysis is described in sequence below. Note that in EDS qualitative analysis it is necessary to operate the EDS Home Window.

☞ Refer to the separate instruction manual, “EDS Operation Program”.

4.1 Measurement Preparations

4.1.1 Displaying the EDS Home Window

1. Open the EPMA Main Menu on the computer display and then click on the **EDS** icon.

The EDS menu appears as shown in Fig. 1.

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

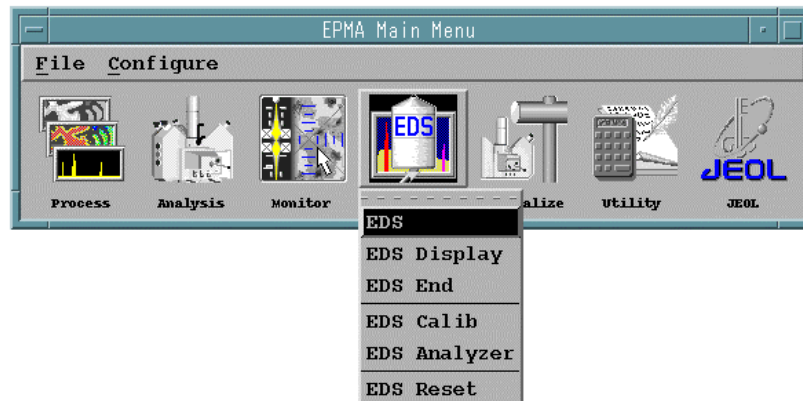


Fig. 1 EPMA Main Menu and EDS pull-down menu

2. Select **EDS** from the EDS menu.

The EDS Home Window appears as shown in Fig. 2.

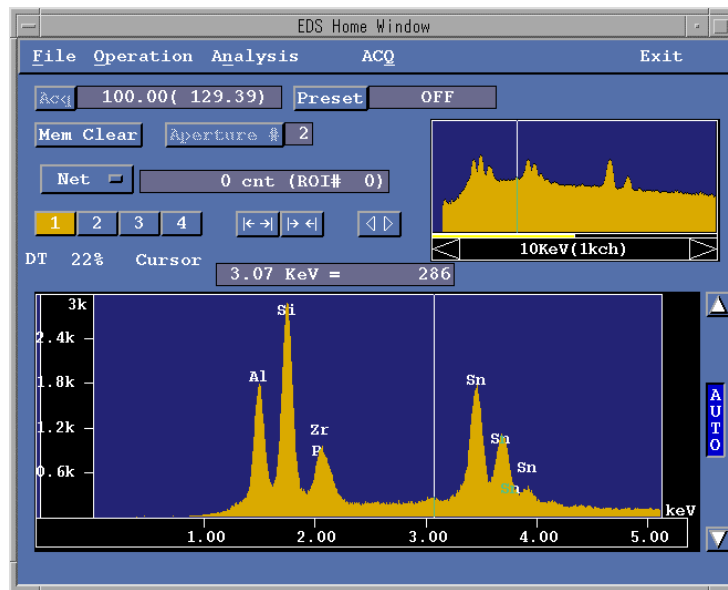


Fig. 2 EDS Home Window

4.1.2 Displaying the EDS Qualitative Analysis function window

1. Click on the **Analysis** icon in the EPMA Main Menu.
The **Analysis** menu appears.
☞ Refer to the instruction manual of the microanalyzer main unit to learn how to open the EPMA Main Menu.
2. Select **EDS Qualitative Analysis** from the **Analysis** menu.
The EDS Qualitative Analysis function window opens as shown in Fig. 3.

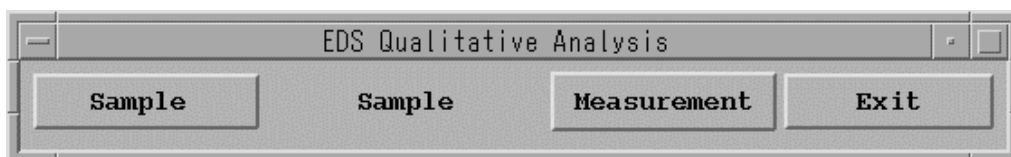


Fig. 3 EDS Qualitative Analysis function window

4.1.3 Setting the group name and the sample name

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 a name under which a group of samples is organized, and it will be convenient to name it after the property of a series of samples or the operator for easier arrangement and filing.

1. Click on the **Sample** button in the EDS Qualitative Analysis function window.
The Select Sample window opens as shown in Fig. 4.
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 in use and the amount of free space at present are shown in kilobytes (kB) in the window.

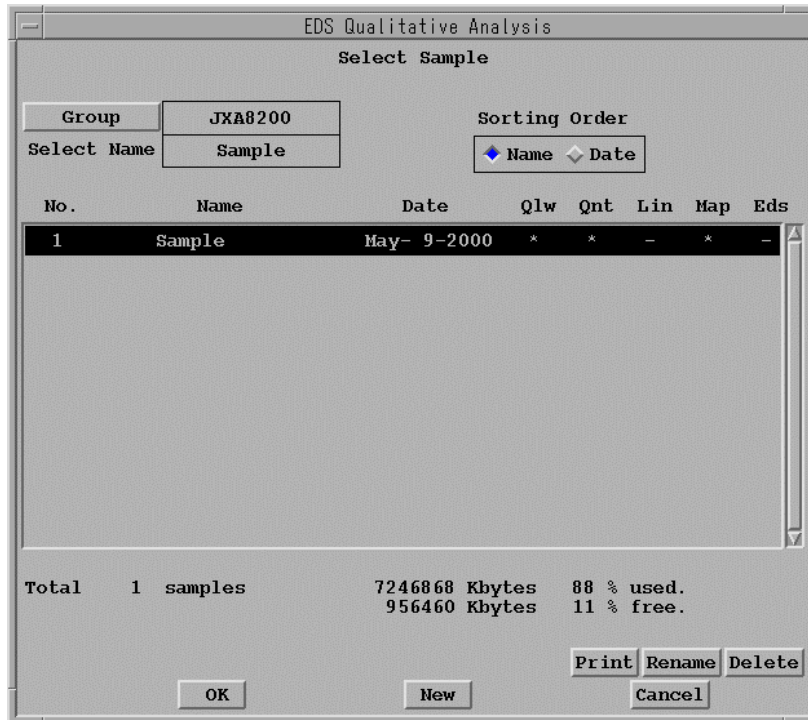


Fig. 4 Select Sample window

2. If you want to set up a new group or select an existing group, 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.

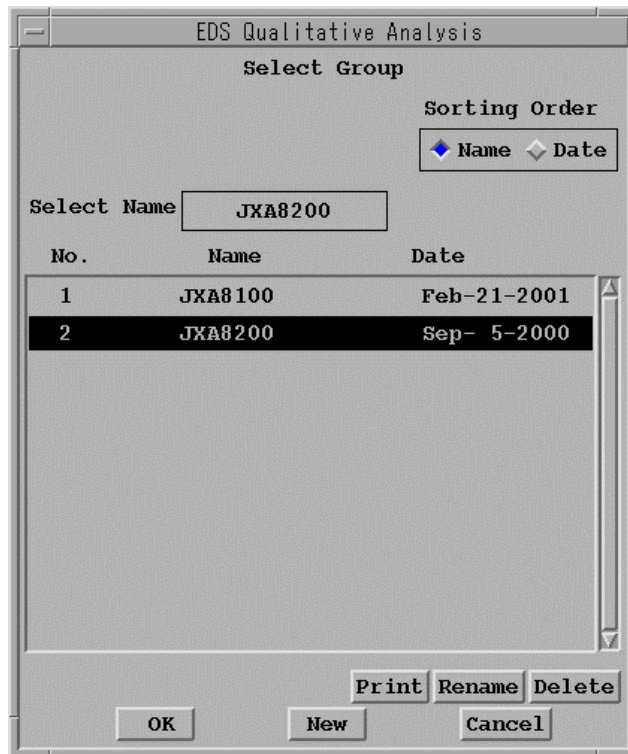


Fig. 5 Select Group window

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 a new Group or Sample name.
Print	Click on the Print button in either window to print the list of Group names or Sample names.
Delete	To delete the Group name or Sample name that has just been recorded, specify it in the appropriate window and click on the Delete button. Delete Group names and Sample names that have been already used for measurement by selecting Utility-File Utility from the EPMA Main Menu.
Sorting Order	Clicking on the Name button in Sorting Order in either window rearranges the Sample names or Group names in alphabetical order. Clicking on the Date button in Sorting Order rearranges them in chronological order.

Button	Function
OK	Click on the OK button in each window to finalize the Sample or Group name and close the window.
Cancel	Click on the Cancel button in each window if you want to cancel the Sample or Group name that was input and close the window.

4.1.4 Setting measurement conditions

- ◆ Click on the **Measurement** button in the EDS Qualitative Analysis function window.

The Measurement Conditions menu appears as shown in Fig. 6. Set the measurement conditions as described below.

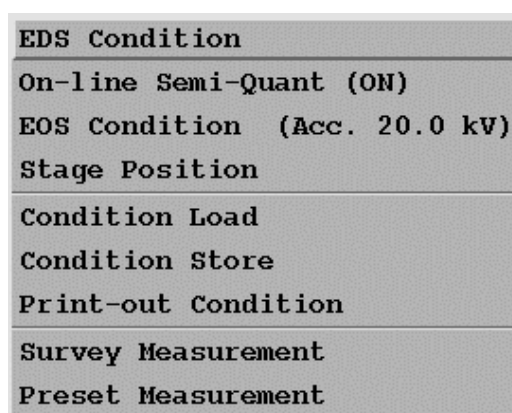


Fig. 6 Measurement Conditions menu

■ EDS Condition

- ◆ Select **Measurement-EDS Condition** from the EDS Qualitative Analysis function window.

The EDS Condition window opens as shown in Fig. 7, allowing you to set measurement conditions.

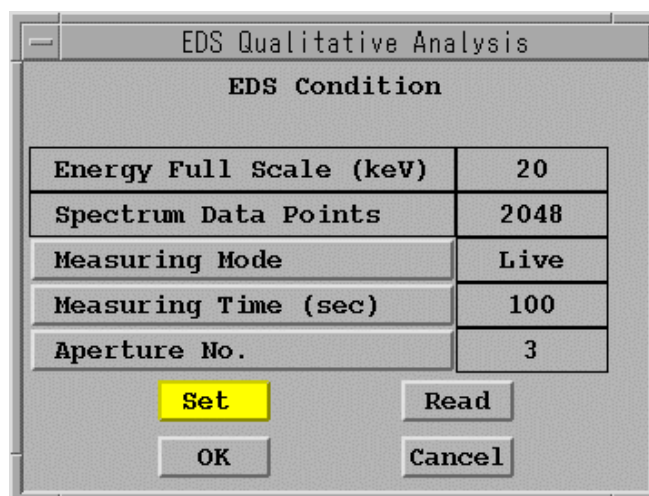


Fig. 7 EDS Condition window

Set conditions of the EDS detector for measurement. The following parameters can be set.

Parameter	Function
Energy Full Scale	The full scale energy is 20 keV.
Spectrum Data Points	The number of data is 2 kch.
Measuring Mode	Select either the effective time (Live Time) or realtime (Real Time) as the measuring mode.
Measuring Time	Set the measurement time in seconds.
Aperture No.	Select an aperture number from 0 to 5. Aperture No. 0 is open. The larger the aperture number, the smaller is the aperture diameter. Aperture No. 6 is closed, so it cannot be used for measurement.

1. To select an aperture number, first set the beam current, then try actually acquiring an EDS spectrum, and finally select the aperture number so that the displayed dead time is 20 to 30%.
2. Set the measurement time to several tens of seconds; then select Live Time.
3. To check the setting of the EDS detector, first display the EDS Home Window if it is not open, then click on the **Set** button and try setting the EDS conditions. Also, to read the parameter settings from the EDS, click on the **Read** button.

■ On-line Semi-Quant

After performing qualitative measurement, you can perform semi-quantitative analysis based on the identified elements.

1. Select **Measurement–On-line Semi-Quant** from the EDS Qualitative Analysis function window.

The On-line Semi-Quant window opens as shown in Fig. 8.

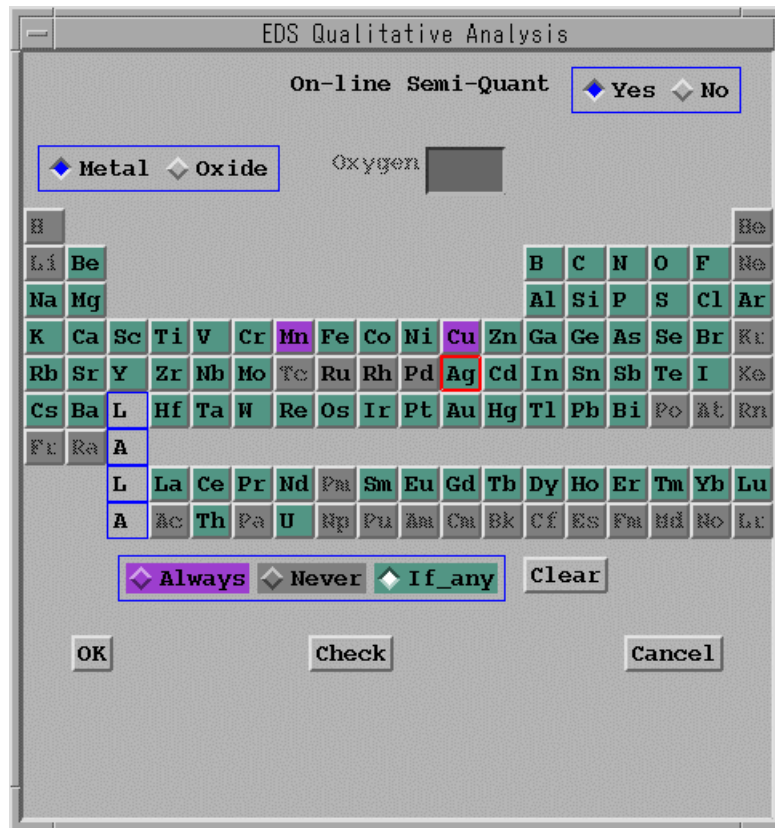




Fig. 8 On-line Semi-Quant window

2. To perform semi-quantitative analysis, click on the **Yes** button in the On-line Semi-Quant window. To perform qualitative analysis alone, click on the **No** button.

If you click on the **Yes** button, carry out the following procedure.

- a. Select **Metal** or **Oxide**.
If you select **Oxide**, type the number of oxygen for computing the chemical formula in the **Oxygen** input box.
- b. Specify whether or not to include each element in the results by selecting **Always**, **Never** or **If_any**.
 - **Always:** Each specified element will be included in the result list of elements for quantitative analysis, regardless of whether or not the element is identified in the sample.
 - **Never:** Even if an element is identified in the sample it is ignored in the results. If you want to set all the elements to **Never**, click on the **Clear** button.

- **If_any:** An element is included in the result list of elements only if it is identified in rank A.
-  If you want to set all the elements to **If_any**, click on the **Clear** button.
3. If you want to specify the line of X-rays to be used for each element, click on the **Check** button; then select the desired line from **K**, **L** and **M**.
The **Check** window opens as shown in Fig. 9.
By default, high-intensity X-rays are used in the computation. Also, you can set the valence for an oxide in this window.

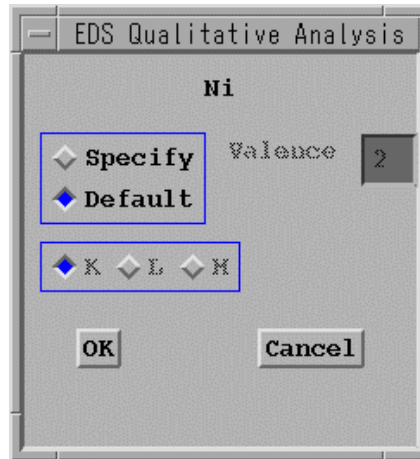


Fig. 9 Check window

Qualitative measurement takes place in the normal way, and elements are identified. Subsequently, if the **Yes** button is selected in the On-line Semi-Quant window, the results of element identification are checked against the specified elements, the results of semi-quantitative analysis are computed, and the results are 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 the present EOS conditions and displays them in the EOS Condition window in which you can input and alter items such as Probe Scan.

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

The EOS Condition window opens.

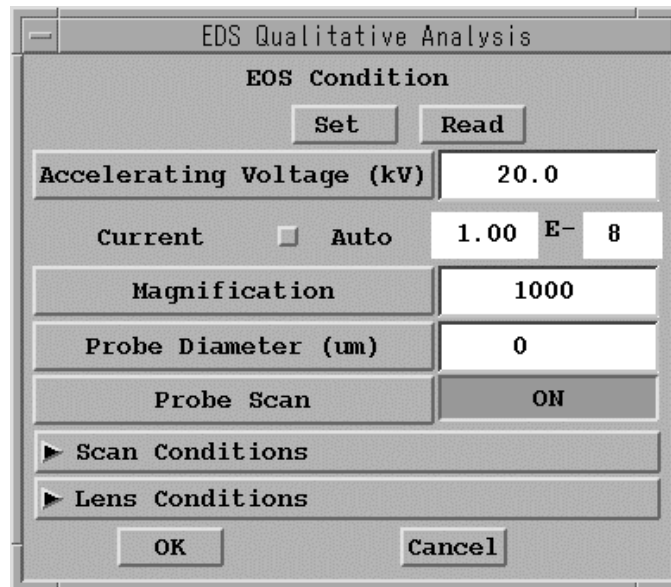


Fig. 10 EOS Condition window

Button	Function
Set	Sets the electron optical system (EOS) to the present conditions.
Read	Reads the present conditions of the EOS and displays them on the EOS Condition window.
Accelerating Voltage	Sets accelerating voltage for 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) for 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.

Button	Function
Scan Speed	Selects scan speed from S1 to S12. The larger the number, the slower the speed.
Focus	Specifies automatic or manual focus.
Stabilizer	Specifies whether the beam stabilizer is to be used (CL&Tilt, CL, or Tilt) or not (OFF).
Lens Conditions	Clicking on the arrowhead of this button opens the window for the following two items.
Condenser Lens	Specifies condenser lens settings (Coarse/Fine) for measurement.
Object Lens	Specifies objective lens 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.

4.1.5 Setting the analysis position

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 Position** from the EDS Qualitative Analysis function window.

The Stage Condition window opens.

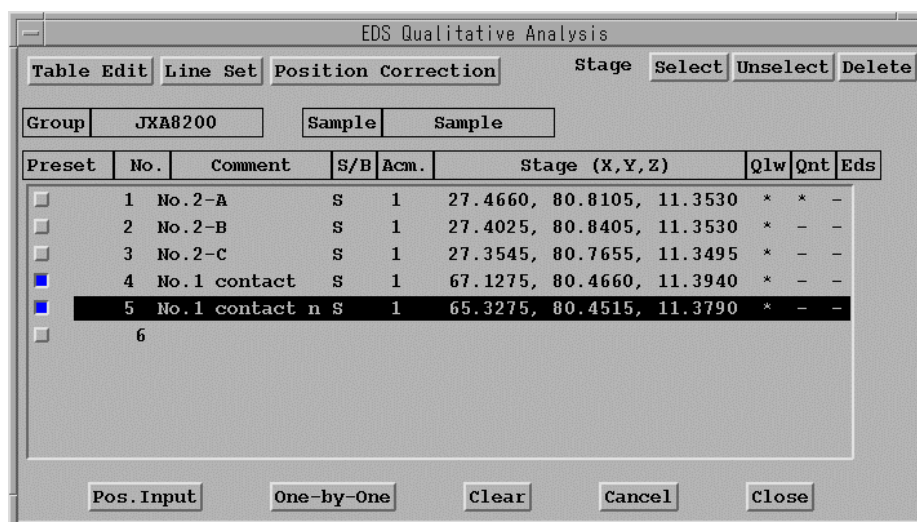


Fig. 11 Stage Condition window

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 EDS qualitative analysis, WDS qualitative analysis, and quantitative 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 accumulations, **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 have been 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	Cancel 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. You can turn all or some of the Preset buttons on at the same time using the Select button. You can turn all or some of the Preset buttons off at the same time using the Unselect button.
Delete	Allows you to delete 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

- ◆ Click on the **Pos. Input** button in the Stage Condition window.
The Stage Condition Input window with the Working Area opens.
This window enables you to set the analysis position of the stage or beam. Do this using the procedure described below.

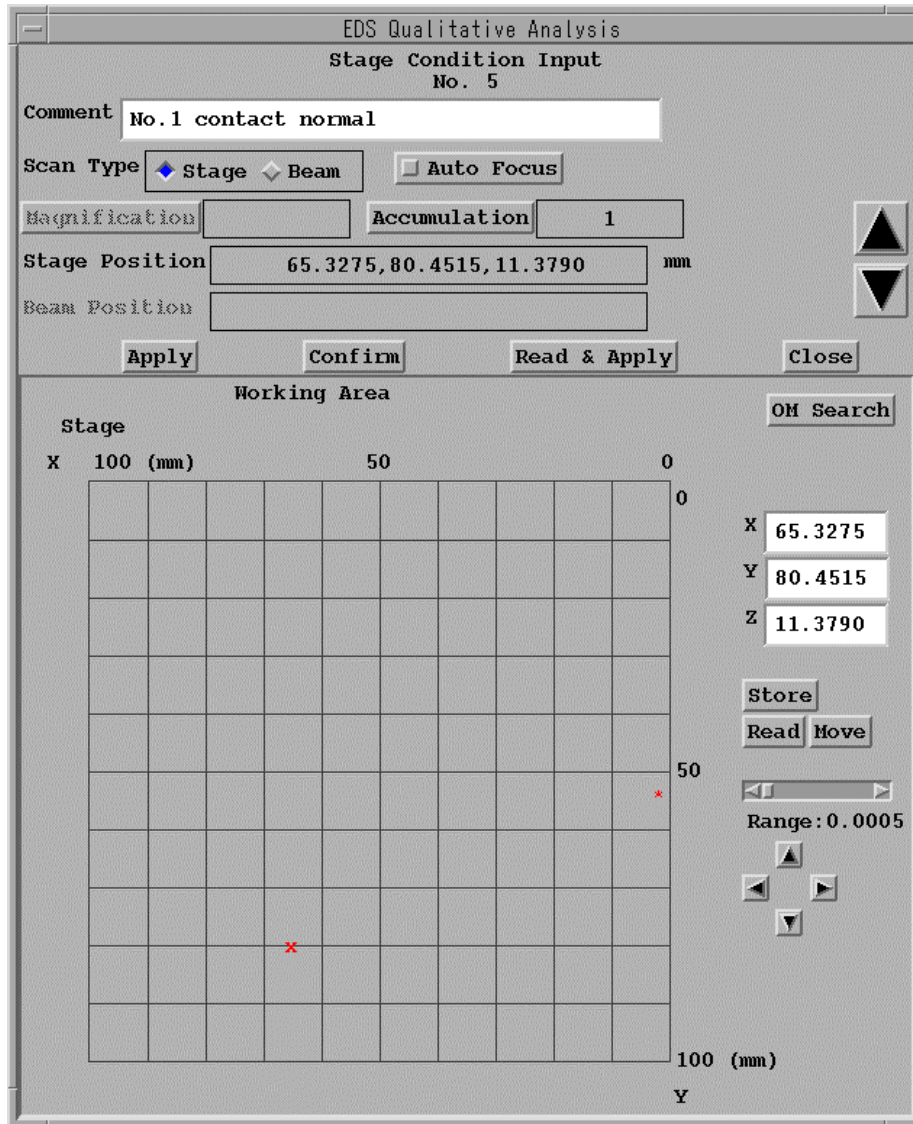



Fig. 12 Stage Condition Input window with Working Area

- 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, while observing the OM image, by using 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 can 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 in 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.

Object	Function
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.
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	Allows you to 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	Allows you to move 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.

4.1.6 Loading measurement conditions

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

1. Select **Measurement-Condition Load** from the EDS 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.

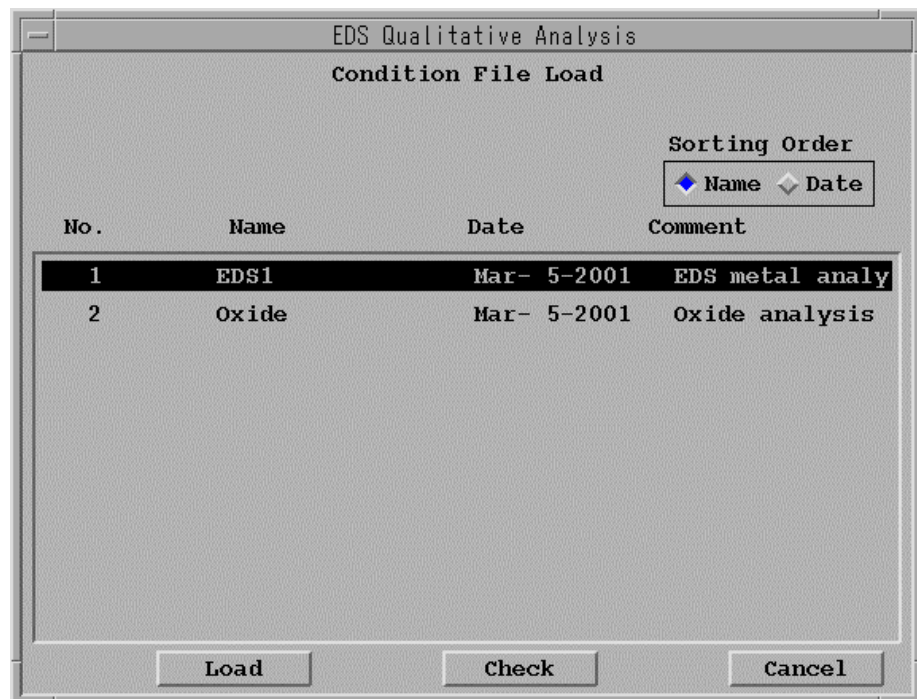


Fig. 13 Condition File Load window

2. To call up a set of 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 EDS 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.

4.1.7 Storing measurement conditions

After you set conditions in the EDS Condition, On-line Semi-Quant and EOS Condition windows, you can store them in a file and give a new name to the file.

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

The Condition File Store window opens.

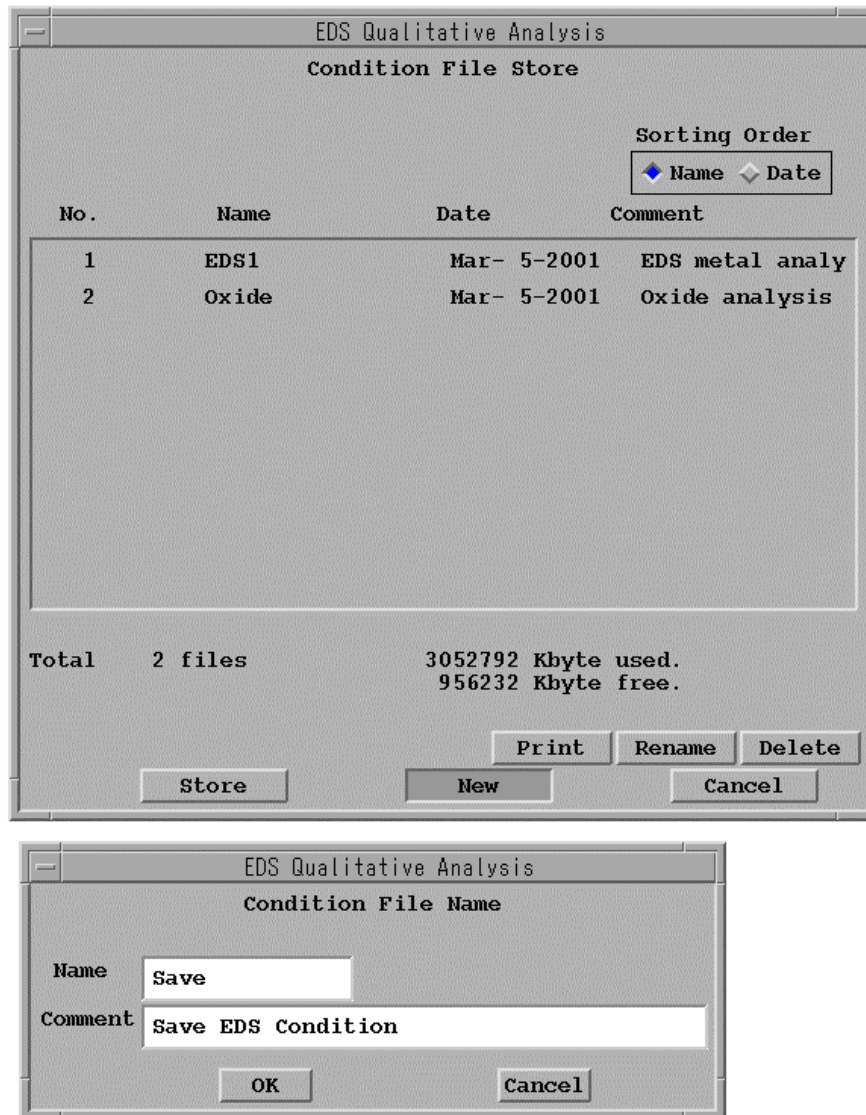


Fig. 14 Condition File Store window

- 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), and 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 printing (**Print**), changing the name (**Rename**) and deleting (**Delete**) using 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.

4.1.8 Printing measurement conditions and results

Selecting **Print-out Condition** from the Measurement menu opens the Print-out window. However, in EDS qualitative analysis, setting is not carried out using **Print-out Condition**. Therefore, set the conditions under which printing during measurement will occur using the EDS Home Window as follows.

- ◆ Select **Operation–Analysis–Result Format** from the EDS Home Window.
This opens a window in which you can set the conditions for printing.
To print the results, turn the **Printer** button in the Result Format window to **On**.
- ☞ For further information, refer to Sect. 4.5.2 “Specifying Result Output” in the instruction manual of the EDS Operation Program.

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 EOS and stage conditions. Use this mode to perform analysis without specifying detailed measurement conditions.

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 **EDS Qualitative Analysis** function window.
The **Preset Measurement** window opens.

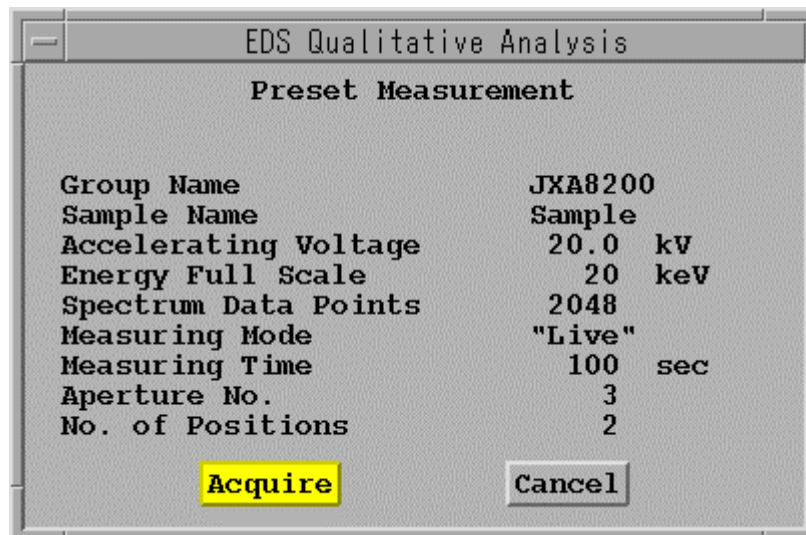


Fig. 15 Preset Measurement window

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 present stage position in the **Survey** mode.

1. Select **Measurement–Survey Measurement** from the **EDS 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.

4.2.3 Quali (E) Analysis Measurement window

During measurement, the Quali (E) Analysis Measurement window appears as shown in Fig. 16, 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.

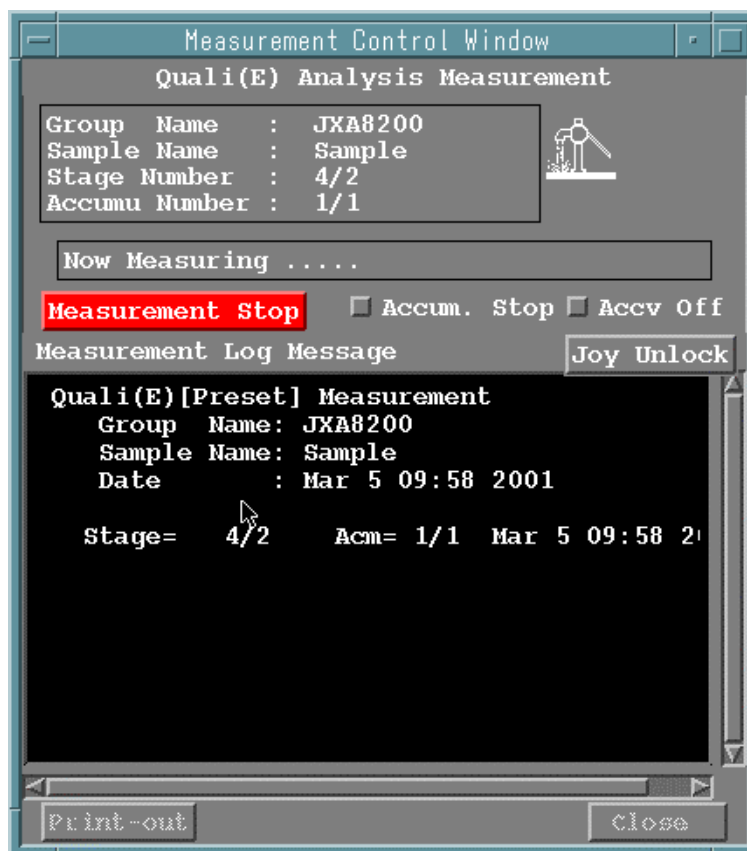


Fig. 16 Quali (E) Analysis Measurement window

4.2.4 Realtime display

During measurement, the spectrum presently being acquired is displayed in the EDS Home Window.