JXA-8100/JXA-8200

ELECTRON PROBE MICROANALYZER/ WD/ED COMBINED MICROANALYZER

OPERATION DIGEST

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.

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ELECTRON PROBE MICROANALYZER/ WD/ED COMBINED MICROANALYZER

OPERATION DIGEST

NOTICE

- This instrument generates, uses, and can radiate radio-frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to the environment, especially radio communications.
- This instrument must not be modified, and products other than those manufactured by JEOL Ltd. must not be attached to this instrument, without prior written permission. If any such modification or attachment is made, all the stipulated warranties and services contracted by JEOL Ltd. or its affiliated company will be void.
- Replacement parts for maintenance of the instrument performance are available for seven years from the date of installation. Thereafter, some of those parts may be available for a certain period of time, and in this case, an extra service charge may be applied for servicing with those parts. Please contact your JEOL service office for details.
- The information in this manual, which is based on specifications believed correct at the time of publication, is subject to change without notice due to improvements made in the instrument.
- In order to assist us in preparing future documentation, please advise your nearest JEOL service office if you find any errors in this manual. Kindly note that while the instrument can be used in combination with various attachments to serve a number of purposes, this special feature of the instrument is only briefly described in this manual, which chiefly provides information on basic operations.
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Note: For servicing or inquires, please contact your JEOL service office.

NOTATIONAL CONVENTIONS AND GLOSSARY

General notations

	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.	
Æ :	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 ac- cording 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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17 BRIEF DESCRIPTION OF JXA-8100/8200 OPERATION58

SAFETY PRECAUTIONS

For the proper use of the instrument, be sure to read the following safety precautions prior to starting operation or maintenance. They contain important information related to safety. Contact your JEOL service office whenever you are unclear about an operation or maintenance.

The safety definitions and their meanings used in our company's operation manuals are as follows:

A 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, may result in minor injury or material damage.
	Material damage includes, but is not limited to, damage to related
	devices and facilities, and acquired data.

Parts of this instrument where safety precautions are required are labeled with illustration signs as shown below.

Do not touch the parts labeled with these signs:



We request that you use the instrument in a proper manner and in the scope of the purposes and usage described in the brochures and operation manuals. Never make modifications such as removing protective parts, exchanging component parts and defeating safety measures.

The safety precautions for the optional attachments of this instrument are given in the individual instruction manuals as required.

M WARNINGS

- General warnings
 - Never expose shielded parts, remove component parts and modify them, or needlessly dismantle them. To do so would expose you to a thermal, electrical or radiation hazard. Never remove the grounding wire from a part that is liable to cause an electric shock or connect it to an unspecified location.
 - Never stand on the frame of the equipment, or the operation console table or chair when carrying out routine operations, maintenance or checks.
 - Switch off the power before carrying out maintenance or checks on a live part of the equipment or in the vicinity thereof, due to the risk of an electric shock.
 - If it is necessary to re-install the equipment in a different location, carry out a detailed survey of the location to which the equipment is to be moved, then consult JEOL's service center.

Warnings concerning nitrogen gas or P-10 gas contained in a high pressure gas cylinder

> • Dry nitrogen gas and P-10 gas for a flow proportional counter is contained in a high-pressure gas cylinder, and must be handled very carefully. Be sure to observe the rules and precautions pertaining to the handling of high-pressure gas.

Carry out the following checks when installing a high pressure gas cylinder.

- ① Is there an oven or a heating appliance in the vicinity of a gas cylinder?
- ② Is there electrical wiring in the vicinity of the gas cylinder?
- ③ Is the gas cylinder installed on a dedicated stand that is attached in such a way that it cannot topple over, and is it attached to a wall with a chain?
- ④ Does the temperature in the place where the gas cylinder is installed exceed 40 ?

Take great care to protect the gas cylinder from impacts when installing it. After installing the gas cylinder, be sure to install the cylinder valve open/close handle to enable the flow of gas to be cut off in an emergency.

When replacing a gas cylinder, check the expiration date indicated on the replacement cylinder, and use a cylinder that has passed the pressure withstand test.

• To avoid possible injury in the event that the pressure gauge or the pressure-reducing valve breaks and fragments fly about, do not stand in front of the pressure gauge when opening the cylinder valve open/close handle, operating the pressure control handwheel to adjust the pressure, or reading the pressure gauge. Be sure to stand obliquely in front of it.

- Do not abruptly open the cylinder valve or abruptly apply pressure to the pressure-reducing valve or the pressure gauge. Slowly turn the cylinder valve open/close handle about one half a turn; verify that the primary pressure rises; then open the cylinder valve fully.
- When handling even a small amount of dry nitrogen gas or P-10 gas in a room, leave the windows and doors open to ensure adequate ventilation.
 - Although nitrogen gas is inert and harmless, if a large amount of nitrogen gas leaks into a sealed room, an oxygen deficiency will occur, resulting in the risk of death by suffocation.
 - ② P-10 gas is argon gas that contains about 10% of methane (which is flammable), so handle it according to the ordinances and rules existing at the installation site. Also, do not use a gas other than P-10 as the detection gas for a gas flow counter.

Warnings concerning the magnetic field in the vicinity of the ion pump

 A person who wears a pacemaker or other medical appliance can be affected by the magnetic field generated by the ion pump magnet, so ensure that persons wearing such appliances keep well away from equipment on which an ion pump is installed.

▲ CAUTIONS

General Cautions

- If an abnormality occurs in the equipment, stop the equipment immediately, then contact your nearest JEOL service center.
- In the event of a sudden power failure, the equipment will stop operating. Also, if the supply of water is cut off while the equipment is operating, the entire equipment apart from the computer will stop automatically. Once the electric power and water supply are restored, restart the equipment after turning off the switches. If the procedure seems unclear, contact your nearest JEOL service center.
- The electron optical column is mounted on the column mount frame via a vibration-isolation device. When you operate a knob, the electron optical column sways a little, resulting in a clearance occurring between the vibration-isolation device and the frame. Be careful not to get your fingers caught in this clearance.
- Take care not to touch the drive motor of the wavelength dispersive X-ray spectrometer (WDS) while the WDS is being driven.
- To prevent an accident or a breakdown of the equipment, take care not to touch any protrusions on the OM television camera at the front of the basic unit or the EDS detector at the top left of the front panel of the basic unit. Always leave the covers of the main panel closed except when you operate the controls on these panels. Also, always remove the specimen exchange rod from the specimen chamber when not performing specimen exchange.
- To prevent an accident or a breakdown of the equipment, be careful not to trip over or step on a cable or hose when you walk behind the basic unit, operation and display panel, or similar location.
- Before replacing the specimen, the WDS slit, one of the various lamps, or other components carefully read the following and also the precautions accompanying the descriptions of the various operation methods in the instruction manual.
- This equipment does not produce noise or vibration that is harmful to the human body. However, if you detect an abnormality when using the equipment, contact your local JEOL service center.

Cautions concerning oil rotary pump

• Never pull out the rubber hose of the oil rotary pump while the pump is operating. If you do so, the oil in the oil diffusion pump will flow in the reverse direction and enter the electron optical column, resulting in a major breakdown.

• If you leave the oil rotary pump running with insufficient oil in it, the pump may be irreparably damaged. You can operate the pump normally until the oil level falls to the lower limit of the oil level indicator, but be sure that the oil level does not fall below this level.

Cautions concerning cleaning

• For cleaning the components of the equipment, use a cleaning liquid that has excellent cleaning power, contains few impurities, is incombustible and highly volatile, and does not harm the human body even when highly concentrated. When using even a small quantity of cleaning liquid indoors, provide adequate ventilation by leaving the windows and doors open.

When handling cleaning liquid, be sure to wear gloves that are resistant to the cleaning liquid used.

Cautions concerning electron gun and high-voltage cable

- Never remove the high-voltage cable from the electron gun. Never move the safety switch of the electron gun. It is possible that these operations could cause serious electrical shock.
- Never pull the high voltage cable. Pulling the cable will cause a loss of electrical contact or damage the cable.

Cautions concerning filament replacement

- When using a footstool while replacing the filament, use a stable footstool.
- Wait for about 1 minute after pressing the GUN VENT button before opening the electron gun chamber, because it takes about 30 seconds for the voltage of the electron gun to fall to 1/1000 after the accelerating voltage is switched off.
- If the filament is burnt out, the Wehnelt unit and cap will be hot immediately afterward, so do not touch these parts. If you do, you may receive a burn. Before replacing the filament, leave the Wehnelt unit at atmospheric pressure for at least 30 minutes to allow it to cool, and confirm that it has cooled adequately.

Cautions concerning replacement of the OM/OMT lamp

- Before replacing the lamp, wait for about 10 minutes. If the lamp goes open circuit, both the lamp itself and other parts in the vicinity will be considerably hot immediately afterward.
- To prevent burning yourself, wear thick gloves when adjusting the position of the lamp.

■ Caution concerning replacement of the WDS illumination lamp

• When removing the spectrometer cover, take care to prevent the cover from dropping. Place the removed spectrometer cover on a level surface in a place where there is no danger of it dropping. The spectrometer cover weighs about 7 kg.

Caution concerning replacement of the illumination lamp in the specimen chamber

• When withdrawing the specimen stage, pull it out gently and slowly. When pushing in the specimen stage, take care not to get your fingers caught between the specimen stage and the specimen chamber.

Caution concerning replacement of the specimen

- Install the specimen holder securely on the specimen-exchange rod to prevent it from dropping off the specimen-exchange rod.
- Caution concerning replacement of the WDS slit
 - Before replacing the slit, switch off the high voltage of the X-ray detector to ensure that you do not receive an electric shock.
- Caution concerning the liquid nitrogen trap (LNT) and the liquid nitrogen baffle (LNB)
 - When replenishing the liquid nitrogen in the LNT or LNB tank, or discharging it from one of these tanks, take care that liquid nitrogen does not get on your skin or splash about.
- Cautions concerning the EDS detector
 - If it is necessary to install or remove the EDS detector, contact JEOL's service center. Do not attempt to carry out this work yourself.
 - When using a footstool while replenishing liquid nitrogen in the EDS detector tank, use a stable footstool. When filling the tank with liquid nitrogen, take care that liquid nitrogen does not get on your skin.
 - Be very careful when handling an EDS detector that uses a beryllium film for the window because beryllium dust or vapor is toxic.

Caution concerning the xenon-filled proportional counter

• Be very careful when handling the xenon-filled proportional counter (XPC) or H-type xenon-filled proportional counter (XPCH) that uses a beryllium film for the window because beryllium dust or vapor is toxic.

Caution concerning TAP analyzing crystals

- The compound TAP (Thallium Acid Phthalate), used as the analyzing crystals, is toxic, so do not touch it for any reason.
- Caution concerning standard specimen holder
 - The Cd standard sample, which is part of the standard specimen holder, is toxic, so do not touch it directly when handling or polishing.
- Caution concerning the objective-lens cooling oil and high-voltage tank insulating oil
 - The objective-lens cooling oil and the high-voltage tank insulating oil are the same oil. This oil has the following properties, so obtain an understanding of these properties before using the oil.

Chemical name:	Petroleum-based hydrocarbon
Composition:	100% lubricating base oil
Flash point:	142 °C
Volatility:	None
Spontaneous ignition	
(spontaneous ignition, reaction with water):	None
Carcinogenicity:	None

When disposing of this oil, contact JEOL's service center and use a suitable disposal method.

Caution concerning Polaroid film

• Read the instructions provided with the Polaroid film for the precautions concerning the Polaroid film developing liquid, coating material and roller-cleaning liquid.

PRECAUTIONS CONCERNING INSTALLATION AND MAINTENANCE

Optional equipment to be procured locally

The equipment and parts to be procured locally must bear the CE mark indicating that they conform to the EU specifications.

Installing and adjusting the equipment

The installation and adjustment of the equipment are performed by JEOL's service engineers.

Installing and adjusting attachments

The installation and adjustment of attachments are performed by JEOL's service engineers.

- Attachments made by JEOL: A service engineer is dispatched from JEOL's service center to perform installation and adjustment.
- ② Equipment purchased from a manufacturer other than JEOL: ordinarily, a service engineer from JEOL's service center and also a service engineer from the manufacturer are dispatched to the customer's premises to carry out installation and adjustment.

Room in which the equipment is to be installed

- Install a suitable electrical distribution board, ventilation equipment, water supply and drainage facilities, and lighting equipment in the room where the equipment is to be installed.
- ② Observe the specifications concerning the allowable environment (temperature, humidity, vibration, stray magnetic fields, and other conditions) in the room in which the equipment is to be installed.

Treating waste materials and equipment

Before disposing of the following materials and equipment, contact JEOL's service center, and treat the materials and equipment using a suitable method.

- ① Basic unit and various auxiliary equipment
- ② High-voltage tank insulating oil and objective-lens cooling oil
- ③ Vacuum pump oil

Maintaining and inspecting the equipment

In order to maintain the performance of the equipment, it is recommended that you have JEOL's service engineer carry out maintenance and inspection.

- ① If you maintain and inspect the equipment, record and keep the results.
- ② Maintenance that can be done by the customer is limited to the following. When carrying out maintenance on the optional attachments, refer to the respective instruction manuals.

Before carrying out this maintenance, however, undertake a training course given by the service engineer, then carry out maintenance in accordance with the relevant items in the instruction manual. Have work other than that listed below done by JEOL's service center.

Replacement of the electron gun filament, electron optical system axis alignment, replacement of the objective lens aperture, replacement of the scintillator, lubrication of the oil rotary pump and inspection of the V belt, inspection of the vacuum rubber hose, replacement of the nitrogen gas cylinder, replacement of the specimen chamber illumination lamp, replacement of the window foil of the gas flow proportional counter, replacement of the P-10 gas cylinder, replacement of the spectrometer illumination lamp, replacement of the OM illumination lamp, maintenance of the computer section (the range indicated in the instruction manuals of the computer and peripheral equipment).

③ Tubes for cooling water or cooling oil last not more than five years in an ordinary installation room and not more than three years in a clean room. If the tubes are used longer than the lifetime specified above, a liquid might leak out from cracks in the tubes, thus causing adverse effects on the installation environment. Be sure to contact your local JEOL service center and have its service engineer replace the tubes within the specified lifetime.

1 PREFACE

This operation digest shows a few examples of the operations for the analysis of specimens with JXA-8100/8200. Even a single function of the JXA-8100/8200 can be operated in many ways, and the combination of operations allows many more ways of operation. In this instruction manual, only a few examples are shown. We hope that you will learn the ways to carry out analysis and the steps necessary for the operations by reading this manual. Then, you can determine the best procedure for your use and purpose, and you can make the most of the instrument.

2 COMPOSITION OF JXA-8100/8200

The JXA-8100 and 8200 are each composed of an electron optical column and its console, a control and display system, a computer system and a pump box placed behind the console. On the electron optical system (EOS) column, a specimen stage and wavelength dispersive X-ray spectrometers (WDS) are installed. In the JXA-8200, an energy dispersive X-ray spectrometer (EDS) is also installed here.

The computer system is an engineering workstation (EWS), so that most operations other than specimen exchange and control-panel manipulation can be performed through windows on the displays.



JXA-8100



Control and display system



Computer system

3 START-UP AND SHUTDOWN

3.1 Start-up

- **1.** Open the valve of the nitrogen gas and check that the pressure on the secondary side is kept at 0.4 to 0.5 MPa.
- 2. Turn the cooling water on.
- 3. Turn on the main power switch on the distribution board.
- **4.** Turn the **POWER** key switch on the main panel to **START** and release the key.
- **5.** Turn the valve of the P-10 gas on and check that the gas flow rate is in the range of 3 to10 cc/min.
- **6.** Confirm that the basic screen (showing the film title or number, accelerating voltage, magnification, micron bar and so on at the bottom of the screen) is displayed on the viewing display.
- **7.** Turn on the power for the computer system (computer, display, printer, MO drive and other peripherals).

The Login window is displayed on the computer display.

- 8. Enter a user name, usually jx1, and then click on the OK button.
- **9.** Enter the password if any. In the case of user jx1, enter jx1jx1 as the password, and then click on the **OK** button.

The start-up program proceeds automatically, displaying the EPMA Main Menu on the computer display. The computer system is now ready to be operated.



EPMA Main Menu

10. If necessary, initialize the positions of the spectrometers and the specimen stage.

Usually this step is needed only if trouble has occurred in the spectrometers or the stage.

- a. Click on the Initialize icon in the EPMA Main Menu.
- b. Click on Quick Initialize in the Initialize menu.
- **c.** Click on the **Position** button for Spectrometer or Stage in the Reset Position window.
- d. Click on the OK button.
- **11.** Confirm that the **HV READY** lamp on the main panel lights up approximately 20 minutes after the power was turned on in the above step 4.

3.2 Shutdown

- 1. Push the ACCEL VOLTAGE ON button on the main panel to shut off the accelerating voltage.
- **2.** Close all the windows for the analysis and data processing other than the EPMA Main Menu on the computer display.
- **3.** Click on the **Initialize** icon in the EPMA Main Menu.
- 4. Click on SYSTEM SHUT DOWN in the Initialize menu.
- 5. Click on the OK button.
 - The power for the computer system turns off automatically.
- **6.** Turn off the computer display, printer, MO drive and other peripherals.
- **7.** Turn off the **POWER** key switch on the main panel, and turn off the main power switch on the distribution board.
- 8. Close the valve of the P-10 gas.
- 9. Close the valve of the nitrogen gas.
- **10.** Turn off the cooling water after 3 to 5 minutes.

4 COMPOSITION OF SOFTWARE (EPMA MAIN MENU)

The rough classification of the software is shown in the EPMA Main Menu. The outline is as follows:

Process:	Displays and analyzes data now being taken (in real time) or taken previously.
Analysis:	Acquires data with the instrument.
Monitor:	Controls the EOS conditions, moves the spectrometers (WDS) and
	the specimen stage and operates other individual functions.
EDS:	Acquires data with EDS detector and processes them (JXA-8200 only).
Initialize:	Used for setting the system configuration, initializating the instrument and other settings.
Utility:	Functions to support analysis operations.
JEOL:	Used mainly for maintenance.



EPMA Main Menu

5 BASIC FUNCTION AND OPERATION OF WINDOWS

Common functions and operations in the windows on the computer display are described below.

- Before some window opens, a frame of the window is displayed. The frame can be moved to the desired position on the display.
- The frame of the window can be controlled as described below.



Description of window frame

- ① Title bar
 - You can move the window to the desired position by dragging the title bar.
 - You can move a window hidden behind another window to the front by clicking on its title bar.
- Minimize button

By clicking on this button, you can minimize the window, that is, displays it as an icon at the bottom right of the display. To restore the original window from the icon, double-click on the icon. If it is not necessary to keep a window open, it is better to close it than to minimize it.

Maximize button

By clicking on this button, you can enlarge the window to fill the screen. To return the window to its original size, click on the maximize button again.

④ Resize border

When the pointer is moved to the border at the top, bottom, left or right or to one of the four corners of the window, its shape changes. You can change the size of the window by dragging the resize border.

- ⑤ Window menu button
 - Moving, enlarging or reducing the window can be controlled using this window menu, too.
 - To close the window, double-click on this button.

• The operations in windows are as follows:



Description of tools in window

1 Buttons

To select or execute an item, click on its button.

② Arrow buttons

To drive the spectrometer or the specimen stage, click on the arrow buttons that point in the direction in which you want to move it.

③ Scroll bar

You can change the numerical value for an item continuously by dragging the scroll bar, or stepwise by clicking on the two arrows.

④ Input box

You can enter numerical values or text directly using the keyboard after positioning the pointer here.

(5) Two-dimensional display

You can control two coordinates indicated on the graph in the window by dragging the block cursor \Box or by double-clicking on the desired position.

Closing windows

- To close a window, usually click on the Close, Exit or Quit button. Although a window can also be closed using the window menu button, this method is not recommended in windows where a Close, Exit or Quit button is displayed.
- When subsidiary windows have been opened by clicking on the button in the main window, clicking on the Close button in the main window closes all these windows.

6 BASIC OPERATIONS OF EOS AND DISPLAY SYSTEMS

The methods for operating the EOS and display systems are classified roughly into three sorts as described below.

- Operations using switches and knobs on the control panel.
- Operations on the viewing display.
- Operations of the EOS Monitor window on the computer display.

Regardless of which method is used, the basic operations of almost all specific functions are closely related or linked to each other. Therefore, any of them may be selectively used according to the current situation.

The main operations are described below.

6.1 Operations Using Switches and Knobs on the Control Panel

The buttons that switch a function to two or more modes are given below. They change the mode cyclically in the order shown each time they are pushed.

IMAGE SELECT	
VIEW	$\begin{array}{c} \text{SEI} \rightarrow \text{COMPO} \rightarrow \text{TOPO} \\ & & \\ & \\ & \\ & \\ & \\ \end{array}$
SCANNING MODE	
QUICK VIEW	$Q1 \rightarrow Q2$
FINE VIEW	$ \begin{array}{c} F1 \rightarrow F2 \\ \bullet \end{array} \end{array} $
ALIGNMENT	
ALIGN	Gun Alignment Tilt \rightarrow Gun Alignment Shift \rightarrow OL Aperture

AUTO

Execute the automatic function specified in SETUP-OPERATION.

РНОТО

Photograph or transfer the image that is on the viewing display.

The status set with these buttons is displayed on the basic screen on the viewing display, or on the EOS Monitor window on the computer display.

6.2 Operations on the Viewing Display

The mouse pointer can be moved seamlessly between the computer display and the viewing display using the Swing Mouse function. To make a change in an input box on the viewing display take effect, press the Enter key on the keyboard.

By double-clicking or dragging and dropping on the display, the viewing area can be shifted by a shift of the electron beam or movement of the specimen stage.

6.2.1 Basic screen

① Electron gun control ③ Menu buttons ④ Icons Column Image Setup Maint <u>Tat</u>≣ нт 20.0 kV емі 0 µA -Menu bar ON OFF 20.0 kV - + COL NOR LDF W.D 8 9 11 13 15 mm с 45 🗘 Ð Control 0 -1 BEI-C BEI-B 6 6 - + MDF \$__ F د> 305 🖓 دک window ECP -+ EMP 134 0 µm - + Close Probe DIA Image display JEOL SEI 20.0 kV x 1,000 10 µm WD 10mm

② Observation condition display

Basic screen

1 Electron gun control

UT .	The accelerating voltage on/off hutton
EII -	The accelerating voltage on/on button
	This button indicates the state of the accelerating
	voltage, and allows you to switch on and off the
	accelerating voltage.
Black background:	The accelerating voltage is off, but cannot be switched
	on.
Blue background:	The accelerating voltage is off, and can be switched on.
Green background:	The accelerating voltage is on.
20.0 kV :	Display of the accelerating voltage
EMI :	Switching between emission current and filament current
0 μΑ:	Display of the current

Observation condition display

The present observation conditions such as image signal, accelerating voltage and magnification are shown, and they can be changed by selecting the desired item from the pop-up menu that appears when you click on each button.

Menu buttons

B Refer to Section 6.2.2.

④ Icons

SHIFT	
S+S	
STAGE	

Clicking on this button returns IMAGE SHIFT to (0,0). Clicking on this button turns the specimen-stage movement mode on (green) or off (gray).



Clicking on this button turns the SRT (scan rotation) on (green) or off (gray).



Clicking on this button opens or closes the Control window.

Clicking on this button moves the PCD (probe current detector) in or out of the column.



Buttons for selection of display mode

Buttons to activate the text mode, display a gray scale, display the clipboard.

6.2.2 Menus



Menu tree

1 Column Menu

Displays the Control window (Section 6.2.1). Adjusts the contrast and brightness. Aligns axis. Controls the optional rotation and tilt specimen holder. Displays the MDA.

2 Image Menu

Image processing and image-data filing.

③ Setup Menu

Setting the conditions related to the electron optical system using the Operation window. Setting the image-transfer conditions for the Ethernet connection.



Operation window

(4) Maint. Menu

Displays the vacuum system condition. Displays the JEOL service menu.

6.3 The Operation of the EOS Monitor Window on the Computer Display

The EOS and display systems can also be operated through the EOS Monitor window that opens on the computer display when you select **EOS Monitor** from the **Monitor** menu.

Clicking on buttons in this window opens windows, in many cases, and allows the EOS to be set or controlled.

For example, clicking on the **Signal**, **Mode** or **Speed** button opens the Scan Control window, in which you can select and reset the items in Image signal, Scan mode, Scan speed and other sections.

For basic operations of the EOS and display systems, the EOS Monitor window is mainly used in addition to the control panel.

Electron Op	otical System 🔄 🗌	J Scan Control - 🗆
File EOS mo	nitor Utility Exit	Reset Scan Control
Filament 133 Hv ON Acc.V 20.0 CL (Current) C 15 F 302 Signal SEI Mode SPOT Speed S1 PCD IN Scan OFF Mag 1000 P.Dia 0 OL 0 mm (609304)	High Volt. Ready Filament W Alignment Contrast/Bright. 3905 1808 Collector -3 - Shift 0 0 Spot 1144 1424 Rotation Optim Apt. 1 AFC ASTG Inst Pic BST 1.20E-06 (A)	Scan OFF Detector OFF Image signal Scan OFF Detector OFF Image signal SEI COMP TOPO EDS AUX1 AUX2 AUX3 ADD XR1 XR2 XR3 XR4 XR5 Scan mode PIC B_UP LSP SPOT AREA Reduce mode FULL 1/4 Scan speed Speed 1 Speed 2 Speed 3 Speed 4 Speed 5 Speed 6 Speed 7 Speed 8 Speed 9 Speed 10 Speed 11 Speed 12 EOS mode NOR LDF MDF ECP EMP Close

EOS monitor window

Scan Control window

7 MONITOR MENU AND MAIN OPERATIONS

The Monitor menu is composed mainly of the items for controlling the EOS, the spectrometers (WDS) and the specimen stage and for displaying their settings. The main items, other than the EOS Monitor window (Section 6.3), are explained below.

Present Positions
EOS Monitor
Ratemeter
Chart Recorder
Peak Search
Stage
SCA Scan
OM Auto Focus
CAF Switch
ОМ
Video Tools >

Monitor menu

7.1 OM Monitor Window

Clicking on **OM** in the Monitor menu opens the OM Monitor and Video Display windows. These windows let you adjust the specimen surface position, select analysis positions, and perform related tasks.



OM Monitor window and Video Display window

Here are some points to note:

- To turn the OM lamp on or off, click on the lamp icon 🔄 in the OM Control area of the OM Monitor window.
- If the optional Transmission Illuminator is installed in your instrument, you can rotate the polarizer or select a lamp through the OM Monitor window.
- By clicking, dragging, or dragging and dropping on the Video Display window, you can drive the specimen stage.

7.2 Stage Monitor Window

Clicking on **Stage** in the Monitor menu opens the Stage Monitor window where the specimen stage is moved and the analysis position on the specimen is set.



Stage Monitor window

The main contents are described below.

- Clicking on the **Holder** button displays the desired specimen holder and grid (scale).
- The present specimen position is shown by the red mark on the two-dimensional display and also by numerical values of its coordinates in the X, Y and Z input boxes. The specimen stage moves when you double-click on the desired position or drag and drop the red mark. It moves also when you enter the coordinates in the X, Y and Z input boxes or click the X, Y and Z arrow buttons.
- Normal Step or Micro Step is selected by clicking on the Normal Step button.

• Clicking on the **Memory** button stores the specimen position. To display the stored specimen position, click on the specimen name buttons under the two-dimensional display.

The specimen stage is usually operated both through the Stage Monitor window and with the joystick controller.

7.3 Ratemeter Window

Clicking on **Ratemeter** in the Monitor menu opens the Ratemeter window. This window displays the present X-ray count rate of the WDS and the probe current, and allows them to be changed.

ratemeter 🛛 🖓						
HV ON	Measure	Rate	meter	Ut	ility _	Exit
Channel	CH-1	CH-2	CH-3	CH-4	CH-5	Ourr.
Crystal	PETH	LDE1H	TAP	PETH	TAPH	Probe
Scale	100	100	100	100	1000	E-07
TI						
I						
I						
Cips	• 0	25	50	75	100	1.20
Max	0	25	50	75	100	1.20
Pos.	107.013	147.467	90.656	107.567	90.656	
Count	123	123	123	123	123	
	Ca Ka	n ka	Al Ka	Ca Ka	Al Ka	

Ratemeter window

Clicking on the **Measure** button opens the Measurement window for X-ray counting to be used as a scaler. Set a measurement time in the Meas. time input box.

7.3.1 SPC window

The SPC window is opened by clicking on any of the CH-1 to CH-5 buttons in the Ratemeter window. In this window, an analyzing crystal can be selected and the spectrometer can be driven to the detection positions of the elements to be analyzed in the specified channel. The detection position can be set in the L (mm) input box, with the scroll bar or in the Periodic Table window.

Clicking on the **SCA** (pulse-height analyzer) button opens the SCA Control window, that is, the window for setting the high voltage to be applied to the X-ray detector, the amplifier gain, and the base level of the pulse-height analyzer (SCA) for the specified channel.



SPC window

Periodic Table window

7.3.2 Periodic Table window

Clicking on the **Element** button in the SPC window displays the Periodic Table window. The spectrometer is set to the detection position of the element to be analyzed when you click on the element symbol and the **Set** button.

8 SPECIMEN EXCHANGE

Six types of specimen stages are available. They are the high-speed large-specimen stage (HSLSS), the high-precision specimen stage (HPSS), the high-speed large-specimen stage Z (HSLSZ), the high-precision specimen stage Z (HPSZ), the large-specimen stage (LSS) and the goniometer stage (GS).

8.1 Mounting the Specimen Holder

1. Click on the **Sample Change** button in the Stage Monitor window and then click on the **OK** button. The specimen stage moves to the specimen-exchange position as shown below.

	HSLSS, HPSS, HSLSZ, HPSZ	LSS	GS
Х	45 mm	0.5 mm	16 mm
Y	1 mm	40 mm	25 mm
Z	11 mm	11 mm	11 mm
Rotation	_	_	$000~(0^{\circ})$
Tilt	_	_	$000 (0^{\circ})$

Table 1Specimen-exchange position for each stage

Set the rotation and tilt controls for the GS manually.

- 2. Mount the specimen holder in the specimen-exchange chamber according to the type of the stage.
- a. HSLSS, HPSS, HSLSZ, HPSZ

Open the specimen-exchange chamber lid, set the specimen holder (except when using the large specimen holder for the HSLSS) on the adapter, fit the adapter (or the specimen holder) slot into the end of the T-shaped exchange rod and mount the adapter (or the specimen holder) on the specimen-exchange platform.

b. LSS, GS

Screw the specimen-exchange rod combined with the specimen-exchange chamber lid into the specimen holder, and install it in the specimen-exchange chamber.

- **3.** Push the vacuum-control button while pushing the specimen-exchange chamber lid to evacuate the specimen-exchange chamber.
- **4.** After checking that the specimen-exchange chamber is evacuated, open the specimen-exchange chamber isolation valve.
 - a. HSLSS, HPSS, HSLSZ, HPSZ

Turn the isolation-valve lever clockwise and pull it out to the right completely to lock the isolation valve.

Then turn the lever counterclockwise to lock the valve.

b. LSS

Turn the isolation-valve knob fully counterclockwise and pull it out to the right completely to open the isolation valve.

c. GS

Pull out the isolation valve knob fully to the right to open the isolation valve.

5. Mount the specimen holder on the specimen stage.

a. HSLSS, HPSS, HSLSZ, HPSZ

Push in the specimen-exchange rod completely. The specimen holder is mounted on the specimen stage. Then turn the exchange rod counterclockwise and pull it out.

b. LSS, GS

Push in the specimen-exchange rod completely to mount the specimen holder on the specimen stage. Then unscrew the exchange rod from the specimen holder and pull it out.

- 6. Close the specimen-exchange chamber isolation valve (perform Step 4 in the reverse order).
- 7. Push the vacuum-control button to vent the specimen-exchange chamber when using any specimen stage except the GS, which is vented automatically in Step 6.
- **8.** When using the LSS or the GS, remove the specimen-exchange chamber lid and the specimen-exchange rod assembly.

8.2 Dismounting the Specimen Holder

- **1.** Perform Steps 1 and 3 through 8 of Section 8.1, "Mounting the Specimen Holder". When you do, carry out Step 5 in the reverse order.
- 2. Dismount the specimen holder from the specimen-exchange chamber or the specimen-exchange rod.

9 GENERATION OF ELECTRON PROBE AND ALIGNMENT OF ELECTRON COLUMN

9.1 Generation of the Electron Probe

This section describes the procedures for operating the electron gun with the tungsten filament.

When using the optional LaB_6 filament, refer to the instruction manual of the LaB_6 electron gun.

- Mount the specimen holder on the specimen stage. Then, move the specimen to a point that you want to analyze and focus the OM image through the Video Display window, using the Stage Monitor window and the joystick controller.
- 2. Press the ACCEL VOLTAGE ON button on the main panel.
- **3.** Click on the Monitor icon and select **EOS Monitor** from the Monitor menu. Next, click on the **Hv** button to turn the accelerating voltage **ON**. The filament heating power is also turned on simultaneously.
- Click on the Acc. V button, and select the accelerating voltage that you want to use from the pull-down menu that appears.
 ✓ Usually, 15 or 20 kV is used.
- **5.** Set the probe current to about 10^{-9} to 10^{-8} A using the **CL (Current)** button.
 - You can also set the probe current using the PROBE CURRENT knob on the OPERATION panel.
- 6. Click on the Filament button in the EOS Monitor window to display the Filament window.

If longer life of the filament is preferred, click on the **Bias** button to display the Bias window. Select $-2 (10 \ \mu\text{A}) \text{ or } -1 (20 \ \mu\text{A})$ and click on the **Start** button.

- 7. Select Auto Saturation and click on the Start button.
 - For daily operation, select the Saturation curve **& Probe Current** and the Search Mode **From Preset Value** in the Optional Condition window.
 - After filament exchange, select the Saturation curve & EMP Pattern and the Search Mode Normal or Show wide range.
- 8. Click on the EMP button in the Filament window and confirm that the center of the EMP (emission pattern) is bright. Next, click on the EMP button to cancel EMP mode.



Relation between probe current and filament setting

The position \mathbf{e} on the graph is the saturation point of filament heating. If the filament heating current is set higher than the saturation point, the filament life may be shortened.

The saturation point of an old filament at the end of its life is 30 - 40 lower than that of a new one. Therefore it is recommended to observe the change of the saturation point routinely.

9.2 Automatic Alignment of the Electron-Gun Axis

Carry out this alignment after the procedure of Section 9.1. This function works properly when the objective lens aperture is set to the right position.

- 1. Click on the **Alignment** button in the EOS Monitor window to display the Alignment window.
- 2. Click on the Auto Alignment button to display the Auto Alignment window.
- **3.** Select the alignment mode and the search range.
 - For daily operation, select Narrow.
 - After filament exchange, select Middle or Wide.
- **4.** If an error message appears at the end of the automatic alignment, adjust the axis according to the procedure in Sections 8.3 8.5.
9.3 Manual Tilt Alignment of the Electron Gun

Carry out the following procedure after having exchanged the electron gun filament, if you find it necessary.

- 1. Click on the EMP button in the Filament window to display the EMP (emission pattern) mode.
- 2. Press the ALIGN button on the Operation Panel to display the Alignment menu on the viewing display, and select Gun Alignment Tilt.
- **3.** Click on the **Alignment** button in the EOS Monitor window to display the Alignment window.
- 4. Select the scanning speed S1, and move the EMP to the center of the viewing display using the ALIGNMENT-X, -Y knobs on the Operation Panel. Select the speed S3 and move the center of the EMP to the cross point of the cross marker.
- 5. Click on the EMP button to cancel the EMP mode.
- 6. Confirm that CL Coarse is 40 50 using the EOS Monitor window.
- Set the PCD button to IN using the EOS Monitor window, and maximize the probe current by adjusting the ALIGNMENT-X, -Y knobs on the Operation Panel.
- 8. Press the STIG button on the Operation Panel to close the Alignment menu.

9.4 Tilt Alignment of the Electron Gun

Center the aperture after the objective lens aperture is changed or when the accelerating voltage is changed greatly.

- Set the PCD button to OUT using the EOS Monitor window, and center a feature and display its image (SEI) on the viewing display. Adjust the contrast and brightness of the image using the CONTRAST and BRIGHTNESS knobs on the Operation Panel, and then focus the image using the FOCUS knob.
- 2. Select an aperture number adequate for your purpose from the OL aperture selector.
- **3.** Confirm that CL Coarse is 40 50 using the EOS Monitor window.
- Press the RDC IMAGE button on the OPERATION panel, and select the scanning speed S1 in the EOS Monitor window. This operation allows quick response for the image movement.
- 5. Press the **WOBB** button on the OPERATION panel and the focus of the image changes periodically. If the feature image shifts, mechanically adjust the X and Y aperture centering knobs of the OL aperture selector to minimize the shift of the image.
- 6. Press the WOBB and RDC IMAGE buttons to turn them off.

9.5 Manual Shift Alignment of the Electron Gun

Carry out the following procedure after having exchanged the electron gun filament, if you find it necessary.

- **1.** Center a feature and display its image (SEI) on the viewing display at an image magnification of $5,000 \times -10,000 \times$.
- 2. Press the ALIGN button on the Operation Panel to display the Alignment menu on the viewing display, and select Gun Alignment Shift.
- **3.** Set PROBE CURRENT–COARSE/FINE to COARSE.
- Adjust the probe current shown in the EOS Monitor window to the order of 10⁻⁹ A using the PROBE CURRENT knob and memorize the position of the feature image.
- Adjust the probe current to the order of 10⁻⁷ A using the PROBE CURRENT knob and observe the position of the feature image. If the image has moved, return the feature image to its former position using the ALIGNMENT –X, –Y knobs.
- Repeat Steps 4 and 5 to minimize the movement. Try to make it less than about 1 μm.
- 7. Press the STIG button to close the Alignment menu.
- To obtain a larger probe current (for example, the maximum probe current), set CL COARSE and CL FINE to their maximum values after the above alignment procedure. Adjust the **Gun Alignment Tilt** and **Shift** alternately to obtain maximum probe current using the ALIGNMENT -X, -Y knobs.

10 IMAGE OBSERVATION

10.1 Observing SEI (Secondary Electron Image)

- 1. Generate the electron probe and align the electron column (see Chapter 9). Select the accelerating voltage according to the type of specimen and the purpose of observation.
- 2. Set the specimen on the specimen stage, observe the OM image and focus it.
- **3.** Set the buttons on the Operation Panel as described below (or confirm that they are set as described below).

IMAGE SELECT-VIEW: SCANNING MODE-PRB SCAN: -QUICK VIEW: SEI (confirm on the viewing display) on (lit) Q1 or Q2

- **4.** Push the **PCD** button to **OUT**. The SEI signal image is displayed.
- If the image is white or black, push the DISPLAY & PHOTO-ACB button on the OPERATION panel, or adjust the scroll bars of the window opened by selecting Column-Cont./Bright. to make the image grayish.
- 6. Focus the image using the FOCUS knob on the Operation Panel.
- **7.** If necessary, change the magnification of the image, focus it and adjust its contrast and brightness using the knobs on the Operation Panel.
- 8. To observe a high-magnification image,
- **a.** Set the probe current to 10⁻¹¹ to 10⁻¹⁰ A using the **PROBE CURRENT** knob on the Operation Panel.
- **b.** Select the desired OL aperture number from **Optim Apt** on the EOS Monitor window.
- C. Push the WOBB button and adjust the X and Y aperture centering knobs of the OL aperture selector to minimize the shift of the image. Then push the WOBB button again to turn it off.
- **d.** Focus the image using the **FOCUS** knob, and adjust the astigmatism using the **ALIGNMENT-X**, **-Y** knobs.

10.2 Observing BEI (Backscattered Electron Image)

In BEI, a COMPO (composition image) or a TOPO (topographic image) can be selected.

- **1.** Observe the SEI of the specimen, focus it using the **FOCUS** knob and adjust its astigmatism using the **ALIGNMENT-X**, **-Y** knobs on the Operation Panel.
- 2. Set IMAGE SELECT-VIEW to COMPO or TOPO by pressing the VIEW button.
- If the image is white or black, push the DISPLAY & PHOTO-ACB button, or adjust the scroll bars of the window opened by selecting Column-Cont./Bright. to make the image grayish.
- **4.** If necessary, change the magnification of the image, focus it and adjust its contrast and brightness using the knobs on the Operation Panel.

10.3 LDF (Long Depth of Focus) Mode and MDF (Maximum Depth of Focus) Mode

The LDF or MDF mode is useful for observing rugged or highly tilted specimens. These modes are especially effective for analyzing rugged specimens with EDS.

- Click on the Mode button in the EOS Monitor window and then click on the NOR button of EOS mode in the Scan Control window. Observe the SEI of the specimen in the NOR (normal) mode using a small OL aperture, adjust the centering knobs of the OL aperture and focus the SEI using the FOCUS knob on the Operation Panel.
- Click on the Mode button in the EOS Monitor window and then click on the LDF button or MDF button of EOS mode in the Scan Control window.
- 3. Focus the image using the FOCUS knob on the Operation Panel.
- 4. Click on the NOR button of EOS mode to return to the normal mode.

10.4 Observing Mixed Image (ADD)

Using ADD, any image signals, for example COMPO and XR1, can be mixed on an image. ADD is useful for understanding the composition distribution of the specimen quickly and for deciding on the analysis point.

- Open the Operation window by selecting Operation from the Setup menu. Then select ADD and the names of the other image signals that you want from Signal Name in the Operation window.
- **2.** If an X-ray image is to be observed, set the probe current and WDS by the procedures in Sections 11.1 to 11.3.
- Open the ADD IMAGE window by selecting Add Image from the Setup menu, then select the names of the signals that you want to add, and click on the APPLY button to activate the ADD settings.

Clicking on the $\mathbf{O}\mathbf{K}$ button closes the ADD IMAGE window.

 Select ADD from the pull-up menu that opens when you click on the image selector button of the observation condition display in the bottom right of the basic screen.

Then, the image of the sum of the signals (ADD) is displayed.

5. When the ADD image is displayed, open the Contrast/Brightness window by selecting Cont./Bright. from the Column menu, and select the name of a signal to adjust the contrast and brightness in the ADD image.

11 BASIC OPERATIONS FOR X-RAY ANALYSIS (WDS)

11.1 X-ray Detection

When the computer system is started up, the conditions stored in advance such as the X-ray detector HV, the amplifier gain and the base level and window of the SCA (pulse-height analyzer) are set. An example of them is as follows.

Crystal	SCA Mode	Gain	Base	Window
ТАР	Diff	32	0.7 V	9.3 V
STE, LDE2	Diff	64	0.7 V	9.3 V
PET	Int	64	0.7 V	_
LIF	Int	32	0.7 V	_
LDE1	Diff	64	0.7 V	9.3 V

Table 2Example of SCA settings

Each X-ray detector HV is set up so that the pulse height of a specified characteristic X-ray has the value shown below with the gain shown above for each crystal.

Crystal	X-ray	Pulse Height
ТАР	Mg Kα	4.1 V
STE, LDE2	C Κα	2.5 V
PET	Τί Κα	5.8 V
LIF	Cu Ka	5.0 V
LDE1	Ο Κα	3.0 V

Table 3Example of pulse-height settings

- 1. Reset the conditions of the SCA using the SCA Control window that is opened by clicking on the SCA button in the SPC window.
- Generate the electron probe (Chapter 9, "GENERATION OF ELECTRON PROBE AND ALIGNMENT OF ELECTRON COLUMN"). Then set the PCD button to IN on the EOS Monitor window.
- **3.** Adjust the probe current to the order of 10⁻⁸ A using the **PROBE CURRENT** knob on the Operation Panel.
- 4. Mount the standard specimen holder on the specimen stage. Then move it to the position of a ZrO₂ standard specimen by using the Stage Monitor window that is opened by clicking on Stage in the Monitor menu. Observe the OM image and focus it by using the Z arrow button of the Joystick Controller.

5. Then turn the PRB SCAN button off and set the PCD button to OUT. A bright spot can be observed on the OM image. Focus the bright spot using the FOCUS knob. Now the probe position (while PRB SCAN is off) is confirmed.

- 6. Select **Peak Search** from the Monitor menu to open the Peak Search window.
- 7. Mount the specimen to be analyzed on the specimen stage, observe the OM image and focus it.
- 8. Turn the PRB SCAN button on and set the PCD button to OUT, observe the SEI or BEI of the specimen and determine the position for analysis on the specimen.
 - \swarrow If you have moved the specimen, be sure to focus the OM image.
- **9.** Move the position for analysis to the probe position (while **PRB SCAN** is off). Then set the **PCD** button to **IN**.
- **10.** Open the Element Condition window by clicking on the **Element** button in the Peak Search window.
- **11.** Specify the element to be detected, an X-ray line, a channel number, a crystal and a **Pksk** number. Then, execute peak search by clicking on the **Search** button.

As a result, a waveform, a peak position, a peak count and other information are displayed, and the spectrometer is set to the peak position. Here **Pksk no.** is the number that specifies the peak search range, usually 1 or 2.

- **12.** Click on the **Save** button in the Element Condition window to save the peak position obtained by the peak search in the Element Condition window. The stored peak positions can be used in the Quantitative and Map analysis programs.
 - If the element conditions were saved in the Element Condition Window of the Quantitative or the Map analysis program, select the element condition by clicking on the **Set** button of the Element Condition window of an analysis program. Reading and setting the conditions, the characteristic X-rays can be detected and measurement condition can be checked.
 - When a peak search is executed by clicking on the **Search** button, the **PRB SCAN** is automatically turned off.

11.2 Setting SCA

Obtaining a pulse-height distribution curve for a specified X-ray signal and setting the SCA according to the pulse-height distribution are performed in the SCA scan window, which is opened by selecting **SCA scan** from the Monitor menu.

- Execute the peak search by using the Peak Search window and set the spectrometer to the peak position (Section 11.1). After that, close the Peak Search window.
- Open the Ratemeter window, set the PCD button to OUT and check the X-ray count rate. If the count rate is more than 10,000 cps, reduce the probe current to make the count less than 10,000 cps. Then set the PCD button to IN and close the Ratemeter window.
- **3.** Open the SCA scan window by selecting **SCA Scan** from the Monitor menu. Then click on the **Signal** button and select a channel.
- Click on the Base L button to open the Base L scan window. Then, specify the conditions for measuring the pulse-height distribution such as Lower (V),

Upper (V), **Step (V)** and **Stime (ms)**. Usually, default values have been set in advance for all conditions.

- 5. Click on the Start button to start the measurement. The pulse-height distribution curve is displayed.
- 6. The SCA parameters can be set according to the pulse-height distribution by using the SCA Control window, which is opened by clicking on the SCA button in the Base L scan window.
- Save the SCA parameters displayed in the Element condition window of the quantitative analysis program or the map analysis program so that the characteristic X-rays can be detected using the stored SCA parameters in the future.

11.3 Observing X-ray Images

- **1.** Detect an X-ray signal from the specimen and set the spectrometer to the peak position (Section 11.1).
- **2.** Set the Operation Panel and viewing display as described below.

X-RAY in IMAGE SELECT:	XR1-5 (Select channel number)	
SCAN SPEED:	3 or more for observation	
	(If X-ray count is high enough, also 1 or 2)	
	3 or more (for photo)	
PRB SCAN:	On (Lit)	

- **3.** Set the **PCD** button to **OUT**. An X-ray image is observed.
- **4.** Observe the X-ray image and set the probe current to an appropriate value.
- **5.** To take a photo, first observe an X-ray image, and then push the **PHOTO** button on the Operation Panel (it is better to freeze the image before pushing the **PHOTO** button).
- When observing a scanning image at high magnification, clicking the icon + at the upper right of the viewing display switches the electron probe to the spot mode, and the probe can be positioned by dragging the mouse. In this way, it is possible to generate the characteristic X-rays from a small part of the specimen surface and to check for the presence of any elements.

12 EDS (JXA-8200 ONLY)

The **EDS** icon of the EPMA Main Menu is used for acquiring EDS (energy dispersive spectrometer) spectra, displaying data and identifying elements.

EDS	Display	
EDS	End	
EDS	Calib	
EDS	Analyzer	
EDS	Reset	
EDS menu		

Clicking on the **EDS** icon starts the program and displays the EDS menu. An outline of the program and its operations are presented below.

12.1 Outline of EDS Program

Clicking on EDS in the EDS menu displays the EDS Home Window.



EDS Home Window

Display of spectra

In the EDS Home Window, two spectra are displayed. The one on the upper right is a spectrum showing the full ranges of energy and count rate, and the other is a partial spectrum of which the ranges can be enlarged or reduced. Besides these, measuring conditions such as Preset, live time, dead time (DT), Aperture #, count rate and ROI count are also displayed.

• Aperture

An aperture in front of the EDS detector is selected using the **Aperture #** button. Aperture #1 is the largest one. The aperture gets smaller as the number increases. Aperture #5 is the smallest and #6 indicates that the aperture is closed.

Preset

The **Preset** button is used to preset the time or count for measurement.

• Acq

Clicking on the Acq button starts the measurement. Data are acquired in the data memory (front memory) specified using a button numbered 1, 2, 3 or 4 and spectra are displayed in the EDS Home Window.

Numbered buttons for data memories

There are four data memories that are specified using the numbered buttons. Of these, the spectra in two memories can be displayed simultaneously. One is the spectrum in the front memory, displayed as bars after you click the left mouse button on a numbered button. The other is the spectrum in the back memory, displayed as dots after you click the right mouse button on a numbered button.

Sample window

Selecting **Sample** from the File menu opens the Sample window where you can input the group name, the sample name and the comment for the sample to be measured in the input boxes.

• Save button in the Sample window

Click on the **Save** button, and then the data in the front memory are stored on the hard disk.

• Group button in the Sample window

Clicking on the **Group** button in the Sample window shows the list of the group names of data saved previously and clicking on the **Sample** button shows the list of the sample names.

After specifying a group name and a sample name and number in each list, click on the **Load** button to load the specified data in a desired data memory.

Energy and count rate

Clicking on a desired position in the spectra area in the EDS Home Window sets a cursor there and displays the energy and count rate at this point. The cursor can also be moved to the left by clicking the left button of the mouse on the \bigcirc button and to the right by clicking the right button on it.

Enlarging or reducing the partial spectrum

The energy range of the partial spectrum is enlarged using the button marked $[+ \rightarrow]$ and reduced using the button marked $[+ \rightarrow]$. The + and – keys of the keypad can be used. The count rate range is controlled using the arrows Δ \Box and **AUTO** buttons at the right side of the spectrum.

Operation menu

Clicking on **Operation** in the EDS Home Window displays the Operation menu; clicking the right mouse button on the spectrum displays the short-cut menu.

The Operation menu has the following items.

• Periodic Table

The periodic table of the elements is displayed where the KLM marker and the label for a specified element can be displayed in the spectrum. They are not displayed if you do not want them to be. Pressing the 4 or 6 key of the keypad shifts the atomic number of the KLM marker down or up by one.

• Peak ID

Selecting **Peak ID** displays element names and X-ray lines at the position specified with the cursor in the spectrum, and the labels of the specified elements can be displayed. They are not displayed if you do not want them to be.

• STD Profile

The profile data of standard samples are generated, deleted and searched.

• Spectra ROI

The window for setting the ROI (Region of Interest) is displayed where ROIs are set or deleted for the specified peaks.

• Analysis

Identification of the elements and semi-quantitative analysis are carried out for the spectrum in the front memory.

- In **Auto Ident**, the elements are identified automatically and the element names are output.
- In the Element List, specify the elements for semi-quantitative analysis.
- In **Semi. Quant**, the semi-quantitative analysis is executed. In this case, the STD Profiles prepared in advance are used for the standard data.

12.2 Operation of EDS Program

An example of the operation is given below.

- Mount the specimen for analysis on the specimen stage, observe an SEI or BEI of the specimen, determine the position for analysis on the specimen and irradiate it with the electron probe.
- 2. Click on the **Preset** button and set the measurement conditions.
 - To specify the measuring time, set **Preset** to **ON**, select **Live Time** and specify a measuring time in seconds.
 - If Auto clear is selected, the spectrum is cleared automatically before acquisition.
 - To determine the rough chemical composition quickly, select **cyclic** and specify a repeat acquisition time. When **Auto ID** has been selected, automatic element identification is also carried out at the same time.
 - To get a higher count rate, select T1 or T2 as the count mode, whereas to get a precise spectrum, select T3 or T4.

- **3.** Specify the number of the data memory to be used for the front memory by clicking a numbered button.
- **4.** Select Aperture **#1** using the **Aperture #** button.
- 5. If the Auto clear button is not selected, clear the spectra shown in the EDS Home Window by clicking on the Mem Clear button, and start the measurement by clicking on the Acq button.
- 6. Check the value of DT (dead time) at the top left of the partial spectrum. If the DT is greater than 30%, decrease the probe current or increase the Aperture # by clicking on the Aperture # button to get the DT less than 30%.
- Click on the Acq button to stop the measurement. Clear the spectra and click on the Acq button to start the measurement again. The measured spectra are displayed.
- 8. Select Analysis from the Operation menu in the EDS Home Window and click on Auto Ident to identify the elements. Or, click the right mouse button on the spectrum and select Semi. Quant from the popup menu.
- **9.** Enlarge the partial spectrum, set the cursor on the spectrum and confirm the identified elements again.
- **10.** To confirm the elements, use the functions of the **Periodic Table** or the **Peak ID** if necessary.
- **11.** After confirming the elements, display the labels for those elements on the spectrum or delete them by clicking on **Periodic Table** or **Peak ID** if necessary.
- **12.** Execute semi-quantitative analysis by selecting **Semi**. **Quant** from the Analysis menu after specifying the elements with the Element List. Or, click the right mouse button on the spectrum and select **Auto Ident** from the popup menu.

12.3 Start-up of Minicup EDS Detector

A Minicup EDS detector does not need liquid nitrogen (LN_2) when it is not being used. The procedure for evacuating and cooling the detector, from room temperature to being ready for analysis, is as follows.





Minicup EDS detector

Minicup evacuation controller

- 1. Confirm that the **TEMP–ROOM** lamp of the minicup evacuation controller is lit.
- 2. Confirm that the EVAC lamp is on. If it is not, wait until the EVAC lamp lights when the specimen chamber reaches high vacuum.
- **3.** Evacuate the detector and pour in LN₂ as follows:
- **a.** If the electron probe is being generated, push the **ACCEL VOLTAGE** button of the Main panel to turn the accelerating voltage off.
- **b.** Push the **START** switch of the Minicup evacuation controller.
- c. Wait about 15 minutes during the evacuation.
- **d.** While the **COOL** lamp is blinking, pour LN_2 into the dewar. To avoid overflowing due to boiling, first fill the dewar to 1/3 of its capacity and wait for the boiling to cease. Then fill it.
- e. A few minutes later, confirm that the COOL lamp is lit and the READY lamp is blinking.
- f. Wait about one hour for the READY lamp to light steadily.
- **4.** Mark the checkbox of **Bias** in the Analyzer window, and then you can use the EDS analyzer.

13 QUALITATIVE ANALYSIS (WDS)

13.1 Outline of Qualitative Analysis Program

This program is used for acquiring spectra of unknown samples using wavelength dispersive spectrometers (WDSs), displaying the spectra and identifying elements. The spectra can be displayed during measurement (in real time) and the elements are automatically identified at the end of the measurement with the names being attached to the peak positions.

The element names are output with the ranks of certainty.

- If the first-order reflection line of K α , L α or M α for an element is detected, the element is placed in the B rank.
- If the element in the B rank fits one of the conditions shown below, it is placed in the A rank.
 - The atomic number of the element is a number from 4 to 17.
 - The first-order reflection lines of K α and K β are found in the same spectrum, and moreover, K $\alpha \ge K\beta$ in net counts.
 - The first-order reflection lines of $L\alpha_1$ and $L\beta_1$ are found in the same spectrum, and moreover, $1.5 \times L\alpha \ge L\beta$ in net counts.
 - The first-order reflection lines of M α and M β are found in the same spectrum, and moreover, $1.5 \times M\alpha \ge M\beta$ in net counts.
 - The first-order reflection lines of K α and L α are found in two spectra that are acquired simultaneously using different crystals.
 - No element is in the A rank, but this element is the only one in the B rank.
- If a line of an element in the B rank overlaps with a higher-order reflection line of another element in the A rank, the element in the B rank is deleted.
- In the ID Doctor, a database that has information on all X-rays including intensity and FWHM and interfering X-rays of each element is examined, and the probability of presence of elements is checked precisely.

13.2 Operation of Qualitative Analysis Program

In general, analysis programs are composed of two parts, measurement (data acquisition) and processing (analysis data processing).

Clicking on the **Analysis** icon in the EPMA Main Menu displays the pull-down menu for the measurement that can be started after selecting an item, and clicking on the **Process** icon displays the pull-down menu for processing. The menus are shown on the next pages.

However, the processing of data acquired using **EDS Qualitative** in the Analysis menu is executed from the EDS menu.



Menu items for Qualitative Analysis

13.2.1 Measurement program for Qualitative Analysis

Clicking on **Qualitative Analysis** in the Analysis menu opens the function window showing the **Sample** and the **Measurement** buttons.

- 1. Clicking on the **Sample** button opens the Select Sample window where the group and sample names for the data to be stored (up to 14 characters for each name) can be entered.
 - a. In the Select Sample window, the list of the group and sample names entered previously is displayed. If the measurement is to be carried out under one of the sample names displayed in the list, select it and click on the OK button.
- **b.** 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.
- C. If the sample name is in another group, click on the Group button to open the Select Group window and select a group name; or after clicking on the New button, enter a new group name. The maximum length is 14 characters.
- 2. Clicking on the **Measurement** button displays the Measurement menu.

Spectrometer Condition (4)				
On-line Semi-Quant (ON)				
EOS Condition (Acc. 20.0 kV)				
Stage Condition (3)				
Condition Load				
Condition Store				
Print-out Condition				
Additional Function				
Survey Measurement				
Preset Measurement				

Measurement menu for Qualitative Analysis

Here, the measurement is executed after measurement conditions have been input. For the measurement, at least **Spectrometer Condition**, **EOS Condition** and **Stage Condition** should be input. If these conditions have been stored in a file using **Condition Store**, the stored conditions can be read and set later using **Condition Load**.

- **3.** Clicking on **Spectrometer Condition** in the Measurement menu opens the Spectrometer Condition window for setting the measurement conditions of the spectrometers. The conditions used previously are displayed. Change the conditions if necessary and click on the **OK** button.
- a. No. of Spectra is set usually to 3 or 4. Almost all elements (atomic numbers 5 to 92) can be detected in 4 spectra using 4 crystals, LDE2 (or STE), TAP, PET and LIF. The elements with atomic numbers over 11 (Na) are covered with 3 crystals: TAP, PET and LIF.
- **b.** For each spectrum, specify Channel, Crystal, Start and End positions of the spectrometers and other conditions.
- **c.** The ranges of the spectrometers are shown below as an example.

General (XCE, TXE)	65 – 250 mm
Н	88 – 240 mm
FCS	70 – 250 mm

- **d.** Step is normally set to 50 μm for TAP, PET and LIF, and to 100 μm for LDE2 (or STE).
- e. Dwell (sampling time) is normally set to 50 to 200 ms. If it is set to 50 ms, the total time will be about 3 minutes.
- f. Confirm that the conditions such as High V. (for the detector), PHA gain, Base L and Window of the SCA are those set in the Initialize menu by an installation engineer (Section 11.1, "X-ray Detection").
- **4.** To get a semi-quantitative result automatically after measurement, select **Yes** on the On-line Semi-Quant window.
- **a.** When some elements should be taken into account, select **Include** and then click on the elements to be included.
- **b.** On the other hand, when some elements should be omitted, select **Exclude** and then click on the elements to be excluded.
- **5.** Clicking on **EOS Condition** opens the EOS Condition window for setting the EOS (Electron optical system).
 - a. The EOS conditions used previously are displayed. Since the generation of the electron probe and the electron column alignment are usually performed before the start of the analysis, click on the **Read** button here to read the present EOS conditions, and then click on the **OK** button.
- **b.** Clicking on the **Set** button sets the EOS to the conditions used previously. In this case, it is recommended to check the previous conditions that are shown on the EOS Condition window, and to adjust the alignment and focus of the electron probe.
- 6. Clicking on Stage Position opens the Stage Condition window where the positions for analysis of the specimen are set.
 - **a.** When a recorded sample name is selected, the list of the positions measured previously is displayed. Clicking on the line indicating the desired position can set one of these positions.
- b. To input a new position for analysis, click on an unused line for the recorded sample name, and click on the **Pos. Input** button to open the Stage Condition Input window. Input a comment in the Comment input box (40 characters maximum). Then input the coordinates in the X, Y and Z input boxes or click on the desired position on the two-dimensional display, click on the **Move** button to move the specimen stage to the specified position and adjust the position with the Joystick Controller, focusing the OM image,

adjust the position with the Joystick Controller, focusing the OM image, to determine the position for analysis.

- C. Click on the Read, the Store and the Apply buttons in this order or click on the Read & Apply button. The specified position is set for the analysis and displayed on the highlighted line in the Stage Condition window.
- d. To carry out the measurement in the One-by-One mode, close the Stage Condition Input window and click on the One-by-One button. Then click on the Acquire button in the One-by-One Measurement window.
- e. Repeat Steps b to d to measure many positions on the specimen in the One-by-One mode.
- f. To carry out the measurements of many positions in the Preset mode, after Steps b and c, click on the arrow button ▼ on the upper right area in the Stage Condition Input window and repeat these steps. Many positions on the specimen can be set for the analysis in succession. Or click on the STORE button on the Joystick Controller; this saves the present X, Y and Z coordinates as a measurement point, and proceeds to the next point. If the last character of the comment is number, it automatically increases by one.
- **g.** Confirm that the Preset On/Off button is on for each position in the Stage Condition window and close this window.
- Clicking on Preset Measurement opens the Preset Measurement window where the measurement is performed in the Preset mode when you click on the Acquire button.
- In Survey Measurement, the measurement is performed for surveying the outline of the analysis. Measured data are stored at position No. 99999 and the data are replaced after another Survey Measurement has been performed.

13.2.2 Data processing program for Qualitative Analysis

Clicking on **Qualitative Analysis** in the Process menu opens the window for displaying the spectra for data processing. Here, the data now being acquired (in real time) or acquired previously are displayed and analyzed.

Displaying data and spectra

Processing data now being acquired (in real time)

- 1. Click on the **Realtime** button and specify the number of spectra to be displayed.
- 2. Click on the Start button.

The spectra being acquired are displayed in real time.

3. To stop the display or after finishing the acquisition, click on the **Stop** button. Then proceed to ■ Analyzing data and spectra below.

Processing data acquired previously

- 1. Click on the **Sample** button.
 - The Sample window opens.
- 2. Select the sample name of data to be displayed from the list of the sample names.

If the sample name is in another group, first click on the **Group** button and then select another group name.

- **3.** Click on the **Max Spectra** button to specify the number of spectra and then specify the spectra to be displayed.
- **4.** Click on the **OK** button.

The spectra, the elements identified automatically and the measurement conditions are displayed.

Then proceed to **A**nalyzing data and spectra below.

Analyzing data and spectra

Click on the Operation button in the Data display window.

Zooming

The Operation menu opens. Both **Spectra Display** and **Spectra Calculation** have sub-menus as shown below.

Operation	
Spectra Display	
KLM Marker	
Peak ID	
Save ID Results	
Off-line ID	
Spectra Calculation	
Redraw Spectra	
Print-Out	
Spectrum Deconvolution	
Semi-Quant Analysis	
ID-Doctor	
Spectrum Analysis	
Spectra Search	

Operation menu

Spectra Display menu

Spectra Display

Ymax r % Normalize

Select Xmin ~ Ymax

Kind of Assignment

Display Parameter

Replace Spectrum

Quant. Background

Reset Spectra

Mixed Spectra Display

Window Lavout

Spectrum Type

Spectrum Color

Display Axis

Draw Mesh

Write Text

Spectra Calculation menu

Spectra Calculation

Background Subtraction

Multiply (SP * k)

(SP - k)

(SP + k)

(SP/k)

Spectra Sub (SP1 - SP2)

Spectra Add (SP1 + SP2)

Dead time Correction

Smoothing

Sub

Add

Devide

Reset

2nd Derivative

Spectrum Shift

Result Store

a. Selecting Spectra Display opens the Spectra Display menu.

• Zooming

Selecting **Zooming** from the Spectra Display menu allows you to zoom in on a specified range of a spectrum. In the zoomed display, the identified elements can be confirmed in detail. The range to be zoomed is specified by clicking on a corner of the rectangular zoom area and dragging the pointer in the diagonal direction to the opposite corner.

• Mixed Spectra Display

Selecting **Mixed Spectra Display** from the Spectra Display menu allows multiple spectra to be displayed at the same time in one spectrum display area when Spectrum Mode–**Single** is selected. This display is useful for comparing spectra to each other. In **Single** mode, the value of base offset (0 - 1) controls the base level of each spectrum.

• Display Axis

By clicking on **Display Axis** in the Spectra Display menu, you can change the marking of the abscissa of the graphs of the spectra from detection position (mm) to wavelength (nm) or energy (keV).

• Write Text

Selecting **Write Text** from the Spectra Display menu allows you to enter comments to be written in the spectra display area.

- **b.** Selecting **KLM Marker** displays the periodic table of elements. In the window, if you click on an element symbol, markers will appear on the spectra at the positions of characteristic X-rays on the spectra, and if you click on the **Set** button, the element name will appear on the spectra.
- **C.** Selecting **Peak ID** displays the window where the possible names of characteristic X-rays at a specified peak on a spectrum are indicated in a list. If you click on the Set button, the selected element name is indicated on the spectrum.

14 QUANTITATIVE ANALYSIS

14.1 Outline of Quantitative Analysis

Programs for quantitative analysis are used for measuring the X-ray intensities of standard and unknown specimens, calculating relative intensities, performing corrections on them and obtaining concentrations of specified elements. The elements to be analyzed range from Be to U, but rare gases and radioactive elements are excepted. The characteristic X-ray lines used in analysis are K α , K β , L α , L β , M α and M β .

14.2 Operations of Programs for Quantitative Analysis

The programs are composed of two parts, measurement (data acquisition) which can be started from the Analysis menu and processing (analysis data processing) which can be started from the Process menu.

The software for measurement is divided into two independent programs, Standard Analysis for measuring standard specimens and Quantitative Analysis for measuring unknown specimens. The standard specimens should be measured before the unknown specimens. If a standard specimen is not available, select **CAL–STD** as the standard in the quantitative analysis, and the semi-quantitative result of the element is shown.

14.2.1 Standard Analysis program

Selecting **Standard Analysis** from the Analysis menu opens the window showing the **Sample** and the **Measurement** buttons.

- 1. Clicking on the **Sample** button opens the Select Sample window where the sample names for the data to be stored can be recorded. The operation is the same as that of the qualitative analysis.
 - Refer to Section 13.2.1, "Measurement program for Qualitative Analysis", Step 1.a.
- 2. Clicking on the Measurement button displays the Measurement menu.

Standard Type (Oxide)					
Element Condition (O Al O)					
EOS Condition (Acc. 20.0 kV)					
EDS Condition					
Stage Condition					
Condition Load					
Condition Store					
Check Data					
Print-out Condition (OFF)					
Measurement Mode (WDS)					
Additional Function (OFF)					
Survey Measurement					
Preset Measurement (1)					

Measurement menu for Standard Analysis

Set Standard Type, Element Condition, EOS Condition, Stage Condition and so on; then carry out the measurement. If these conditions have been stored in a file using Condition Store, the stored conditions can be recalled and set later using Condition Load.

- If the EDS is to be used simultaneously, the EDS Home window should also be opened. The window can be minimized after necessary operations in the window have been completed.
- Select Standard Type from the Measurement menu and specify the type of sample, Metal (element measurement) or Oxide (chemical-formula measurement).
- 4. Selecting Element Condition opens the Element Condition window for setting the conditions of elements. Here, input the composition of the standard specimen, the names of elements to be measured, the spectrometer conditions and other information.

The conditions used previously are displayed. Those conditions can be used after changing only the items desired to be changed.

- a. Click on the Standard Composition button to open the window for inputting the composition of the standard specimen. In this window, specify the data type and then input the element and composition data. Click the OK button, and close the window.
 - When inputting the data, select **Atom** for atomic ratio data, **Mole** for mole ratio data or **Mass** for mass percentage data.
 - If **Oxide** has been selected for the type of sample (Step 3 above), input the value of valence.
 - If the value shown automatically is not correct, input the correct value.
 - Even if an element name only is input in the Element column, the element is treated as an oxide with the specified valence, unless the data are input as **Atom**.
- **b.** Enter the names of elements to be measured.
 - For measurement with the WDS, click on the **Elements** button in the WDS area to open the WDS Elements window, and then in the window specify the names of elements to be measured by clicking on the element symbols in the periodic table. To delete elements input previously, click on the element symbols in the Select Elements area, displaying them in reverse video, and click on the **Clear** button.
 - For measurement with the EDS, click on the **Elements** button in the EDS area and operate similarly to specify the elements to be measured. To delete elements input previously, do as above.
- C. When you have entered the names of elements to be measured in the WDS area, click on the Condition button. The WDS Element Condition window showing the measuring order of each channel appears. If many elements are assigned to a specific channel, it is better to move elements to other channels by dragging the element button so as to distribute them as equally as possible.
- d. Click on the Condition button in the WDS area to open the WDS Element Condition window where the spectrometer and the SCA conditions set automatically or used previously are displayed. Click on the OK button to measure with these conditions. To select other conditions, click on the Elem-i ("i" is a number) button to open the WDS Element Data Table window and select desired conditions using the Select No. button.

- e. To change the condition values, click on the value button and input desired values. Since the values in the WDS Element Data Table used previously can be used for other measurements, however, new condition values should be made after clicking on the New button rather than changing the conditions used previously.
 - In **Spect. Pos**, the counted peak position of the spectrometer has been input for your instrument at our factory. If you have performed peak searching with **Peak Search** in the Monitor menu, the peak position obtained can be input here with the **Read** button in the Element Condition window.
 - In **Back(+)** and **Back(-)**, the positions of the background, usually ±5 mm as defaults, are input. If the specimen is a compound and the peaks of other elements are detected around the background positions, you should change these positions.
 - In Time/Count/Area, specify Preset Time, Preset Count or Area Count.
 - In **Peak Seek W**, usually select 1 or 2 to specify the peak search range. This is the same as **Pksk no.** in **Peak Search** in the Monitor menu.
 - Mes.Time and Bac.Time are preset values for the Preset Time, while Mes.Count and Bac.Count are those for the Preset Count.
 - The SCA conditions can be set in the SPC window that is opened by selecting **Ratemeter** or **SCA scan** from the Monitor menu (@ Section 11.1). To read the set SCA conditions, click on the **Select No.** button in the WDS Element Data Table window, and then click on the **Read** button.

Click on the **OK** button to finalize the conditions.

- f. When you have entered the names of the elements to be measured in the EDS area, click on the Condition button in the EDS area to open the EDS Element Condition window. Here, ROIs set automatically or used previously are displayed. You can select them by clicking on the OK button. To select different conditions, operate in the same way as for the WDS.
- 5. Clicking on EOS Condition opens the EOS Condition window for setting the EOS (electron optical system). Here the EOS conditions used previously are displayed. As the generation of the electron probe and the electron column alignment are usually performed before the start of the analysis, click on the Read button here to read the present settings. Then, click on the OK button. After reading the column conditions, also check the probe scan conditions.
- 6. For measurement with the EDS, clicking on EDS Condition opens the EDS Condition window for inputting EDS measurement conditions. Acquire some data in advance with the Acq button in the EDS program (Section 12.2 Operation of EDS Program), set conditions for the measurement, and click on the Read button to read the condition that are set at present. Then, click on the OK button.
- Clicking on Stage Condition opens the Stage Condition window where you can set the positions of the specimen for measurement. The operations are almost the same as those for qualitative analysis (@ Section 13.2.1, "Measurement program for Qualitative Analysis", Step 6).
- **a.** When a recorded sample name is selected, the position measured previously is displayed. This position can be used for measurement again.
- b. To input a new position for measurement, click on the Pos. Input button to open the Stage Condition Input window. Then, input the coordinates in the X, Y and Z input boxes, or click on the desired position on the two-dimensional

display. Click on the **Move** button to move the specimen stage to the specified position and adjust the position with the Joystick Controller, focusing the OM image, to determine the position for measurement.

- Click on the Read, the Store and the Apply buttons in this order or click on the Read & Apply button. The specified position is set for the measurement and displayed on the highlighted line in the Stage Condition window.
- d. To carry out the measurements in the One-by-One mode, close the Stage Condition Input window and click on the One-by-One button in the Stage Condition window. Then, click on the Acquire button in the One-by-One Measurement window.
- e. To carry out the measurements in the Preset mode, confirm that the Preset On/Off button is on for each position in the Stage Condition window and close this window.
- 8. Repeat steps 1 to 7 for each standard sample.
- 9. Clicking on Preset Measurement opens the Select Preset Samples window.
- **10.** Click on the **Mes** buttons for the samples to be measured, check total sample numbers and click on the **Acquire** button to start the measurements.

14.2.2 Quantitative Analysis (for unknown specimens) program

Selecting **Quantitative Analysis** from the Analysis menu opens the window showing the **Sample** and the **Measurement** buttons. Since the procedure is almost the same as for standard analysis, it is only outlined below.

- 1. Clicking on the **Sample** button opens the Select Sample window where the sample group and sample names for the data to be stored can be recorded.
- 2. Clicking on the Measurement button displays the Measurement menu. Set the measurement conditions; then carry out the measurements.

Corr. Method (Oxide ZAF)				
Element Condition (W:6 E:3 C:2)				
EOS Condition (Acc. 15.0 kV)				
EDS Condition				
Standard Condition				
Substrate Composition				
Stage Condition (50)				
Condition Load				
Condition Store				
Print-out Condition (ON)				
Measurement Mode (WD-ED Para.)				
Additional Function (OFF)				
Survey Measurement				
Preset Measurement				

Measurement menu for Quantitative Analysis

If the EDS is to be used simultaneously, the EDS Home window should also be opened.

- **3.** Selecting **Corr. Method** from the Measurement menu opens the Correction Method window. Specify the type of sample, **Metal** or **Oxide**, and select a correction method.
- **4.** Selecting **Element Condition** from the Measurement menu opens the window where you can set the elements to be measured, the spectrometer and other conditions.
 - **a.** Input the names of elements to be measured in the WDS (or EDS) area in the same way as for the standard analysis.
 - b. The entries in the Element Data Table should be the same as those of the standard analysis for each element except for the positions of the back-ground, Peak Seek W and preset values for Time/Count. Peak Seek W is usually 1 for ordinary measurements of unknown specimens.
 - **c.** In the CAL area, input the names of elements that are not to be measured but to be processed in calculations, the modes for calculating them and the entries for them. The calculation modes should be specified for the elements which are known to exist but are not required to be analyzed or cannot be analyzed with the EPMA.
 - **d.** If the sample type is Oxide, click on the **Valence** button and specify the valence for each element.
 - e. Clicking on the Condition button in the CAL area opens the CAL Element Condition window where the calculation modes and the data (fractions) set automatically or used previously are shown. To change the conditions, click on the Elem-i ("i" is a number) button to open the Calculation Mode window, and then specify the Mode and the Fraction. The Mode is selected from the list shown below for each element.

Calc. Mode	X-ray
Fix	The concentrations of the specified elements (or oxides) are input as known values and the data of measured elements are processed.
Atomic Ratio	The atomic ratios of the specified elements to the measured elements are input as known values and the data are processed.
Mole Ratio	The molar ratios in oxide analysis are input as known values and the data are processed.
Mass Ratio	The mass ratios are input as known values and the data are processed.
Difference	An element (or oxide) is specified as the element remaining after all other elements have been accounted for and the data are processed.
Anion	An element is specified to be combined as anions with the measured metal elements and the data are processed.

Table 4	Calculations	mode for (Ouantitative	Analysis
	Curculations	mowe for v	V autoreautor v v	. Aller y DID

5. Selecting **EOS Condition** from the Measurement menu opens the EOS Condition window for setting the EOS. Usually click on the **Read** button here to read the present conditions.

The probe conditions other than **Probe Current** should be the same as those for the standard analysis.

6. For the measurement with the EDS, selecting EDS Condition from the Measurement menu opens the EDS Condition window for setting the measurement conditions of the EDS.

 \swarrow Set it to the same conditions as those for the standard analysis.

- 7. Selecting Standard Condition from the Measurement menu opens the Standard Condition window. Confirm the data of standard specimens. The data should be taken with the same Acc.V, spectrometer and crystal as those in the conditions specified in Steps 4 and 6. To select other data, click on the Elem-i ("i" is a number) button to open the Standard Data Select window and select the desired data. When no appropriate standard specimen was measured, select CAL-STD which estimates the X-ray intensity of pure material from a semi-quantitative calibration curve.
- 8. Selecting Print-out Condition from the Measurement menu opens the Print-out Condition window where the items to be printed are specified. To turn printing on, set Measurement Condition on. If the sample type is Oxide and its chemical formula is anticipated in advance, click on the No. of Oxygen button and input the number of oxygen atoms in the formula (the default is 24).

Then, the number of the other element atoms in the formula will be output in the **Cation** column for the measured result.

9. Selecting **Stage Condition** from the Measurement menu opens the Stage Condition window. In the window, set the positions for measurement on the specimen.

K For details, refer to the procedure for qualitative analysis.

10. Click on the One-by-One button in the Stage Condition window to open the One by One Measurement window or select Preset Measurement from the Measurement menu to open the Preset Measurement window. Then, click on the Acquire button.

The measurements are carried out and the results are output.

14.2.3 Data-processing program for Quantitative Analysis

Selecting **Quantitative Analysis** from the Process menu displays the menu for data processing.

Qualitative	Analysis		
Quantitative	Analysis 🖻		
Line	Analysis	Summary	
Мар	Analysis	Off-line	Correction
Phase Analysis			
Miniature Display			

Data-processing menu for Quantitative Analysis

In **Summary**, the data taken in the quantitative analysis are displayed and rearranged. In **Off-line Correction**, the calculations for correction are performed again for other measurement conditions or for new data.

14.2.4 Summary program for Quantitative Analysis

Selecting **Summary** from the data-processing menu opens the window showing the **Sample** and **Summary** buttons.

- 1. Click on the **Sample** button to open the Select Sample window, in which select the group and sample name for the data to be displayed.
- 2. Click on the **Summary** button to display the Summary window where the data of a specified item at specified positions are displayed in a specified form.
- a. Clicking on the Select All button specifies all measured positions.
- b. After clicking on the Clear All button to cancel the positions specified previously, specify a position with the Single button. Clicking on the Check data button displays the Text Editor window, and you can edit some information such as comments.
- C. After you click on the Normal button, you can output all data by clicking on the Type out button.
- d. Select an item from the items ranging from Mass% to L-value, click on the Type out button to output the data of the selected item. To output the data from left to right, click on the Row button, and from top to bottom, the Column button.
- e. To output all measured positions, click on the Stage button, and to output the data for standard specimens, click on the Standard button.
- **f.** To export the results to PC spreadsheet software, set the spreadsheet button on.

14.2.5 Off-line correction program for Quantitative Analysis

Selecting **Off-line Correction** from the data processing menu opens the window showing the **Sample** and **Conditions** buttons. As most operations are the same as those of measurement in the quantitative analysis, only the differences are described below.

- Click on the Sample button and select the group and sample name for the data to be reprocessed. If corrections are to be performed for new data, click on the New button and record a group and sample name.
- 2. Clicking on the Conditions button displays the Off-line Correction menu.

Analysis(WD/ED Quant.)
Corr. Method (Metal ZAF)
Element Condition (W:3)
EOS Condition (Acc. 20.0 kV)
EDS Condition
Standard Condition
Standard Data
Substrate Composition
Condition Load
Condition Store
Print-out Condition (OFF)
Additional Function
K-ratio input & Correction
Intensity input & Correction
File read & Correction

Off-line correction menu for Quantitative Analysis

Here, the correction is executed after the conditions are input in the same way as for the measurement. To execute the correction, select one of the last three items from the Off-line Correction menu according to the data to be corrected.

- **3.** Selecting **Standard Data** from the menu displays the Standard data window showing a list of the data of the standard specimens. Here the standard data selected with Standard **Condition** can be checked in detail, modified or input.
 - a. To input data, click on the value button, then input numerical values directly.
- **b.** The take-off angle of X-rays can be changed by clicking on the **Takeoff** button.
- c. To make the modifications take effect, click the Save button.
- **4.** Selecting **K-ratio input & Correction** from the menu displays the K-ratio input & Correction window showing K-ratios in the selected data.
 - a. The data are corrected again if you click on the Apply button.
- **b.** The input data are corrected if you input a K-ratio directly from the keyboard and click on the Apply button.
- **C.** To correct the K-ratios read from other data, click on the **Read** button, specify the group and sample names and the number of the specimen, and click on the **Apply** button.
- 5. Selecting Intensity input & Correction from the menu displays the Intensity input window showing Current and Net Intensity of the selected data. The data are corrected when you click on the Apply button. Alternatively, the data can be input directly from the keyboard. Other operations are also the same as those of Step 4.
- 6. Selecting File read & Correction from the menu displays the File read & Correction window showing a list of the measured positions in the selected data. The data are corrected when you click on the Apply button after specifying positions with the buttons at the left end.

15 MAP ANALYSIS

15.1 Outline of Map Analysis

The program for map analysis is used for acquiring X-rays and image signals from a two-dimensional area on the specimen, storing the data on the disk and displaying maps of the data on the computer display. The data are acquired in a stage scan in which the electron probe is stationary and the specimen stage is driven to cover the specified area, or in a beam scan in which the electron probe is scanned on the stationary specimen. The program provides functions for simple image processing like smoothing, line profile, and distance measurement.

15.2 Operating the Map Analysis Program

The program is composed of two parts, measurement (data acquisition), which can be started from the Analysis menu, and analysis data processing, which can be started from the Process menu.

15.2.1 Measurement program for Map Analysis

Clicking on **Map Analysis** in the Analysis menu opens the window showing the **Sample** and the **Measurement** buttons.

1. Clicking on the **Sample** button opens the Sample window where the group and sample names for the data to be stored can be entered. The procedure is the same as that of the qualitative analysis.

Program for Qualitative Analysis".

2. Clicking on the Measurement button displays the Measurement menu.

Element Condition (W:6 I:1)
EOS Condition (Acc. 20.0 kV)
EDS Condition
Stage Condition (2)
Condition Load
Condition Store
Print-out Condition
Survey Measurement
Preset Measurement

Measurement menu for Map Analysis

Here, the measurement is executed after you set **Element Condition**, **EOS Condition** and **Stage Condition** (Pixel size, Pixel no. and other parameters). If the EDS is to be used simultaneously, the EDS Home window should also be opened.

- **3.** Clicking on **Element Condition** opens the window where the elements to be measured and the spectrometer and other conditions are set.
- **a.** Input the names of the elements to be measured in the WDS (or EDS) area as in standard analysis.

The Standard Analysis program" Step 4.

- b. For measurement using the WDS, click on the Condition button in the WDS area to open the WDS Element Condition window where the spectrometer and the SCA conditions set previously are displayed. If you wish to select other conditions, click on the Elem-i ("i" is a number) button to open the WDS Element Data Table window and select desired conditions using the Select No. button.
 - These spectrometer and SCA conditions are used for other programs as well, but the items from Back (+) to Bac. Count are not used for map analysis.
- C. To change the condition values, click on the value buttons and input desired values. For details, refer to the procedure for the standard analysis. In Spect. Pos, a counted peak position or the peak position used previously is displayed at first. After peak searching using Peak Search in the Monitor menu, the peak position obtained can be input here using the Read button.
- d. For measurement using the EDS, click on the Condition button in the EDS area to open the window where ROIs for the elements to be measured are displayed. If you wish to select other ROIs, click on the Elem-i ("i" is a number) button to open the EDS Element Data Table window and select desired conditions using the Select No. button.
 - ROI (Region of Interest) is the energy measurement range, determined by Start 1 (keV) and End 1.
- e. To change the ROIs, click on the value buttons and input desired values, or click on the **Read** button to read the present conditions after setting ROIs manually on a spectrum acquired in advance. The ROIs should be set not to overlap each other.
- f. To acquire image signals, click on the Signal button in the IMS area to open the Image Signal window. To specify the image signals, turn on the signal by clicking on the button to the left of its signal name. Present Contrast and Brightness values are read when you click on the Read button.
- 4. Clicking on EOS Condition opens the EOS Condition window for setting the EOS. Usually, click on the Read button here to read the present conditions as the electron column alignment is performed before the start of the analysis. In the beam scan, the magnification and scan area are controlled by the settings of scan conditions.
- 5. Clicking on EDS Condition opens the EDS Condition window for inputting EDS measurement conditions. Although the conditions used previously are displayed, the items Measuring Mode and Measuring Time are not used in the map analysis. Here, it is recommended to acquire some data in advance using the Acq button in the EDS program, to set conditions for the measurement and to click on the Read button to read the present conditions.
- **6.** Clicking on **Stage Condition** opens the Stage Condition window where you can set areas for measurement and measurement conditions on the specimen.
 - **a.** When a recorded sample name is selected, the areas measured and the conditions used previously are displayed. These areas can be set for measurement again.

- b. To input new areas for measurement, click on the Area Input button to open the Stage Condition Input window where the conditions are input and to open the Analysis area Working window simultaneously where the measurement areas are set.
- c. Input measurement conditions as discussed below:
 - In Scan Type, select a scan method, Stage scan by Y-axis or Beam scan. For stage scan, select a stage scan method, **Stage(uni)** which stands for uni-directional movement of the stage or **Stage(bi)** which stands for bi-directional movement. Stage scan is generally used for measuring areas wider than about 100 μ m, while Beam scan is for narrower areas. **Stage(uni)** is used for measurement with pixels less than about 10 μ m to reduce the influences of stage backlash. **Stage(bi)** is for larger pixels which are not affected by stage backlash. This method reduces the measurement time.
 - In **Stage Drive**, specify the step size of the stage driver, Normal step (0.5 μm minimum) or Micro step (0.02 μm minimum).
 - In **Pixels** (**X**,**Y**), input the numbers of pixels for the X and Y axes. Numbers of pixels from 10 to 1024 are accepted, but numbers from 200 to 600 are normal.
 - In **Pixel size** (**X**,**Y**), input the pixel sizes for the X and Y axes in μ m. For stage scan, the acceptable range is from 0.5 μ m (0.02 μ m for Micro step) to 1000 μ m. For beam scan, the pixel sizes are set automatically.
 - In **Dwell Time**, input the measurement time for each pixel in ms. The dwell times are normally set to 10 to 50 ms.
 - In **Magnification**, input the magnification of the image for beam scan. In beam scan, the measurement area corresponds to the scanning image and the width of the area depends on the magnification.
- d. Set the measurement area as discussed below:
 - For stage scan, the size and shape of the measurement area are determined by inputting the measurement conditions. The measurement area is set on the specimen by specifying the center, start or end point in the area. The start point is at the upper right corner of the area (rectangle), and the end point at the lower left corner.
 - (i) Input the coordinates of the desired point in the X, Y and Z input boxes or click on the desired position on the two-dimensional display; click on the **Move** button to move the specimen stage; adjust the position using the Joystick Controller to finalize the specified point; then focus the OM image and click on the **Read** button.
 - (ii) Click on the **Store** button, specify **Center**, **Start** or **End** and click on the **Apply** button.
 - (iii) Click on the Confirm button and the specimen stage moves to the start point. Focus the OM image here and push the TEST and STOR buttons. The specimen stage is moved to the four corners of the measurement area in sequence each time the STOR button is pushed. Focus the OM image at each point. The Z axis is controlled during measurement on the plane calculated from the coordinates of the four corners. Finally, click on the Apply button.
 - For beam scan, the measurement area corresponds to the central part of the viewing image which is 1280×1024 pixels. Therefore, the measurement area on the specimen is set by the magnification of the image. The electron beam is scanned on this area digitally with a maximum of $1024 \text{ dots} \times 1024 \text{ dots}$.

The pixel size is expressed as the number of dots per pixel and the acceptable pixel sizes in the program are 1, 2, 4 and 8 dots.

- To set the measurement area, observe the SEI or the OM image, adjust the area using the Joystick Controller to determine the measurement area and click on the **Read**, the **Store** and the **Apply** buttons in that order.
- e. The measurement in the One-by-One mode can be carried out with the **One-by-One** button in the Stage Condition window.
- f. To carry out the measurements of many areas, click on the arrowhead button ▼ on the upper right area in the Stage Condition Input window and repeat the area-setting procedure. Many measurement areas can be set on the specimen in succession.
- 7. The measurement in the Preset mode can be carried out with the Preset Measurement after confirming that the Preset On/Off button is on for each measurement area in the Stage Condition window.

15.2.2 Data processing program for Map Analysis

Clicking on **Map Analysis** in the Process menu opens the window for processing the map data. Here, the map data now being acquired (in real time) or acquired previously are displayed and analyzed.

- 1. Clicking on the **Realtime** button opens the Realtime Control window where the map display in real time is started.
- a. The maps are displayed when the **Start** button is pressed after specification of the order in which each map is to be displayed and the number of maps to be displayed.
- b. To control the color levels, click on the Stop button and then click on the Auto button for automatic control during measurement or click on the Manual button which allows the color levels to be set manually. Then start the display again.
- **C.** To stop the display or after finishing the measurement, click on the **Stop** button.

Then go to Step 3 for other displays.

- **2.** Map data acquired previously are displayed after you click on the **Sample** button and specify the sample name and the data.
 - **a.** Select a group and sample name for the data to be displayed.
- b. Click on the Max Maps[n] Colors[m] ("n" & "m" are numbers) button, specify the numbers of maps and colors, select the area in which to display the color bars and click on the OK button. To show the stage map with the same direction as that of the viewing display, select Real Image of stage scan maps from Map Rotation.
- C. Specify the data to be displayed in the Sample window. To show a scanning image or a saved OM image, click the Photo button, and select an image. The maps and the parameters of the specified data are displayed. Then go to Step 3 for other displays and analysis of the data.
- **3.** Clicking on the **Operation** button displays the Operation menu.

Here, the map data are reprocessed and displayed under different conditions. Simple image-processing operations like smoothing or sharpening and analyses like line profile or distance measurement can also be performed.

Operation Menu	
Map Display	7
Map Analysis	
Map Calculation	>
Combination Map	
Print-Out	
Calibration Factor	
Reset Map	
Element Ratio Map	
Particle Measurement	
Contour/Bird's Eye View	

Operation menu for Map Analysis

4. Clicking on Map Display opens the Map Display menu.

When you select any item from the Map Display menu, the window corresponding to the selected item appears together with the Selection Map window.

a. In the Selection Map window, specify the map to be operated on and maps to be processed.

If the **All** button is off, only the map to be operated on is processed. If the **All** button is on, all displayed maps are processed.

- **b.** Clicking on **Scale** in the Map Display menu opens the Scale window that allows a part of the map to be enlarged.
 - Click on the **Center** button in the Scale window; then click on the **Magnify** button and input a magnification to the current map. After you click on the center of the area to be enlarged, the specified area can be enlarged by pressing the **Apply** button.
 - Click on the **Outline** button; then diagonally drag a corner of the rectangle to be enlarged to specify the area. The specified area can be enlarged pressing the **Apply** button.
- **c. Single Map Display** in the Map Display menu allows the map specified in the Selection Map window to be enlarged to fill the map area.
- **d.** Clicking on **Level Modify** displays the Level Modify window where the histogram relating the number of pixels to X-ray intensities for the map specified in the Selection Map window is shown and the color levels of the map can be changed.
 - Clicking on the **Equal Width** button divides the difference between Upper and Lower into equal widths. The color levels of the map are controlled by changing the values of Upper and Lower. Finally, the **Apply** button puts this into effect.
 - If the **Equal Area** button is clicked on, the difference between Upper and Lower is divided so that the areas of all levels are almost equal.
 - If the **Arbitrary Level** button is clicked on, the values for each level can be input arbitrarily.
 - If the **Colors** button is clicked on, the levels can be adjusted finely on a color scale.
 - In the Calibration Factor, **A** and **B** values for converting X-ray intensities to concentrations can be input.

For details, refer to Step 8.

5. Clicking on Map Analysis opens the Map Analysis window as well as the Selection Map window.

- a. In the Selection Map window, a map to be operated on and maps to be processed are specified. The operation is the same as that of the Map Display.
- b. The 1 Point Analysis button in the Map Analysis window allows the X-ray intensity at the specified point on the map to be read out and displayed. It is displayed in the parameters area if the display area of the color bars is set to On Map. If not, it is displayed on the map.
- C. The 9 Point Analysis button allows the average X-ray intensity of nine points including the eight points surrounding the specified point to be displayed.
- **d.** The **Distance** button allows the distance between two points on the map to be displayed. The two points are specified by clicking on a point and dragging the pointer to the other point on the map. The display area is the same as that in the 1 Point Analysis.
- e. The Line Profile (Hor) button allows the line profile along a horizontal line specified on the map to be displayed.
 - The horizontal line is specified by double-clicking on any point on the desired line on the map.
 - If the pointer is dragged in the vertical direction from the specified horizontal line, the average values of data between the horizontal line through the start point and the horizontal line through the end point are displayed.
 - If the **On Map** button is clicked on, the line profile for each element is displayed on the respective map. If the **Result Map** [n] ("n" is a number) button is clicked on, the line profiles for all elements are displayed on map number n.
 - The full-scale values of the profiles are the maximum values of the profile data or values input using the **Max Values** button.
- f. The Line Profile (Ver) button allows the line profile along a vertical line specified on the map to be displayed. The operations are the same as those of Step 5.e.
- **g.** The Line Profile (Arb) button allows the line profile along an arbitrary line on the map to be displayed. The line is specified by clicking on a point and dragging the pointer to another point on the map. If the pointer is dragged after a line has been specified, the average values of data between the line through the start point and the line through the end point are displayed.

6. Clicking on **Map Calculation** opens the Map Calculation menu.

When you select any item from the Map Calculation menu, the window corresponding to the selected item appears together with the Selection Map window.

a. In the Selection Map window, specify the map to be operated on and maps to be processed.

 \swarrow The operation is the same as that of **Map Display** (S Step 4.a).

- b. The X-ray counting losses due to the dead time of the X-ray detector and counting circuits can be corrected for by selecting Dead-time Correction in the Map Calculation menu.
- **c.** Filtering in the Map Calculation menu lets you perform the operations for image processing like Smoothing, Sharpening or Edge Enhancement.
- **d.** Map Math in the Map Calculation menu lets you do calculations like adding, subtracting, multiplying or dividing by a constant K for one set of map data, or calculations between two sets of map data.

- 7. Clicking on **Print-Out** opens the Print-Out window where you can select data and output it to the printer. The **Map Intensity (Concentration)** button lets you output the map data in the pixels specified by **From:** and **To:**.
- 8. Clicking on Calibration Factor opens the Calibration Factor window where calibration factors A and B which are used for converting the X-ray intensities of map data to concentrations are input or changed. The calibration factors A and B are obtained by measuring X-ray intensities of known specimens, fitting a linear function between the X-ray intensity and the concentration, and calculating the slope A and the intercept B.
 - Slope A: X-ray intensity per 1% of concentration and 1 μ A of probe current (count/(ms· μ A·%))
 - Intercept B: Background intensity per μA of probe current (count/(ms· μA))

Clicking on the **Default** button opens a window listing existing standards and values of A and B calculated from the standard measurement data. Select an appropriate standard. When no standard is listed, select **CAL-STD**.

16 BACKING UP MEASURED DATA AND REPROCESSING STORED DATA

16.1 Backing up and Reprocessing Stored Data

The measured data are automatically stored on the hard disk. The JXA-8100 and 8200 have hard disks with a capacity of 4 GB, 9 GB or more depending on the system and about 3 GB or more is available for measured data. But as the disk fills up over a long period of time, it becomes necessary to arrange the data on the disk or to copy them to an MO disk.

Rearranging and copying of the data can be carried out by selecting **File Utility** from the Utility menu.

16.2 Checking the Space Available on the Hard Disk

You can check the amount of space available on the hard disk in the following way.

- Double-clicking on the Console icon on the computer display opens the window where OS commands are accepted.
- **2.** Input >bdf and the status of the file system is displayed.

kbytes	used	avail	capacity	Mounted on
Ø	Ø	Ø	0%	/proc
246463	21613	200204	10%	Ţ.
1015542	739141	215469	78%	/usr
Ø	Ø	Ø	0%	/dev/fd
246463	34491	187326	16%	/var
6708669	538302	6103281	9%	/export/home
1015542	252584	702026	27%	/opt
644936	504	644432	1%	/tmp
1086222	326680	705231	32%	/mo
	kbytes 0 246463 1015542 0 246463 6708669 1015542 644936 1086222	$\begin{array}{cccc} kbytes & used \\ 0 & 0 \\ 246463 & 21613 \\ 1015542 & 739141 \\ 0 & 0 \\ 246463 & 34491 \\ 6708669 & 538302 \\ 1015542 & 252584 \\ 644936 & 504 \\ 1086222 & 326680 \\ \end{array}$	kbytes used avail 0 0 0 246463 21613 200204 1015542 739141 215469 0 0 0 246463 34491 187326 6708669 538302 6103281 1015542 252584 702026 644936 504 644432 1086222 326680 705231	kbytes used avail capacity 0 0 0 0% 246463 21613 200204 10% 1015542 739141 215469 78% 0 0 0 0% 246463 34491 187326 16% 6708669 538302 6103281 9% 1015542 252584 702026 27% 644936 504 644432 1% 1086222 326680 705231 32%

This is an example of the file system of the JXA-8100 with the magneto-optical (MO) disk mounted. The /export/home entry shows the user directory and /mo shows the MO disk. The file system information is also displayed when the MO disk is mounted.

3. Click on the minimize button to close the window.

16.3 Inserting and Removing the MO Disk (Media)

- **1.** Inserting the disk
 - a. Insert the disk into the drive unit.
- b. Click on File Utility on the Utility menu and select Media Access. Then after selecting Initialize Media, click on the Mount button in the Initialize Media window.
- 2. Removing the disk
- **a.** Confirm that data are not being written on or read from the disk; then click on the **Unmount** button in the Initialize Media window.
- **b.** Push the eject button on the drive unit and the disk is ejected.

If the Unmount button in the Initialize Media window is not clicked on, the disk is not ejected. Be sure to click on the Unmount button.

16.4 Recording onto the MO Disk (Media)

Clicking on **File Utility** in the Utility menu opens the window showing the **Media Access** and the **Data Arrange** buttons. In **Media Access**, data are copied from the hard disk to the MO disk or from the MO disk to the hard disk. In **Data Arrange**, the data are copied on or deleted from the hard disk.

16.4.1 Media Access

Clicking on the **Media Access** button displays the menu having the items **Initialize Media**, **Backup** and **Restore**.

- 1. Clicking on **Initialize Media** opens the window where a disk is specified, it is initialized if it is new, it is mounted on the system and it is unmounted.
- 2. Clicking on **Backup** opens the Backup window where the data are copied to the disk.
 - a. Click on a media-selection button to select a medium.
 - b. Select All to copy all data or Specify to copy only specified data.
 - C. If you selected Specify, you can specify the group and sample names and the type of data. The group name and sample name can be specified after selecting All or Specify in the Select Group and Sample window. Here, the New Group and New Sample buttons do not mean anything.
 - d. Select the type of analysis for Measured Data and Measurement Conditions.
 - e. Select WDS or EDS for Standard Data.
 - f. Click on the **Backup** button and the copying is executed.
- **3.** Clicking on **Restore** opens the Restore window where the data are restored from the disk.
- a. The procedure is almost the same as that for **Backup** (Step 2).
- **b.** If you select **Specify**, after clicking on the **Listing** button, the list of the group and sample names of the data stored on the disk is displayed. The group name and sample name for restoring can be specified there.
- c. Click on the **Restore** button and the restoring is executed.

16.4.2 Data Arrange

Clicking on the **Data Arrange** button displays the menu having the items **Measured Data**, **Standard Data** and **Measurement Conditions**. Here, the data on the hard disk are copied, moved or deleted. Copying makes another data file having the same contents, but moving deletes the original data file after copying it.

- Clicking on Measured Data opens the Measured Data Arrange window where you can copy, move or delete the measured data after specifying the group and sample names and the analysis points for Source and Destination.
- a. The procedure is almost the same as those of the Backup. But here New Group and New Sample can be specified for Destination.

- **b.** The analysis points are specified in the Select Number window that is opened using the **No** button. The data at the specified analysis points can be copied only to the same group and sample names.
- **c.** To display a list of the group and sample names and the types of the analysis, click on the **Listing** button.
- d. Click on the Copy, Move or Delete buttons depending on what you want to do.
- 2. Clicking on Standard Data opens the Standard Data Arrange window where you can copy, move or delete the WDS and EDS standard data after you specify the sample names for the **Source** and the **Destination**.
- **3.** Measurement Conditions is mainly used for deleting the measurement conditions data of the specified type of analysis.

16.5 Change Directory

Selecting **Change Directory** from the Initialize menu opens the window in which you can change the directory. If the current directory (usually /export/home/jx1) is changed to the MO disk (/mo), the data stored on it can be read and processed.

16.6 How to Back up Measured Data and Reprocess Stored Data

- **1.** Backing up measured data
- a. Check the space available on the hard disk (Section 16.2) and estimate the space necessary for the backup. For example, a color map acquired with 400 pixels × 400 pixels uses 320 KB per chemical element. Saved in compressed format, the data usually occupy less space.
- **b.** Insert an MO disk in the drive (@ Section 16.3).
- **c.** Initialize the MO disk if it is new (@ Section 16.4.1).
- **d.** To append data to the MO disk, click on the **Listing** button in the Restore window and check the data stored on the MO disk.
- e. If the same group name and sample name as those of the data to be backed up are found, move the data to a different group name or sample name using Data Arrange before backing up (Section 16.4.2).
- f. Click on the **Backup** button to execute the backup (@ Section 16.4.1).
- g. Remove the MO disk after finishing the backup (@ Section 16.3).
- 2. How to reprocess stored data
 - a. Insert an MO disk in the drive, and mount it (@ Section 16.3).
- **b.** Click on **Change Directory** in the Initialize menu.
- C. Input /mo in New directory input box using the keyboard or specify /mo after clicking on the List button. Then click on the OK button.
- **d.** Operate the data-processing programs in the Process menu to display and analyze the data on the MO disk.
- e. To return to the original directory after finishing the data processing, input /export/home/jx1 if the login code is jx1 in the New directory input box in the Change Directory window and click on the OK button.
- f. Unmount the MO disk and remove it.
17 BRIEF DESCRIPTION OF JXA-8100/8200 OPERATION

SHUT DOWN

Halted

1. Turn ACCEL VOLTAGE off.

4. Turn the valves of gases off.

3. Turn Main POWER (Key Switch) off.

5. Turn the water off after about 3-5 minutes.

2. Click "SYSTEM SHUT DOWN" in Initialize menu.

START UP

- 1. Turn the water on.
- 2. Turn the valves of nitrogen gas and P-10 gas on.
- 3. Turn Main POWER (Key Switch) on.
- 4. Turn OPE POWER on.
- 5. Turn computer system POWER on.
- 6. Log in.

EPMA Menu start HV Ready

Possible to operate

PREPARATIONS

1. ELECTRON COLUMN ALIGNMENT

Turn ACCEL VOLTAGE on. Set or adjust the following in Monitor menu.

> Monitor Stage EOS Monitor Filament GUN Tilt GUN Shift

15-20 kV

Change specimen and set position.

Adjust filament heating current to get the saturation point.

Adjust to get the maximum probe current.

Adjust the feature image (SEI) to be stationary when probe current is changed in order of $10^{-9}-10^{-7}$ A.

WOBB

Adjust the centering of the OL aperture to minimize the shift of the feature image. (This can be done even just after GUN tilt adjustment.)

2. CONFIRMATON OF SEM IMAGE

Confirm the sharpness or the steadiness of the SEM image with a suitable specimen before analyzing of unknown specimens.

OBSERVATION OF SEM IMAGE

1. Set the specimen on the stage and focus the OMTV image on the specimen.

2. Set the following

IMAGE SELECTOR	SEI
SCAN SPEED	1
PRB SCAN	ON
PCD	OUT
All of Contract Deislateres	(It is seen to much

3. Adjust Contrast, Brightness. (It is easy to push the ACB button on operation panel.)

4. Increase the magnification of the SEM image and focus the image.

 Decrease the probe current to the order of 10⁻¹¹-10⁻¹⁰ A and select OL aperture acording to the recommended number indicated on the Control window. (If necessary, adjust Contrast, Brightness.)

6. Push the WOBB button and adjust the centering of the OL aperture to minimize the shift of the feature image.

7. Focus the image with STIGMATOR X, Y and FOCUS.

QUALITATIVE ANALYSIS





QUANTITATIVE ANALYSIS

1. Standard Analysis



2. Quantitative Analysis



COLOR MAP ANALYSIS



