XM-17330/27330

STANDARD SAMPLE ANALYSIS PROGRAM

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

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NOTATIONAL CONVENTIONS AND GLOSSARY

General notations

	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, 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 qui

uble-click:	To press and release the left mouse button twice quickly.
ubic click.	to press and release the tert mouse button twice quickly.

	Drag:	To hold down the left mouse button while moving the mouse
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1 GENERAL

This program is a tool for executing standard sample analysis using EPMA, a process necessary for quantitative analysis. Usually, accurate quantitative analysis requires that standard samples of a known concentration be measured in advance.

The function of this program is not restricted by any special correction method. That is, the data measured with this program are applicable directly to a variety of correction methods to be used for quantitative analysis, such as the ZAF method, optional (Z) method, optional B & A method, and optional thin-film correction method. In addition, this program is designed to extend its application to the calibration-curve method, WDS/EDS combined analysis method, and others. Another consideration incorporated in the program is data compatibility with other programs including qualitative analysis, quantitative analysis.

Even beginners will find this program easy to understand, yet it provides capabilities ranging from basic operations to advanced levels of standard-sample analyses that are common in quantitative analysis.

2 SPECIFICATIONS

Measurable elements in standard samples:

Measuring method:

WDS spectrometer X-ray counting method: WDS spectrometer X-ray measurement time: EDS spectrometer X-ray counting method: EDS spectrometer X-ray measurement time: Number of accumulations per measurement point:

Peak-search realtime display during measurement: Type of standard samples: Up to 30 elements (including up to 20 elements for EDS measurement) Asynchronous concurrent drive for each X-ray spectrometer Fixed time or fixed count 0.1 to 1,000 s Fixed time (live time or real time) 1 to 10,000 s 1 to 100, selectable from values of accumulation after measurement Possible Metal or oxide

3 MENU STRUCTURE



* Only for JXA-8200.

4 OPERATION

4.1 Measurement

The general procedure for measurement is as follows.

After selecting a standard sample name, enter its chemical composition, element to be measured, conditions of the electron optical system and the coordinate position. Do this once for each standard sample. Then, perform measurements by using **Preset**. You can carry out measurements one by one by using **One-by-One** in place of **Preset**. If once you measured standard samples, usually it is not necessary to perform measurements any more.

4.1.1 Starting and terminating Standard Sample Analysis Program

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

The Analysis menu opens.

- **Refer to the instruction manual of the microanalyzer main unit to learn how to open the EPMA Main Menu.**
- 2. Select Standard Analysis from the Analysis menu.

The Standard Analysis function window opens.



Fig. 1 Standard Analysis function window

3. To terminate this program, click on the Exit button.

4.1.2 Selecting a sample name

You can name each standard sample using up to 14 characters. The measured data of standard samples are stored in element name, measured X-rays, order, accelerating voltage, channel and analyzing crystal separately. Consequently, if data are different, you can store the data using the same standard sample name.

Furthermore, if you group multiple standard samples and name the group, you can store them separately from other groups. In this case, standard sample groups can share composition and coordinate positions.

To save a sample name, display the Select Standard Sample window, click on the New button, and then enter the sample name.

Then, carry out the following procedure.

- Standard Analysis Select Standard Sample Sorting Order Group A11 🔷 Name 🐟 Date Select Name Si_metal NDS Name EDS No. Date A1203 Sep-12-2000 × 1 Ni_calib Sep-12-2000 2 Sep-12-2000 3 Cr_metal 4 Si_metal Sep-12-2000 Dy Sep-12-2000 5 6 **TiO2** Sep-11-2000 7 Ho Aug-31-2000 в Er Aug-31-2000 9 Vb Aug-31-2000 -10 Aug-31-2000 ы Total 57 samples 2264460 Kbyte used. 1744564 Kbyte free. Search Print Rename Copy Delete Cancel OK New
- Click on the Sample button in the Standard Analysis function window. The Select Standard Sample window opens.

Fig. 2 Select Standard Sample window

In this window, a list of standard sample names recorded in a group, measurement dates, and indications of whether WDS or EDS analysis methods have been applied will be displayed.

If **All** is selected as **Group**, all the standard samples in all the groups will be listed. Also, the hard disk space already used and the free space are displayed in kilobytes. This window has the following functions.

Button	Function
New	After clicking on the New button, you can enter a new standard sample name. The maximum length is 14 characters. You can use alphanumerics, +, -, _, =, and . (the period cannot be the first character).
Sorting Order	Clicking on the Name button in Sorting Order in the window rearranges the display of standard sample names in alphabetical order. Clicking on the Date button in Sorting Order rearranges it in chronological order.
Search	Clicking on this button opens the Standard Search window as shown in Fig. 3. Enter element name in the Element input box; then the list of standard samples containing that element name will be displayed. Click on any one of the buttons in the list, and then its standard sample will be selected.

- Standard Analysis						
Elemen	t si	Total	18 data			
No.	Standard	Acc.V	сн сгуз	cps/uA		
1 0	CaSiO3	15 Ka	2 LIF	105441		
2 💠	CaSiO3	15 Ka	3 EDS	6075		
3 🗇 (CaSiO3	15 Ka	3 LIFH	185330		
4 🗇 (CaSiO3	15 Ka	4 PETH	156306		
5 🗇 (CaSiO3	15 Ka	5 NSTE	455874		
6 💠 :	5102	15 Ka	2 LIF	212945		
7 💠 :	\$102	15 Ka	3 EDS	11758		
8 💠 :	\$i02	15 Ka	3 LIFH	377793		
9 💠 :	5i02	15 Ka	4 PETH	315000		
10 💠 :	si02	15 Ka	5 NSTE	931812		
ок	Cancel					

Fig. 3 Standard Search window

Button	Function		
Print	Click on this button to print the list in the Select Standard Sample window.		
Rename	After clicking on this button, you can enter new names for the standard samples.		
Delete	If you entered a standard sample name by mistake using the New button, click on the Delete button. To delete standard samples that have already measured data, be sure to select Utility-File Utility from the EPMA Main Menu.		

2. Click on the **Group** button at the top left of the Select Standard Sample window.

The Select Group window opens as shown in Fig. 4.

- Standard Analysis					
Select Group					
Sorting Order EOS Condition 20.0 kV Name * Date Use the same EOS Condition Group Name : Hetal Comment : JEOL Standards					
Group All Area Ketal	Sample Cr_metal Si_metal Ti_metal Ni_metal B_DUDDY Ho_metal Fe_metal				
OK New	Edit Delete Cancel				

Fig. 4 Select Group window

This window displays stored group names and standard sample names belonging to the selected group.

The All entry in the Group column is a reserved group name that you can select when you do not want to use the Group function.

The Select Group window also has the following functions.

Button	Function		
EOS Condition	Allows you to set conditions of the electron optical system (EOS) specific to the selected group.		
Use the same EOS Condition	If this button is selected, all of the EOS conditions set by using the above button will be applied to all the standard samples.		
List Intensity	Displays the measured-data list of the standard samples that belong to the selected group as shown in the example in Fig. 5.		

Hano	Elon Acc.V	Хгау СН	Crystal	Current	Peak	Not	Date
Cr_metal	Cr 20.0	Ka 1	LIF	9.9810E-09	159.126	3386.3	Jul 23
Cr_motal	Cr 20.0	Ка 3	LIF	9.9810E-09	159.531	2662.6	Jul 23
Cr_metal	Cr 20.0	Ka 5	LIFH	9.9810E-09	159.211	9534.0	Jul 23
Si_metal	S1 20.0	Ка 2	TAP	9.9500E-09	76.965	29636.5	Jul 23
Si_metal	Si 20.0	Ка 3	PET	9.9500E-09	228.385	2943.1	Jul 23
Si_metal	\$1 20.0	Ka d	PETH	9.9500E-09	227.364	7612.8	Jul 23
Ni_metal	Ni 20.0	K.a 1	LIF	9.9950E-09	114.736	3464.5	Jul 23
Ni_metal	N1 20.0	Ka 3	LIF	9.9950E-09	115.582	5236.9	Jul 23
Ni_metal	Mi 20.0	Ка 5	LIFH	9.99502-09	115.001	14192.3	Jul 23
B_DUMNY	B 15.0	Ka 2	LDE2	2.0270E-06	171.938	27858.1	Jul 25
Ho_metal	Mo 20.0	La 3	PET	9.8630E-09	173.408	1375.4	Jul 23
Ho_metal	Mo 20.0	La 4	PETH	9.8630E-09	172.469	3981.0	Jul 23
Fe_metal	Fe 20.0	Ка 1	LIF	1.0040E-08	134.327	3609.6	Jul 23
Fe_metal	Fe 20.0	Ka 3	LIF	1.0040E-08	134.961	4244.3	Jul 23
Fe_metal	Fe 20.0	Ка 5	LIFH	1.0040E-08	134.513	13115.0	Jul 23

Fig. 5 Example of List Intensity Data

3. When you want to create a new group, click on the **New** button, or when you want to edit standard sample names belonging to the selected group, click on the **Edit** button.

The Edit Group window opens as shown in Fig. 6.

- Standard Analysis					
Edit Gr	oup				
Group Name Metal Sorting Order					
Comment JEOL Standards					
Installed standards Import from Qnt Cr_metal Si_metal Ti_metal B_DUN9Y Ho_metal Fe_metal	Standard list Search Al203 Ni_calib Cr_metal Si_metal Dy Ti02 Ho Er Yb W				
Total + 7	Total : 57				
Select : 0	Select : 0				
OK Clear A	11 Cancel				

Fig. 6 Edit Group Window

In the Edit Group window, you can enter a group name of up to 14 characters and a comment of up to 40 characters. In the left column of this window, the standard sample names belonging to the specified group name are displayed, while in the right column, all of the stored standard sample names are displayed.

- To store a standard sample name in the left column, select the standard sample name in the right column, and then click on the left arrow ←.
- To delete a sample name from the left column, select the standard sample name in the left column, and then click on the right arrow →.

The Edit Group window has also the following functions.

Button	Function
Import from Qnt	Selects a standard sample for the selected group, from those which are presently stored in Standard list for the quantitative analysis.
Search	Allows you to enter elements in the Search column and displays the list of stored standard samples.
Deselect	Deselects the selected samples.
Clear All	Clears all of the stored standard samples.

4.1.3 Entering measurement conditions

Click on the Measurement button in the Standard Analysis function window.
 The Measurement menu opens as shown in Fig. 7.

Click on any desired item to enter measurement conditions before starting the measurement.

Standard Type (Metal)
Element Condition (0 Al 0)
EOS Condition (Acc. 15.0 kV)
EDS Condition
Stage Condition
Condition Load
Condition Store
Check Data
Print-out Condition (OFF)
Measurement Mode (HDS)
Additional Function (OFF)
Survey Measurement
Preset Measurement (1)

Fig. 7 Measurement menu

Standard Type

This function allows you to specify the type of samples to be analyzed.

 Click on Standard Type in the Measurement menu of Fig. 7. The Standard Type window opens as shown in Fig. 8.



Fig. 8 Standard Type window

- Click on Metal if the sample to be measured is metal (non-oxide), or click on Oxide if it is oxide.
- K If you want to perform measurement by using the calibration-curve quantitative analysis program, click on the **Calibration Curve** button in the Standard Type window. In calibration-curve quantitative analysis, after you have obtained calibration curves from standard samples, you convert the X-ray intensities to the corresponding concentration in the unknown sample measurement. Details will be described later.

Element Condition

The Element Condition function allows you to enter the chemical composition of a standard sample to set elements to be measured and measurement conditions.

Click on Element Condition in the Measurement menu of Fig. 7.

The Element Condition window opens as shown in Fig. 9.

-			St	andar	d Analy	sis		
		1	Elem	ent (Conditi	on		
Standard	Composition	1	2 E)	lener	its			
NDS	Element Neas. order Condition	La	La	La	F La	i F		
EDS	Element Condition	La	La					
INS	Signal							
Close					Total	Acquisition	8	Elements

Fig. 9 Element Condition window

1. Click on the **Standard Composition** button in the Element Condition window.

The Standard Composition window opens as shown in Fig. 10.

Here, you perform the following Steps a to c to enter chemical compositions.

After you have performed this operation, you need not do it again in subsequent measurements.

- If you want to change the composition of a standard sample, measure the standard sample. By simply changing the values in the Standard Composition window the composition of the standard sample cannot be applied to correction calculations.
- In the ordinary standard-sample analysis, you enter the chemical composition of a standard sample. However, if you specify the calibration-curve mode, you can enter different chemical compositions. Details will be described later.



Fig. 10 Standard Composition window

a. If the standard sample is metal, click on the Atom button (for atomic proportion) or Mass button (for mass concentration).
 If the standard sample is oxide, click on the Atom button, Mass button or

If the standard sample is oxide, click on the **Atom** button, **Mass** button or **Mole** button (for mole ratio of oxides).

- b. Enter element names in the Elements (Elem.) input boxes using the keyboard. In the Value input boxes at the right of the Elem. input boxes, key in the values corresponding to the selected button in Step a. (Fig. 10.)
 - Solution If an oxide is specified, the valence input boxes are also displayed; you can enter the valences there.)
 - The numerical entry for mass concentration may be a percentage or a fraction (ratios add up to 1).
- C. To determine the direction in which the cursor will move, click on the Vertical button or Horizontal button.

Selecting **Vertical** moves the cursor vertically, while **Horizontal** moves it horizontally.

Entry example

As an example, to enter the composition of $CaSiO_3$ as an oxide, make the following entries for each mode.

• Mass mode

Elem.	Val.	Value
Ca	2	48.272
Si	4	51.728

• Atom mode

Elem.	Val.	Value
Ca	2	1.000
Si	4	1.000
0	-2	3.000

- In the Atom mode, there is no need to actually enter the valence. However, be sure to enter the valence for oxygen in the case of an oxide.
- Mole mode

Elem.	Val.	Value
Ca	2	1.000
Si	4	1.000
🖌 T., 4h		

- \swarrow In the **Mole** mode, enter CaO: SiO2 = 1:1.
- Click on the WDS-Element button in the Element Condition window if you want to carry out measurement by using wavelength dispersive spectrometer (WDS).

The WDS Elements window will open as shown in Fig. 11, allowing you to set elements to be measured as described below.

MDS Elements
Standard Composition
La F
Select Elements
La La F La F
OK Clear Cancel
UN UNU CARGE

Fig. 11 WDS Elements window

• Adding element names

The element names entered in the Standard Composition window are displayed under **Standard Composition**.

In the window of Fig. 11, click on the button of the desired element name in the **Standard Composition** area. If you click on a single element twice or more, the element will be measured twice or more in the same sequence. A different spectrometer can be specified for each element measurement sequence, so that the desired elements can be efficiently measured using the multiple spectrometers installed in the basic unit. This feature helps reduce the time for quantitative analysis that spans two or more sequences.

Deleting unnecessary element names

The list of element names is shown under **Select Elements** in the WDS Elements window of Fig. 11.

To delete an unnecessary element under **Select Elements**, highlight it by clicking on it, and then click on the **Clear** button. To delete two or more elements at one time, position the mouse pointer on one of the desired elements and drag it until all the elements you want to delete are highlighted; then release the mouse button and click on the **Clear** button.

 When you want to use the EDS spectrometer in the JXA-8200 series for measurement, click on the EDS-Element button in the Element Condition window.

The EDS Elements window appears as shown in Fig. 12, allowing you to set elements to be measured in this window.

- Standard Analysis				
EDS Elements				
Standard Composition				
La P				
Select Elements				
La La				
OK Clear Cancel				

Fig. 12 EDS Elements window

The steps for adding and deleting elements are the same as for WDS. However, in EDS, K-line and L-line, or L-line and M-line are sometimes observed on an EDS spectrum at the same time. In such a case, specify an element twice, and then measure each X-ray that was generated from the same element.

In the correction calculation of an unknown sample, if you specify the X-rays from the first measurement of an element, then the second measurement of the same element will be executed automatically, and it will be used for peak separation calculation. Click on the WDS–Meas. order button in the Element Condition window. The Measurement order window appears as shown in Fig. 13, allowing you to set channels to be used.

		Standard Analy Heasurement or	rsis der	
CH-1	CH-2	CH-3	CH-4	СН-5
	La (PETJ)	La (LIFH)	La (LIFR)	F (TAP)
	La(LIP)			F (TAP)
Cancel			Print ind	crystal -
	CH-1	CH-1 CH-2 La(PETJ) La(LIF) Cancel	Cancel	Standard Analysis Heasurement order CH-1 CH-2 CH-3 CH-4 La(PETJ) La(LIFH) La(LIFH) La(LIF) La(LIFH) La(LIFH) La(LIFH) Cancel Print ind

Fig. 13 Measurement order window

If you make the selection so that measurements are not concentrated in any one specific channel, you can shorten the overall measurement time.

Drag an element label using the mouse to set the measurement order. Dragging up and down changes the measurement order and dragging left and right changes the channel number. If the measured X-rays are out of range of spectrometric analysis, the mouse cursor turns to a cross and you cannot select the channel for analyzing the X-rays. If you want to change the X-rays to be used, do so by using WDS-Condition in the Element Condition window.

If the same analyzing crystal is used for the same element in the same spectrometer channel, the data of the first measurement will be overwritten with that of the second measurement.

5. Select **WDS–Condition** from the Element Condition window. **The WDS Element Condition window opens.**

MDS Element Condition					
No.of Elements 6 Tos. (nm) \Diamond Have. (A) \Diamond Have. (nm)					
	Elem- 1	Elem- 2	Elem- 3	Elem- 4	Elem- 5
Elements	La La	La La	La La	F	La La
X-ray Name Order	La 1	La 1	La 1	Ka 1	Lа 1
Channel Crystal	2 PETJ	3 LIFH	4 LIFH	TAP	LIF
Spect. Pos.(mm) Back (+) (mm)	85.424 5.000	185.427	185.674	87.310	185.397
Back (-) (mm) Time/Count	Time 1	Time 1	Time	Time	Time
Peak Seek W. Hes. Time (sec)	10.0	10.0	10.0	10.0	10.0
Bac. Time (sec) Mes. Count	10000	10000 500	10000 500	10000	10000 500
Bac. Count PHA gain	64 1700	32 1700	32 1700	32 1700	32 1700
High V.(V) Base L.(V)	0.70	0.70	0.70	1.00	0.70
Diff/Int	1 Int	Int 1	Int 1	Int 1	Int 2
sequence.	<u>م</u>				2
Peak overlap				Move	
	OK	J		Cance1	

Fig. 14 WDS Element Condition window

The measurement conditions for elements that are measured by WDS are displayed in the list in the WDS Element Condition window.

6. To modify the measurement conditions, select Elem-1, 2, 3 ...

The WDS Element Data Table window will be displayed as shown in Fig. 15. You can modify the measurement conditions that appear in the window.

	MDS Element Data Table					
Element La						
Select No.	1	2	3	4	5	j –
Hamo	La	La	La	La	La	
X-ray Name	La	Ьа	La	La	Ha	
Order	1	1	1	1	1	
Channel	2	3	4	2	5	
Crystal	LIF	PETH	LIFH	PETJ	NSTE	
Spect.Pos.(mm)	85.440	185.435	185.669	185.393	162.089	
Back(+)(mn)	3.000	5.000	5.000	5.000	5.000	
Back (-) (mn)	3.000	5.000	5.000	5.000	5.000	
Pune/Count/Area	т	T	т	т	T	
Nor Time(reg)	1	1	0	1	1	
Bac Time(sec)	10.0	10.0	10.0	10.0	10.0	
Hes. Count	5.0	5.0	5.0	5.0	5.0	
Bac. Count	10000	10000	10000	10000	10000	
PHA Gain	500	500	500	500	500	
High V.(V)	64	32	32	32	32	
Base L.(V)	1700	1700	1700	1700	1700	
Window(V)	0.70	0.70	0.70	0.70	1.00	
Diff/Int	9.30	9.30	9.30	9.30	9.00	
	Int	Int	Int	Int	Int	
			1.	. (
	New Copy Exchange Delete					
	Set Read					
	ок		c	ancel		

Fig. 15 WDS Element Data Table window

By using the WDS Element Data Table, change the element conditions as follows. When you want to set a new element condition, click on the New button. A blank line is created for the new conditions. The element table can have up to twenty sets of element conditions for each element. You can change the element conditions of the elements to be measured by clicking on **Select No**. In this window, the following operations are possible.

Button	Function
Select No.	Select the element conditions of the elements to be measured.
New	Creates a new set of element conditions. The element table can have up to twenty sets of element conditions for each element.
Сору	Copies the element conditions of the elements to be measured to the element conditions of the specified number.
Exchange	Exchanges the element conditions of the elements to be measured with the element conditions of the specified number.
Delete	Deletes the element conditions of the elements to be measured. At this time, the element table is left-justified.
Set	Of the element conditions of the elements to be measured, the following conditions of the EPMA basic unit are set: Crystal (analyzing crystal), Spect. Pos. (spectrometer position), and SCA conditions (PHA gain , High V. , Base L. , Window , Diff/Int).
Read	 The following conditions mentioned above are loaded from the EPMA basic unit: Crystal (analyzing crystal), Spect. Pos. (spectrometer position), and SCA conditions (PHA gain, High V., Base L., Window, Diff/Int). Use this button when you want to change the element table to reflect the element conditions after you check these conditions on the monitor screen. Sefore executing Read, it is convenient to align the stage to the specimen position using the Stage Monitor on the monitor screen, to check the peak position using the Peak Search, and to adjust the Content of the specimen position using the Peak Search and to adjust the the specimen position using the Peak Search and to adjust the the specimen position using the Peak Search and to adjust the the specimen position using the Peak Search and to adjust the the specimen position using the Peak Search and to adjust the the peak position using the Peak Search and to adjust the the specimen position using the Peak Search and to adjust the the peak position using the Peak Search and to adjust the the peak position using the Peak Search and to adjust the the peak position using the Peak Search and to adjust the the peak position using the Peak Search and to adjust the the peak peak peak peak peak peak peak pea
OK	Finalizes the element conditions and changes the WDS Element Condition to reflect the element conditions of Select No.
Cancel	Erases the element conditions.

The items that you can change in the WDS Element Data Table are the following.

Button	Function	
Name	Assign any name by inputting up to 8 characters. Element name is shown as the default.	
X-ray Name	Select an X-ray name from Ka, Kb, La, Lb, Ma, and Mb, where "a" and "b" mean " " and " " respectively.	
Order	Select the order of X-rays from primary to tenth.	
Channel	Select a spectrometer channel. If the X-rays to be measured are out of the spectroscopic range of the channel, the channel number is dimmed.	
Crystal	Select the name of the crystal that you want to use from the list of crystals loaded in the spectrometer channel. Crystal names that you cannot select are dimmed.	
	As a special case, after you change the analyzing crystals while measurement is in progress, sometimes you cannot choose the desired analyzing crystals correctly; if this happens, then select again the correct X-rays by using X-ray Name.	

Button	Function
Spect. Pos.	Specify the position of the spectrometer. The default is the value calibrated to the theoretical position of the X-ray spectrometer. The position of the spectrometer actually measured after Peak Search will be displayed after sample measurement.
Back (+)	Allows you to specify the background offset on the higher angles. If you specify zero, the background measurement is not executed.
Back (-)	Allows you to specify the background offset on the lower angles. If you specify zero, the background measurement is not executed.
Time/Count/Area	Select the fixed-time (T) or fixed-count (C) measurement method. The fixed-time measurement is selected as the default. Selecting the fixed-time measurement performs the peak/background measurement during the specified time. Selecting the fixed-count measurement performs the measurement until you obtain the specified number of counts, but the measurement stops when the designated time is finished.
	Furthermore, from the window, you can select the area method, for which please refer to the separate instruction manual of the Quantitative Analysis Program.
Peak seek W	Select a peak search parameter from 0 to 4 . Usually, select 1 as the parameter. If you select 0 , the peak search will not be executed. The detailed peak search algorithm will be explained later.
Mes. Time	Specify the measurement time at the peak position in seconds.
Bac. Time	Specify the measurement time at the background in seconds. Usually, specify about half of the time of the measurement at the peak position.
Mes. Count	Specify the number of counts at the peak for the fixed-count measurement method.
Bac. Count	Specify the number of counts at the background for the fixed-count measurement method.
PHA gain	Select the gain of the SCA (Single Channel Analyzer).
High V.	Set the value of the high voltage of the SCA.
Base L.	Set the base level of the SCA.
Window	Set the width of the window of the SCA.
Diff/Int	 Select differential mode (Diff), or integral mode (Int) as the SCA mode. By default, the SCA parameters to which the hardware is set as the standard configuration will be read. To set the hardware more precisely, select the SCA parameters at the actual peak position on the SCA monitor, and then load them.

7. When you want to measure elements using the EDS spectrometer in the JXA-8200 Series, click on the **EDS–Condition** button in the Element Condition window.

The EDS Element Condition window appears as shown in Fig. 16. Check the measurement conditions and change them if necessary.

- Standard Analysis					
No.of Elements 2					
Elements Name X-ray Name No. of Regions Start1 (keV) End1 (keV) Start2 (keV) End2 (keV)	Elem- 1 La Ma 1 0.560 1.290 - -	Elem- 2 La La 1 3.760 6.430 -]		
	OK			Cancel	

Fig. 16 EDS Elements Condition window

8. To change the measurement conditions, click on the Elem–i button in the EDS Element Condition window.

The EDS Element Data Table window appears as shown in Fig. 17.

- Standard Analysis			
EDS Element Data Table			
Element La RO	1- 2		
Select No.	1	2	
Name	La	La	
X-ray Name	Ma	La	
No.of Regions	1	1	
Start 1 (keV)	0.560	3.760	
End 1 (kaU)	1.290	6.430	
End I (Kev)	-	-	
start 2 (kev)	-	-	
End 2 (keV)		<u></u>	
		New	Copy Exchange Delete
	Set		Read
	ОК		Cancel

Fig. 17 EDS Element Data Table window

In this window, set the Region of Interest (ROI) for each X-ray of an element to be measured. You can set up to two ROI's for each condition. By default, the range of each ROI includes the line and line. The minimum range of each ROI requires 14 channels.

When you observe K line and L line, or L line and M line on an EDS spectrum at the same time, store each X-ray separately, and measure it as two elements.

When you measure multiple elements in a standard sample of complicated composition at the same time, the ROI's overlap; then a warning message appears, and the overlapping ranges will be separated automatically. This is because the influence from another element should be avoided by all means when you create a reference profile of a standard sample. In the case of an unknown sample, overlapping ROI's will cause no problem since the peaks are fit by the least-squares method.

Button	Function
Select No.	Select the element conditions of the elements to be measured.
New	Creates a new set of element conditions. The element table can have up to twenty sets of element conditions for each element.
Сору	Copies the element conditions of the elements to be measured to the element conditions of the specified number.
Exchange	Exchanges the element conditions of the elements to be measured with the element conditions of the specified number.
Delete	Deletes the element conditions of the elements to be measured. At this time, the element table is left-justified.
Set	Sets the EDS to the present ROI conditions of elements.
	When you want to use Set , or Read , activate in advance the EDS Home Window.
Read	ROI information will be loaded from the EDS unit. For this purpose, set ROI in the EDS Home Window; then the ROI number will be that which is shown in Select No. That is to say, No. 1 corresponds to ROI No. 1, and No. 2 corresponds to ROI No. 2.
ОК	Finalizes the element conditions and changes the EDS Element Condition to reflect the element conditions of Select No.
Cancel	Erases the element conditions.

In this window, the following operations are possible.

The items that you can change in the EDS Element Data Table are the following.

Button	Function
Name	Assign any name by inputting up to 8 characters.
X-ray Name	Select an X-ray name from Ka, Kb, La, Lb, Ma, and Mb. By default, ROI No.1 will be set wide enough to include both the line and the line.
No. of Regions	Enter the number of ROI's to be set. Usually enter 1.
Start 1 (keV)	Specify the energy at the left end of the first ROI.
End 1 (keV)	Specify the energy at the right end of the first ROI.
Start 2 (keV)	Specify the energy at the left end of the second ROI.
End 2 (keV)	Specify the energy at the right end of the second ROI.

EOS Condition

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

 Select Measurement–EOS Condition from the Standard Analysis function window.

The EOS Condition window opens.

- Standard Analysis		
EOS Condition	n	
Set	Read	
Accelerating Voltage (kV) 20.0		
Current _ Auto 1.26 E- 8		
Magnification	3700	
Probe Diameter (um) 0		
Probe Scan ON		
► Scan Conditions		
► Lens Conditions		
OK Cancel		

Fig. 18 EOS Condition window

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

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

 \swarrow For quantitative analysis, you usually set the probe current in the range of 1 to 5 \times 10⁻⁸A. However, when you analyze standard samples using both WDS and EDS spectrometers in the JXA-8200 Series, the intensity of X-rays is too high for the EDS. So you need to adjust the aperture of the EDS detector to reduce the intensity of X-rays that enter the detector. When you analyze standard samples using the EDS only, open the aperture of the EDS detector fully and decrease the probe current to the range of 1 to 10 \times 10⁻¹⁰A. This minimizes, during analysis, any damage to the sample due to electron-beam irradiation.

EDS Condition

With the JXA-8200 Series EPMA, when you specify the EDS as the spectrometer, you need to set the measurement conditions for the EDS.

 Select Measurement–EOS Condition from the Standard Analysis function window.

The EDS Condition window opens as shown in Fig. 19.

- Standard Analysis	
EDS Condition	
Energy Full Scale (keV)	20
Spectrum Data Points	2048
Measuring Mode	Live
Measuring Time (sec)	100
Aperture No.	2
Set Re	ad
OK Can	cel

Fig. 19 EDS Condition window

The following items are for controlling the EDS detector.

Button	Function
Energy Full Scale	Is set to 20 kV.
Spectrum Data Points	Are set to 2 K channels.
Measuring Mode	Selects Live Time or Real Time as the measurement mode.
Measuring Time	Specifies the measuring time in seconds.

Clicking on the **Set** button sets the EDS to the spectrum collection conditions presently shown in the EDS Condition window.

Clicking on the **Read** button reads the present EDS spectrum collection conditions into the EDS Condition window.

Note that the **Set** and **Read** buttons can be used only when the EDS Home Window is on the screen.

When you have selected an aperture number, set the beam current, and then adjust the Dead Time indication to 20-30%, while actually collecting EDS spectra.

Stage Condition

The Stage Condition window allows you to specify the stage position of a standard sample before measurement. The procedure for specifying the stage position of standard samples is the same as that for ordinary point analysis in quantitative analysis. However, one of the characteristics of the standard-sample analysis is that the number of coordinate point is only one, since there is only one stage position of a standard sample. Multiple analysis points can be specified only in the calibration-curve analysis. Another characteristic is that accumulation is used for avoiding irregularity of standard samples, or for minimizing counting errors.

 Select Measurement–Stage Condition from the Standard Analysis function window.

The Stage Condition window opens as shown in Fig. 20.

To enter the stage position, click on the No. 1 line and specify the coordinate point by using **Pos. Input**.



Fig. 20 Stage Condition window

Stage Condition Input window

The Stage Condition Input window allows you to specify the analysis positions in the stage mode or beam mode.

Click on the Pos. Input button of the Stage Condition window. The Stage Condition Input window opens as shown in Fig. 21.



Fig. 21 Stage Condition Input window

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

Button	Function
Comment	Lets you input the comment on the standard sample up to 40 characters.
Scan Type	Specifies the stage control (Stage) or the beam control (Beam).
Auto Focus	If the optional auto focusing device is installed in the instrument, you can select the automatic focusing of the stage before measurement.
Magnification	Specifies the magnification of the EOS. This function is in effect when the beam control (Beam) is selected. To read the magnification, click on the Read button.
Accumulation	Specifies the number of accumulations (100 times maximum). Enter a number, and then the Method to set Accumulation window will open. Select Joystick (for arbitrary position), Line (linear), Grid (two-dimensional), or Fix (fixed coordinates). If you select Line or Grid , enter the number of steps and the scan width (in µm). After specifying Accumulation, confirm each coordinate by using the Confirm button to be explained later.



Fig. 22 Accumulation window

Button	Function
Stage Position	Displays the present recorded position of the stage.
Beam Position	Displays the present position of the beam (in effect onlyif Beam is selected).
Apply	Lets you enter the analysis point, and record it in the list of coordinates.
Confirm	After you have specified the number of accumulations, be sure to click on the Confirm button. Move to the accumulation point by using the Joystick Controller, and confirm the focusing; then press the STORE button of the Joystick. Repeat this operation once for each accumulation. If you click on the Cancel button before finishing this step, the remaining points of accumulation will be neglected, and the number of accumulations will be reset to the number of times that you clicked on the Confirm button.

- Standard Analysis Adjustment of Stage Adjust stage with Joystick and press Store Button !					
No	x	¥	z		
1	66.7577	37.6873	11.7075	P	
2	66.6577	37.5873	11.7075		
3	66.6577	37.6873	11.7075	μ	
4	66.6577	37.7873	11.7075		
5	66.7577	37.5873	11.7075	7	
OH Search Cancel					



Button	Function
Read & Apply	Reads the stage position, and also the beam position if necessary; then records them in the list of coordinates. This operation can be done also by using the STORE button of the Joystick Controller.
Close	Closes the Stage Condition Input window. If you have not yet finalized the change of the measurement position, the Confirmation window opens.
Large arrow buttons in up-and-down directions	Moves the coordinate position to the previous one or the following one. If you have not yet finalized the change of the coordinate position, the Confirmation window opens.
OM Search*	If the optional auto focusing device is installed in the instrument, you can perform the automatic focusing of the stage at the present stage position.
X, Y, Z	Displays the coordinates of the stage position. Click on the Read button, and then the present position of the stage will be displayed.
Store	Displays the values of X, Y, Z in the Stage Position indication box.
Read	Reads the present stage position and displays it in the X, Y, Z indication boxes.
Move	Moves the stage to the coordinates specified in X, Y, Z.
Range	Specifies the amount of movement (in mm) with the small arrow buttons by using the scroll bar.
Small arrow buttons	Move the stage by the specified step size in the vertical and horizontal directions.

* **OM Search** is an optional function.

• Entering the analysis position in the Stage mode

- 1. Confirm that Scan Type is Stage. If it is not, click on the Stage button.
- 2. Move the stage to the analysis position that you want to analyze, while observing the OM image, by using the joystick of the Joystick Controller of the EPMA main unit; then after focusing on the position, eliminate backlash by using the TEST button of the Joystick Controller.

It is especially necessary to eliminate backlash before you perform continuous analysis in the Preset mode.

3. Click on the **Read** button to display the present stage position; then click on the **Store** button to enter the coordinates of the position.

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

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

• Entering the analysis position in the Beam mode

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

• One-by-One Measurement

1. Click on the **One-by-One** button in the Stage Condition window.

The One-by-One Measurement window appears as shown in Fig. 24.

-		St	andard Anal	ysis		
		One by	One Measu	rement		
Sample Na Standard Accelerat Neasureme Position	me Type ing Voltag nt Mode	je	LaF3 Alloy-M 20.0 kt NDS-EDS 1 X: 66.75	V Parallel 17 Y: 37.	.6873 Z:	11.7075
Element Channel Signal Crystal	Elem-1 La 2 HDS PETJ	Elem-2 La 3 HDS LIFH	Elem-3 La 4 HDS LIFH	Elem-4 F 5 NDS TAP	Elem-5 La 8 EDS ROI-1	Elem-I La 9 EDS ROI-:
-	Fi	Acquire		Canc	9e1	H

Fig. 24 One by One Measurement window

2. Click on the Acquire button.

The program executes a single measurement.

If you want to measure multiple standard samples at one time, use Preset Measurement, which will be described later.

Condition Load

The Condition Load function is used to recall conditions of standard samples that you recorded in advance.

 Select Measurement–Condition Load from the Standard Analysis function window.

The Condition File Load window opens as shown in Fig. 25. This window displays **Name** (file names), **Date** (the dates when they were recorded) and **Comment**.

- Standard Analysis							
	Condition File Load						
Select Name			Sort Order				
No.	Name	Date	Comment				
1	Dy	Sep-12-2000	LEE element				
2	A1203	Sep-12-2000	Al Oxide				
3	PBS	Sep-12-2000	PBS measurement				
			۲I ۲I				
I	load	Check	□ Update peak pos. Cancel				

Fig. 25 Condition File Load window

To call up the conditions, select the desired file from the list of recorded conditions in the window, and then click on the Load button. The conditions that will be loaded are Standard Type, Standard Composition, Element Condition, EOS Condition, EDS Condition, Print-out Condition, Measurement Mode, and Additional Function.

If you have selected the **Check** before loading, the stored conditions are displayed in the Listing window in which you can confirm the conditions.

If you have selected **Update peak pos.** button before loading, the stored conditions will be loaded after they are updated.

If you specify a condition file for a sample with a composition different from that of the specified standard sample, a warning message appears, asking you to confirm.

Condition Store

The Condition Store function allows you to save the measurement conditions.

- Select Measurement–Condition Store from the Standard Analysis function window.
 - The Condition File Store window opens The Condition File Store window opens.
- 2. Click on the New button in the Condition File Store window. The Condition File Name window opens.

-	Standard /	Analysis
	Condition F	ile Name
Namo	LiF	
Comment	LiF measureament	
	OK	Cancel

Fig. 26 Condition File Name window

3. Enter the desired file name (up to 14 characters) and a comment (up to 40 characters) in this window and click on the **Store** button of the Condition File Store window.

This procedure allows you to record in a file the analysis conditions you have set. The conditions that will be stored are **Standard Type**, **Element Condition**, **EOS Condition**, **EDS Condition**, **Print-out Condition**, **Measurement Mode**, and **Additional Function**.

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

Check Data

This window is used to check data for standard samples that have already been measured, and also to select accumulated data.

 Select Measurement–Check Data from the Standard Analysis function window.

The Standard data window opens as shown in Fig. 27.

	Off-line Correc	stine	
Tabuott	ndard data		
Ho. Elem CH(Cryst) Name	Acc V Ht. % STD Fac.	L Value Current Not int. Acm	
1 C 1(LDE1H) L6_C_6_Mn	15.0 19.223 10.5243	121.595 1.5200-06 24555.7 20	5
2 # 2(L3P) L4_C_8_Mo	15.0 0.738 2.1265	172.078 1.5208-86 1813.1 20	
3 Hn 4(PETH) L0_C_9_Hn	15.0 32.940 0.2814	146.282 1.5206-06 12991.1 20	
77L 3			
Have Report	Print	Cancel	

Fig. 27 Standard data window

The window displays element name (in the **Elem** column), standard sample name (in the **Name** column), accelerating voltage (in the **Acc V** column), mass percent (in the **Mass** % column), standard sample factor (in the **STD Fac.** column), peak position (in the **L. Value** column), beam current (in the **Current** column), net X-ray intensity (in the **Net int.** column), and number of accumulations (in the **Acm** column), in that sequence.

• To select accumulated data, click on the Acm button, and click on the number of accumulation button.

The Select Accumulations window opens as shown in Fig. 28, allowing you to select the desired accumulated data.

Sel	ect Accumulations	
c	in LS_C_S_Mn	
No.	cps	
1	25997.3	
2	22308.9	
3	24802.3	
4	26099.0	
5	25082.3	
6	22923.2	
7	23854.3	
8	23847.3	
9	26786.8	
10	23429.0	
Average	24512.8 / 18	
Signa	1548.99	
ОК	Print Cancel	ľ

Fig. 28 Select Accumulation window

Click on the X-ray data to be removed from the accumulation if you want to remove them. New average intensities are calculated. To save this data, click on the **Save** button. If you click on the **Reset** button, the changes made to the data will be deleted. You can print this table by clicking on the **Print** button.

- If you measured different X-rays simultaneously for the same element, you can output the standard sample factor for only one of the X-rays. This is due to the limitation of the software. In the case of an actual unknown sample, calculation is executed using the correct X-rays. The ZAF factor is always calculated as the standard sample factor here, regardless of the correction mode for the unknown sample. Needless to say, when correction is performed for an unknown sample, calculation takes place according to the actual correction mode, so there is no problem.
- When using calibration curves instead of a standard sample, the A and B factors can be output for Check Data. This function enables you to easily check the A and B factors when you want to display the results of map analysis in concentration.

Print-out Condition

 Select Measurement–Print-out Condition from the Standard Analysis function window.

The Print-out Condition window opens as shown in Fig. 29.

-	Standard Analysis				
Γ	Print-out Condition				
	Measurement Condition				
	Measurement Result				
4	Full 🔷 Brief				
	OK Cancel				

Fig. 29 Print-out Condition window

Of the two items shown in the window, click on one or both of the two so that the selected items can be printed; then click on the **OK** button.

Turning off Measurement Result stops printing during measurement.

K The contents of the output are the same for both **Full** and **Brief** for the present.

Measurement Mode

With the JXA-8200 Series, this function allows you to specify the sequence of measurement when you use both the WDS and the EDS. For measurement using either only the WDS or only the EDS, any measurement mode specified here will be invalid.

 Select Measurement–Measurement Mode from the Standard Analysis function window.

The Measurement Mode window opens as shown in Fig. 30.



Fig. 30 Measurement Mode window

Button	Function
WDS-EDS Parallel	Performs measurements using both the WDS and EDS simultaneously.
EDS->WDS Serial	Performs measurements first with the EDS and then with the WDS.
WDS->EDS Serial	Performs measurements first with the WDS and then with the EDS.

Additional Function

GP Refer to "Additional Function" in the separate instruction manual, "QUANTITATIVE ANALYSIS PROGRAM".

- Additional Function
Peak Search
💠 Always 🔷 None 💠 Every
Background Measurement
😞 Always 🗢 None 😞 Every
Synchronus Measurement
◆ Asynchronous 😞 Synchronous
Peak Search Position
OK Cancel

Fig. 31 Additional Function window

Survey Measurement

This function allows you to check the peak intensity preliminarily before executing definitive measurement. Thus, the acquired data cannot be used for actual correction in quantitative analysis.

This window is used to carry out measurement at the present stage position as well as under the present EOS conditions.

 Select Measurement–Survey Measurement from the Standard Analysis function window.

The Survey Measurement window opens as shown in Fig. 32.

	Survey Measurement						
Sample Na Standard Accelerat Measureme Position	me Type ing Volta nt Hode	ю	LaF3 Alloy-M 10.0 kV NDS-EDS ⁻ X: 66.757	7 ¥:37.	6873 Z:	11.7075	
Element Channel Signal Crystal	Elem-1 La 2 MDS PETJ	Elem-2 La 3 NDS LIFH	Elem-3 La 4 MDS LIFH	Elem-4 F 5 NDS TAP	Elem-5 La 8 EDS ROI-1	Elem-I La 9 EDS ROI-2	
	a					×	
		Acquire		Cano	el		

Fig. 32 Survey Measurement window

Preset Measurement

The Preset Measurement function enables you to measure multiple standard samples continuously.

 Select Measurement–Present Measurement from the Standard Analysis function window.

The Select Preset Samples window opens as shown in Fig. 33.

- Standard Analysis								
Select Preset Samples								
	Select	ted List	Sor	ting O	rder			
Gr	oup :	A11	4 B	lane 🔫	Date			
	No.	Name	Date	NDS	EDS			
11	10	н	Aug-31-2000	*	- 4			
	11	Nb	Aug-31-2000	*				
	12	PBS	Aug-31-2000	-	· • 🗌			
	13	HONAZITE	Aug-31-2000	*	• -			
	14	Th02	Aug-31-2000	*				
	15	PrF3	hug-31-2000	*				
	16	NdF3	Aug-31-2000	*				
	17	LaF3	Aug-31-2000	*				
	18	CeF3	Aug-31-2000					
	19	HOn	Aug-31-2000	-	* 17			
Total 2 samples selected								
Acquire Clear All Cancel								

Fig. 33 Select Preset Samples window

To select the standard samples to be measured, click on the buttons at the left of their No. and Name, and click on the Acquire button; then measurements begin. During measurement, the Preset Measurement Listing window shown in Fig. 34 displays the analysis progress for the measurement being made.

If you want to stop measurement while it is in progress, click on the **Measurement Stop** button in the Measurement Control window; then the measurement stops and all the data acquired up to that time will be stored.

Standard Measurement WDS elements CH Acc.v (1) 15.0 (5) 15.0 Element Crystal TAP Peak Pos. 108.236 BG_L 10.000 BG_U (mm) 13.000 X-ray (nm) 2.36200 1 0 2 Al Ka TAP 90.563 0.83393 5.000 5.000 Ka 30 Ka TAP (5) 15.0 112.590 2.36200 10.000 13.000 Element Peak Back Pksk Gain High.V Base.L Window.W Mode 1700 1 0 2 Al 3 0 10.0 10.0 5.0 (s) 5.0 (s) 1 64 32 1.0 -(V) (V) Int 1700 1 Int 5.0 (s) 32 1700 1.0 (¥) 10.0 Int Standard Data Standard name Mass(%) ZAF Fac. Element 1 0 2 Al 3 0 1.0000 11.1895 4.4945 31.0862 5.8915 0.3600 A1203 47.0773 52.9227 0.7629 A1203 1.0000 1.0000 47.0773 11.1895 31.0862 0.3600 A1203 Acc. Voltage : X= 41.9821 Y= Dated on Sep 12 13:34 2000 MDS only No. of Sec. 61.5662 Z= 11.7120 Probe Dia. : 10 Scan : Off No. of accumulation : 1 Standard Intensity of WDS Curr.(A) : 1.000E-06 Elem. Cryst. CH L(mm) 1 0 TAP 1 108.236 Net(cps) Bg-(cps) Bg+(cps) 500.0 501.0 501.0 S.D.(%) D.L.(ppm) 2.44 9332 542298.2 26532.7 0.26 Previous 108.236 13364.3 323 Aug 29 22:24 2000 2 A1 TAP 3 0 TAP 4688 90.563 2500.0 5 2501.0 2501.0 1.09 5 112.590 2500.0 2501.0 2501.0 1.09 4170 Previous 112 590 143789 4 3553 6 1913 0 Ø 49 450 Aug 29 22:24 2000

Fig. 34 Preset Measurement Listing window

- For the JXA-8200 Series, when you execute an analysis that involves EDS control or EDS measurement, make sure that the EDS Home Window is either being displayed on the screen, or reduced to a small icon. If you execute EDS control or EDS measurement when the EDS Home Window is not activated, the instrument will stop measurement due to an error.
- If you have selected Analysis-Peak Search Monitor from the EPMA Main Menu prior to measurement, the Peak Search Monitor window will be displayed. In the window, you can observe WDS element peak profiles in real time during measurement. However, never open the Peak Search Monitor window during peak search; if you do, peaks may not be accurate.

4.1.4 Measuring standard samples

Measure a standard sample as follows.

Set the conditions of the electron optical system, such as the accelerating voltage, and drive the sample to the analysis position. Close the beam with PCD and measure the beam current, then open the beam and perform a peak search.

A peak search takes place based on the peak search number. By default, the peak search conditions are defined as follows.

Peak search No.	Full width at half maximum (FWHM)	Number of points	Sampling time	
3	8.0	400	0.1 s	Coarse
2	4.0	200	0.1 s	Medium
1	1.0	100	0.1 s	Fine
0	0.0	0	0.0	None

If you want to change these parameters, execute **Check pksk parameter** on the peak search. Note, however, that if you change these parameters, the peak search may fail, so ensure that the parameters are changed only by an adequately skilled operator.

The FWHM used in the above table is calculated using the following equation. FWHM = $(A \times L \text{ (mm)} + B) \times Pksk$

You can verify these values by clicking on the JEOL icon in the EPMA Main Menu, and then using Check Config-Crystal defaults (the fourth and fifth items of this window are the A and B parameters, and the seventh item is the Pksk parameter). To change the FWHM, correct the Pksk parameter, not the A and B parameters. The A and B parameters are shared with the qualitative analysis program, so the results of element identification in qualitative analysis may sometimes be abnormal if you change A or B.

The new peak position after the peak search of the standard sample will be re-written in the condition file of each analysis software. This enables each software to share the peak positions that are common to the standard sample. However, you cannot change the stored contents of the condition file.

After a peak search, the X-ray intensity is measured at the peak position, the background low-angle side, and the background high-angle side, in that order. When Accumulation is specified, measurements are repeated in the above sequence for each point, and the average value will be obtained. However, the peak search takes place only at the first point. The dead-time correction will be performed for the measured X-rays, and the results will be displayed in cps.

The measured X-ray intensity is saved based on the Sample name, element name, X-ray name, order, accelerating voltage, channel and analyzing crystal. Consequently, so long as these are not identical, they can be saved under one standard sample name.

The results output to the printer consist of the X-ray intensity that has been corrected for background, background low-angle side intensity, background high-angle side intensity, S.D. (%) and S.V. (%) (Fig. 35).



Fig. 35 Relation between peak and background

The net X-ray intensity is calculated using the following equation.

• Intensity after dead-time correction

$$I(cps) = \frac{X/t}{1 - \tau X/t}$$

where	X :	Count number of measured X-rays				
	t:	Count time (s)				
	:	Dead time (In the JXA-8200,), which is about 1 μ s, is set in the				
		Hardware Configuration window.)				

• Net X-rays

$$I_{net} = I_{peak} - \frac{I_{PBH} \overline{L_{PBL}} + I_{PBL} \overline{L_{PBH}}}{\overline{L_{PBL}} + \overline{L_{PBH}}}$$

- I_{net} , I_{peak} : Intensities of net X-rays and measured X-rays at the peak position
- I_{PBH} , I_{PBL} : Intensities of background X-rays at high and low angles

 $\overline{L_{PBH}}$, $\overline{L_{PBL}}$: Separations between peak and high and low angles where background was measured

• Standard deviation in X-ray counting

Standard deviation in X-ray counting is obtained as the value corresponding to 1σ using the following equation.

$$S.D.(\%) = \frac{100}{I_{net}} \sqrt{\frac{I_{peak}}{t_{peak}}} + \left(\frac{\overline{L_{PBH}}}{L}\right)^2 \frac{I_{PBL}}{t_{PBL}} + \left(\frac{\overline{L_{PBL}}}{L}\right)^2 \frac{I_{PBH}}{t_{PBH}}$$

 t_{peak} , t_{PBL} , t_{PBH} : Counting time (in seconds) at the peak, and of the background signals at low and high angles

$$L = \overline{L_{PBH}} + \overline{L_{PBL}}$$

• Standard variance after accumulating signals

When measurement of X-rays at multiple points is performed using accumulation, standard deviation is calculated using the following equation. However, if the number of accumulations is one, this item is not calculated.

$$S.V.(\%) = \frac{100}{I_{net}} \sqrt{\frac{(I_i - \overline{I})^2}{n - 1}}$$

- I_i : Intensity of X-rays on each measurement
- \overline{I} : Average intensity of net X-rays
- *n*: Number of accumulations

• Detection limit

The detection limit is calculated in ppm using the following equation.

$$D.L. = \frac{1}{\frac{I_{net STD}}{mass(\%)_{STD}}} \sqrt{\frac{2 \times I_{back}}{t_{back}}}$$

I_{back} :	Average intensity of background X-rays
t_{back} :	Counting time of the background signals
I_{netSTD} :	Intensity of net X-rays of the standard sample
$mass(\%)_{STD}$:	Mass concentration in the standard sample

4.1.5 Area intensity measurement/FIT mode measurement

G Refer to Section 8.4 "Area Intensity Measurement/FIT Mode Measurement" in the separate instruction manual, "QUANTITATIVE ANALYSIS PROGRAM".

4.2 Measurement Using Calibration Curve

You can measure up to 20 standard samples, and can obtain the relationship between concentration and X-ray intensity using the method of least squares. The curve equation can be selected from the first order to the third order.

The calibration-curve method enables you to perform measurement to a very high accuracy, since a lot of standard samples are used. The slope and background of the calibration curve are also used as data for displaying the concentration of elements in map analysis and line analysis.



Fig. 36 Curve equation obtained using the method of least squares

First order:Int = Ax + BSecond order:Int = $Ax^2 + Bx + C$ Third order:Int = $Ax^3 + Bx^2 + Cx + D$

where Int is in counts/($ms \cdot \mu A$), and x is mass%.

The operation method is roughly the same as that employed by the ordinary standard sample analysis program. Consequently, this instruction manual describes only what is different, that is, Standard Type, Element Condition, Stage Condition and Check Data.

Standard Type

1. Select Measurement–Standard Type from the Standard Analysis function window.

The Standard Type window opens as shown in Fig. 37.



Fig. 37 Standard Type window

2. Select Calibration Curve in the Standard Type window.

Element Condition

- **1.** Select **Measurement–Element Condition** from the Standard Analysis function window.
 - The Element Condition window opens as shown in Fig. 9.
- 2. Click on the Standard Composition button in the Element Condition window.

The Standard Composition window opens as shown in Fig. 38.

E											
	Standard Composition										
Q A	🕹 Atom 🕹 Hole 🔶 Hass										
Curs	Cursor 🔷 Vertical 🔹 Horizontal										
No.	No. of Standards 5 Standards										
No.	Elen.	Std-1	Std-2	Std-3	Std-4	std-5					
1	Cr	0.6930	0.3000	1.3100	0.0660	0.0072					
2	Mo	0.1900	0.0700	0.0300	0.4900	0.0050					
3	с	0.3910	0.1630	0.6260	0.8700	0.0067					
4	P	0.0160	0.0440	0.0290	0.0100	0.0025					
5	s	0.0150	0.0370	0.0057	0.0250	0.0055					
6											
7											
8											
9											
10											
TTL	5	1.3050	0.6140	2.0007	1.4610	0.0269					
		ial				Delete					
	OK Cancel										

Fig. 38 Standard Composition windo	ow
------------------------------------	----

3. Click on the **No. of Standards** button, and then enter the number of standard samples in the window opens.

The number of columns for inputting chemical composition increases to the number of standard samples.

- **4.** Using the keyboard, enter each element name and also its mass concentration in the columns for inputting chemical composition.
 - Be sure to enter values as mass concentrations when using the calibration-curve measurement method.
 - **K** To perform a high-precision calibration-curve measurement, prepare multiple standard samples whose element composition and concentration are close to those of an unknown sample, and whose concentration is different.

Stage Condition

In **Stage Condition**, enter as many coordinate positions as the number of standard samples. The procedure for specifying the stage position for calibration-curve measurement is the same as that for ordinary standard samples.

- **G** Refer to Stage Condition in Section 4.1.3.
- When a trace element is used as a standard sample to obtain a calibration curve, specify Accumulation using the Pos. Input button for a better measurement in the wide range of X-rays.
- Select Measurement–Stage Condition from the Standard Analysis function window.

[- Standard Analysis									
I	Position Correction Stage									
l										
l	Sample		LA	5						
	Preset	N	».	Comment	S/B	Acm.	st	age (X,Y,	Z)	
ľ		1	No.	1	S	5	26.8549,	57.7524,	11.5950	1
I	-	2	No.	2	S	5	23.5079,	57.7358,	11.6055	
I	-	3	No.	3	s	5	19.1408,	56.8937,	11.6200	
I		4	No.	4	S	5	15.0554,	56.8109,	11.6305	
I	-	5	No.	5	s	5	11.0245,	56.8122,	11.6415	
I										
I										
I										
I										
1										
I		_	_	-		- 1	- 1	-		
i		Pos.	Inpu	t	One-by-	one	Clea	ar	Close	

The Stage Condition window opens as shown in Fig. 39.

Fig. 39 Stage Condition window

Preset Measurement

The Preset Measurement function enables you to measure multiple standard samples continuously. The resulting calibration curves will be displayed in the Calibration curve window.

 Select Measurement–Preset Measurement from the Standard Analysis function window.

The Select Preset Samples window and Calibration curve window open.

 To specify the names of samples whose calibration curves are to be obtained, click on their buttons at the left of No. and Name, and then click on the Acquire button.

The measurement of the sample selected for obtaining a calibration curve will be executed and its calibration curve will be displayed in the Calibration curve window as shown in Fig. 40.



Fig. 40 Select Preset Samples window

- When you want to obtain the calibration curve of a trace element, set **Peak Search** of the Additional Function window to **None**.
- In the calibration curve method, the peak intensity including background intensity is used as characteristic X-ray intensity; set **Background Measurement** of the Additional Function window to None.

Check Data

This function enables you to check A and B factors of a calibration curve after measurement, to perform least square calculation again by selecting accumulated data for standard samples that have already been measured, and also to change the order of a calibrated curve.

 Select Measurement–Check Data from the Standard Analysis function window.

The Calibration curve window opens as shown in Fig. 42.

2. Select File-Element from the Calibration curve window.

The Intensity window opens as shown in Fig. 41.



Fig. 41 Intensity window

The Intensity window displays the results of calibration-curve measurement in the sequence of element name (Element), standard sample name (Name), order of calibration curve (Order), slope A and intercept B (in the case of higher orders, also C and D are displayed), and the number of standard samples (STDs).

Clicking on each of the No. buttons in Fig. 41 enables you to confirm the calibration curve corresponding to the selected element.

To change the order of calibration curve for a standard sample, click on the **Order** button, and then select the desired order from **1** to **3**.



Fig. 42 Second-order calibration curve

To re-calculate a calibration curve after changing the data of standard samples, click on the **STDs** button; then the data of standard samples will be displayed in the Standard-sample selection window. Select again the desired standard samples by clicking on the buttons on the left side; then a new calibration curve will be obtained after automatic re-calculation. You can enter concentration and intensities using the keyboard.

Calibration curve										
	c c	H-1 (LDE2H)	15.0 kV							
	A: 10	5.513170 B: 10	.015111							
No.	Mass%	cps/uA	Diff	ACH						
1	0.3910	17048.0	576.2	20	4					
2	0.1630	12092.0	-614.8	20						
3	0.6260	21046.8	694.4	20						
4	0.8700	23738.2	-643.3	20						
5	0.0067	10113.3	-12.5	20	L L					
TTL 5										
	or	Print		1						
1		Print	<u></u>	ancei						

Fig. 43 Standard-sample selection window

To check the accumulated data of each standard sample, click on the ACM button of Fig. 43; then the Select Accumulations window opens as shown in Fig. 44. This window displays the X-ray intensity of each accumulation.

Every time you click on the desired item, selection changes between on and off. When you have measured an uneven sample, to obtain its calibration curve, select only necessary items.



Fig. 44 Select Accumulations window