INSTRUCTIONS

C
CONTENTS

XM-17330/27330

BASIC SOFTWARE/ QUANTITATIVE ANALYSIS PROGRAM

IXM-17330/27330-1QN (608001)
DEC2001-03210210
Printed in Japan

JEOL
Serving Advanced Technology

NOTATIONAL CONVENTIONS AND GLOSSARY

■ General notations

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

■ Mouse operation

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

CONTENTS

1 GENERAL

This program is software for performing quantitative analysis by using the JXA-8100/8200 EPMAs. Usually, standard specimens whose concentration is known should be measured in advance for an accurate quantitative analysis. Before using this program, measure standard samples by using the separate program titled “Standard Sample Analysis Program”, which is provided by us together with the main unit.

If very high accuracy is not a critical concern, you may use the CAL-STD mode, a calculation mode to be used in case standard samples are not available.

This program uses the ZAF correction method as standard. In addition, the $\phi(\rho Z)$ method, B & A method, thin-film correction method and other similar methods can be used as optional correction methods. The program is also applicable to quantitative analysis based on the calibration curve method or by using the WDS/EDS combined microanalyzer (a standard feature for the JXA-8200).

Another consideration incorporated in the program is compatibility of its basic analytical data and operations with other programs such as the Qualitative Analysis Program and Map Analysis Program.

For example, stage position data and the method of entering it in this program are compatible with those for the Qualitative Analysis Program. Element condition data and its entry method are also compatible with those for the “Line Analysis and Map Analysis programs”.

In short, this program offers operational ease and advanced functions of quantitative analysis in one package.

2 SPECIFICATIONS

Number of elements to be measured in an unknown specimen: 30 elements maximum
Measuring method: Asynchronous concurrent driving measurement or synchronous measurement for each spectrometer
WDS spectrometer X-ray counting method: Fixed time, fixed count, area, FIT, or waveform separation
WDS spectrometer X-ray measurement time: 0.1 to 1,000 s
EDS spectrometer X-ray counting method: Fixed time (live time or real time)
EDS spectrometer X-ray measurement time: 1 to 10,000 s
Number of preset measurement points per specimen: 1 to 10,000
Number of accumulations per measurement point: 1 to 100
Real time peak search display during measurement: Possible

ZAF correction calculation for unknown specimen:	
	ZAF quantitative correction calculation for metals ZAF quantitative correction calculation for oxides
Correction calculation mode:	Normal Fix Atomic ratio Weight ratio Difference Anion Standardless (standard specimen measurement not necessary)
Output of analysis results:	Summary display of calculation results Qualitative analysis results Semi-quantitative analysis results Chemical formula End member computation
Offline correction calculation:	Executable by changing measured data, standard specimen data, etc.
ZAF correction calculation method	
• Atomic number correction:	Philibert-Tixier method*
• Absorption correction:	Philibert method**
• Fluorescent excitation correction:	Reed method***
Elements to be measured:	76 elements from Be to U, excluding rare gases and radioactive elements
Analyzable elements (matrix):	92 elements from H to U
X-ray names available for measurement:	$K\alpha$, $K\beta$, $L\alpha$, $L\beta$, $M\alpha$, $M\beta$ and their higher-order lines up to the specified orders

---

[*] J. Philibert and R. Tixier, *Brit. J. Appl. Phys.* (J. Phys. D), Ser. 2, 1, 685 (1968)

[**] J. Philibert, *X-ray Optics and X-ray MicroAnalysis*, Academic Press, New York, p. 379 (1963)

[***] S. J. B. Reed, *Brit. J. Appl. Phys.*, 1965, 16, 913 (1965)

3 PROGRAM STRUCTURE

The structure of this program is shown in the tree diagram below.

- Analysis
  - Quantitative Analysis
    - Sample
    - Measurement
      - Correction Method
      - Element Condition
      - EOS Condition
      - EDS Condition*
      - Standard Condition
      - Substrate Composition**
      - Stage Condition
      - Condition Load
      - Condition Store
      - Printout Condition
      - Measurement Mode*
      - Survey Measurement
      - Preset Measurement

- Process
  - Quantitative Analysis
    - Summary
      - Sample
      - Summary
      - List order
      - Analysis
      - Calculation
      - Chemical Formula
    - Off-line Correction
      - Sample
      - Condition
        - Analysis
        - Correction Method
        - Element Condition
        - EOS Condition
        - EDS Condition*
        - Standard Condition
        - Standard Data
        - Substrate Composition**
        - Condition Load
        - Condition Store
        - Printout Condition
        - K-Ratio input & Correction
        - Intensity input & Correction
        - File read & Correction

*: Available only for JXA-8200

**: Optional software

4 ZAF CORRECTION METHOD

The ZAF correction method determines the mass concentration C_A of an element A from the following equation:

$$C_A^{UNK} = K_A^{UNK} \times G_Z \times G_A \times G_F$$

$$= K_A^{UNK} \times \frac{R_A^{STD} \times P_A^{STD}}{R_A^{UNK} \times P_A^{UNK}} \times \frac{f_A^{STD}(\chi_A)}{f_A^{UNK}(\chi_A)} \times \frac{1}{1+\frac{I_A(f)}{I_A(d)}}$$

where $K_A^{UNK} = \frac{I_A^{UNK}}{I_A^{STD}}$ and I_A are the X-ray intensities (in cps) per unit current of element A after dead-time correction and background correction; UNK indicates the unknown sample; STD indicates the standard sample; and G_Z, G_A, and G_F are atomic-number correction coefficient, absorption correction coefficient, and fluorescence correction coefficient, respectively.

The following are brief descriptions of atomic-number correction, absorption correction, and fluorescence correction.

- Atomic number correction (Z)

  Atomic-number correction deals with differences in the behavior of incident electrons between an unknown sample and a standard sample. This correction is usually considered in two factors: the P factor, including the stopping power term, which indicates penetration of electrons into the sample, and the R factor which indicates the backscattering of electrons from the sample. The two factors tend to cancel each other, so that the correction factors are not very large in most cases.

- Absorption correction (A)

  Absorption correction deals with differences in the degree of X-ray absorption between an unknown sample and a standard sample. This correction gives consideration to the mass absorption coefficient, incident electron energy, and X-ray take-off angle.

- Fluorescence correction (F)

  Fluorescence correction needs to be considered if the characteristic X-rays from an element, B, are at the short-wavelength (high-energy) side of the absorption edge of another element, A. In this case, the X-rays from element B may excite element A. The same is true with fluorescence excitation by continuous X-rays, but the effect usually is negligible.

4.1 Atomic Number Correction

The basic equation of atomic number correction is

$$G_Z = \frac{R^{STD}}{R^{UNK}} \times \frac{P^{STD}}{P^{UNK}}$$

where R is the backscattering factor and P is the penetration factor.
The backscattering factor of an unknown sample is determined from:

$$R = \sum_j C_j \, R_{ji}$$

$$R_{ji} = R\left(Z_{j,} U_i\right)$$

where C_j : concentration of element j, $\Sigma Cj = 1$

Z_j:	atomic number of element in matrix
U_i:	over-voltage ratio (= E_0/E_{Ki})
E_{Ki}:	K-shell excitation energy of element i
E_0:	accelerating voltage

The value R_{ji} is determined by calculating the values*, that are given by Duncumb-Reed, through the method of least squares:

$$\begin{aligned} R(Z_j, U_i) = 1 - X \cdot V \Big[& -0.187204 + 1.67366V - 1.51187V^2 + 0.707063V^3 \\ & + X\left\{3.63547 - 1.9914V - 1.6539V^2 + 0.477246V^3\right\} \\ & + X^2\left\{-4.04514 - 4.11268V + 9.26509V^2 - 2.79199V^3\right\} \\ & + X^3\left\{1.54054 + 3.43134V - 5.22007V^2 + 1.23353V^3\right\} \Big] \end{aligned}$$

where $X = Z_j/100$, $V = 1 - 1/U_i$

The penetration factor P_i is given by

$$P_i = \frac{1}{M}\left[\left(U_i - 1\right) \frac{1}{W_i} lnW_i \left\{ li \left(U_i W_i\right) - 1i \left(W_i\right) \right\} \right]$$

where li (x) is a logarithmic differential function expressed as

$$li\left(x\right) = C + ln \left| ln\, x \right| + \sum_{r=1}^{\infty} \frac{\left(lnx\right)^r}{rr!}$$

C: Euler's constant

$$lnW_i = \frac{1}{M} \sum_j C_j \frac{z_j}{A_j} ln \frac{1.166 E_{Ki}}{J_j}$$

i: emitter, j: absorber

$$M = \sum_j C_j \frac{z_j}{A_j}$$

Aj: atomic weight of element j

J_j: average ionization potential of element J (eV)

---

* P. Duncumb. and S. J. B. Reed, NBS Special Publication, 298, P. 133 (1968)

$$J_j = 9.76 Z_j + 58.8 Z_j^{-0.19} \qquad \text{(Berger-Seltzer}^{*}\text{)}$$

$$= Z_j \left(9.76 + 58.8 Z_j^{-1.19} \right)$$

For each element, the atomic weight is given in the file atom.asc in the directory /opt/epma/phys, while the excitation energy is given in the file abs_edge.dat in the same directory.

---

[*] M. J. Berger and S. M. Seltzer, *Nat. Acad. Sci.*, Nat. Res. Counc. Publ. 1133, P. 205 (1964)

4.2 Absorption Correction

Absorption correction uses the following Philibert formula:

$$f(x) = \frac{1+h}{\left(1+\frac{x}{\sigma}\right)\left\{1+h\left(1+\frac{x}{\sigma}\right)\right\}}$$

where

$$h = 1.2\frac{A}{Z^2}$$

A: atomic weight
Z: atomic number

$$\sigma = \frac{4.5\times10^5}{E_0^{1.65} - E_K^{1.65}} \quad \text{(Lenard's coefficient)}$$

E_0: accelerating voltage (in keV)
E_K: K-shell excitation energy (in keV)

$$x = (\mu/\rho)\,\mathrm{cosec}\,\theta$$

θ: X-ray take-off angle
μ/ρ: mass absorption coefficient

The mass absorption coefficient is determined as follows:

- Z < 10 (wavelength λ is more than 12Å): The values given by Henke[*] are used.
- Z ≥ 10 (wavelength λ is 12Å to 0.7Å): The values calculated by Heinrich method[**] are used.

Heinrich method

- The mass absorption coefficient of characteristic X-rays (radiation) from an element for the absorber is given as:

$$\mu/\rho = C_i(Z)\,\lambda_R\, n_i(Z)$$

where C_i (Z) and n_i (Z) are values that depend on the atomic number (Z) of the absorber. The value i varies with the relation between the X-ray wavelength λ_R and the absorber's absorption edge.

- The wavelengths and coefficients needed for calculation are as follows:

Wavelength as a function of Z:	wavelength of X-rays (Kα, Kβ, Lα, Lβ, Mα, Mβ)
Absorber as a function of Z:	wavelength of absorption edge (K, L_1, L_2, L_3, M_1, M_2, M_3, M_4, M_5) coefficient C_i (C_K, C_{KL}, C_{L1}, C_{L2}, C_{LM1}, C_{M1}, C_{M2}, C_{M3}, C_{M4}, C_{MN}) coefficient n_j (n_K, n_{KL}, n_{LM}, n_{MN})

---

[*] B. L. Henke, *Adv. X-ray Anal.*, 9, 460 (1966)
B. L. Henke et al., *Atomic Data and Nuclear DataTables*, vol 27, No.1 (1982)

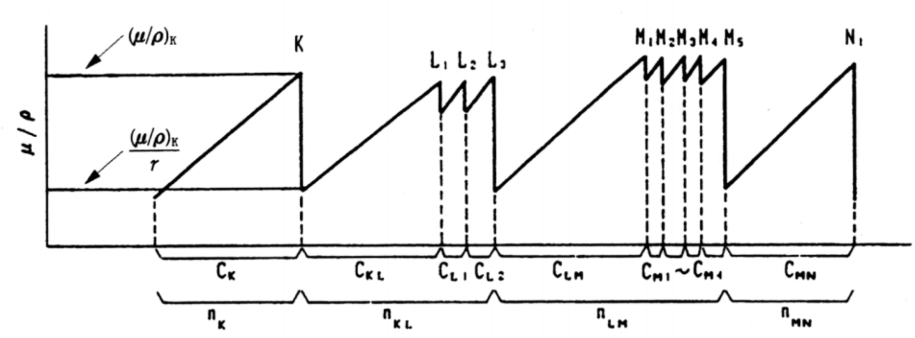
[**] K. F. J. Heinrich, *The Electron Probe* (New York, Wiley), P. 296 (1966)

All of these values are approximated by the quartic equation for the atomic number:

$$X = \exp\left\{A(0) + A(1)Y + A(2)Y^2 + A(3)Y^3 + A(4)Y^4\right\}$$

where Y = ln (Z)
A (i): constant

- The suffix i in C_i (Z) and n_i (Z) is determined by placing the absorption edges relative to the order of wavelength and identifying the positions of wavelength, λ_R, of characteristic X-rays, as shown below.



- ✍ The values C_i and n_i are given in a table by Heinrich. The constants for A (0) to A (4) are determined so that they may come close to the values in the table. However, C_{M4} is unknown.
- ✍ In C_i, the values C_{L1} and C_{L2} are proportional to C_{KL}, while the values C_{M1}, C_{M2}, C_{M3} and C_{M4} are proportional to C_{LM}. Therefore, only C_K, C_{KL}, C_{LM} and C_{MN} are calculated in the quartic equation; the others are determined by multiplying them with the proportional constants.
- ✍ For n_i, Heinrich always specifies $n_{LM} = 2.6$ and $n_{LM} = 2.22$.
- ✍ The values for the absorption edge and wavelength are also calculated with the quartic equation of Z.

These mass absorption coefficient values are stored in the following files in the directory /opt/epma/phys/mac: Ka.mac, Kb.mac, La.mac, Lb.mac, Ma.mac, and Mb.mac.

4.3 Fluorescence Correction

Fluorescence correction usually refers to determining the value ${}^{\mathrm{fl}}\gamma_{\mathrm{A}}$ in the equation below. Assuming the specimen is a binary element system in which element A is excited by element B, we have:

$${}^{fl}\gamma_A = \frac{I_A(f)}{I_A(d)} = \frac{1}{2} C_B \frac{r_K^A - l(\mu/\rho)_B^A}{r_K^A(\mu/\rho)_B^{AB}} W_K^B \frac{A_A}{A_B}\left(\frac{U_0^B-1}{U_0^A-1}\right)^{1.67}\left\{\frac{1n(1+x)}{x}+\frac{1n(1+y)}{y}\right\}$$

where

I_A (d): intensity of primary X-rays of element A subject to direct excitation

I_A (f): intensity of X-rays due to fluorescence excitation of the other element B

C_B: concentration of element B

r_K^A: absorption edge jump factor for element A

$(\mu/\rho)_B^{AB}$: specimen's mass absorption coefficient for X-rays from element B

$(\mu/\rho)_B^A$: element A's mass absorption coefficient for X-rays from element B

W_K^B : K-shell fluorescence absorption rate of element B

A_A: atomic weight of element A

A_B: atomic weight of element B

$$U_0^A = E_0 / E_K(A)$$

$$U_0^B = E_0 / E_K(B)$$

$$x = \left\{(\mu/\rho)_A^{AB} / (\mu/\rho)_B^{AB}\right\} \mathrm{cosec}\theta$$

$$y = \sigma / (\mu/\rho)_B^{AB}$$

Considering the excitation caused by other X-rays, Reed transformed the above equation to the following:

$${}^{fl}\gamma = C_B J_A D \frac{(\mu/\rho)_B^A}{(\mu/\rho)_B^{AB}} \{g(x) + g(y)\}$$

where

$$J(A) = \frac{1}{2} P_{ij} \frac{r_{K(L)}^A - 1}{r_{K(L)}^A} W_{K(L)}^A \frac{A_A}{A_B}$$

$$D = \left(\frac{U_0^B - 1}{U_0^A - 1}\right)^{1.67}$$

$$g(x) = \frac{ln(1+x)}{x}$$

P_{ij}: constant related to difference in generation of K-line and L-line

The values J (A) are given by Reed in a table.
The values are stored in the file Reed_JA.dat in the directory /opt/epma/phys.

✍ Whether fluorescence excitation occurs is checked by comparing the wavelengths at the absorption edges of each element and the wavelengths of all other X-rays generated from other elements.

✍ Fluorescence excitation can be caused by X-rays not even intended to be measured. This program also takes such X-rays into account in the correction calculations.

5 ACTUAL CALCULATIONS

Correction factors for a standard specimen of known composition should be determined in advance through the ZAF routine. (Even for pure substances, correction factors are not 1.)

The relative intensity K is determined by making a dead-time correction and background correction on the measured X-rays.

Dead-time correction

The counting rate Icps due to dead-time correction is obtained from the equation:

$$Icps = \frac{X/t}{1 - \tau X/t}$$

where

X: number of count of X-rays under measurement

t: measurement time

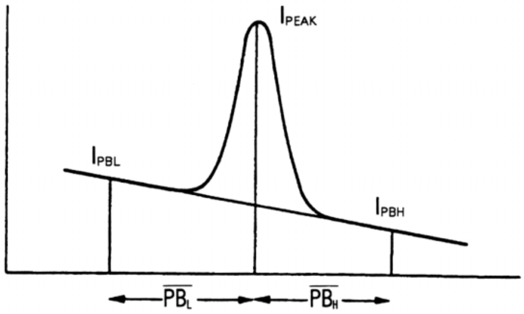
τ: dead time

(For the JXA-8100, $\tau \fallingdotseq 1.1$ μs. This is to be specified in the Hardware configuration window*.)

Background correction

The X-ray intensity Inet, due to background correction, is obtained from the equation:

$$Inet = Ipeak - \frac{I_{PBH}\overline{PB_L} + I_{PBL}\overline{PB_H}}{\overline{PB_L} + \overline{PB_H}}$$



where

Ipeak: X-ray intensity at peak position

I_{PBL}, I_{PBH}: X-ray intensities of backgrounds on low-angle side and high-angle side

$\overline{PB_L}$, $\overline{PB_H}$: Distance between peak position and background measurement position

---

* Refer to the instruction manual of the EPMA instrument.

Relative intensity K

The relative intensity K is obtained from:

$$K = \frac{I^{UNK} CUR^{STD}}{I^{STD} CUR^{UNK}} C^{STD}$$

where

I^{UNK}, I^{STD}: X-ray intensities of unknown specimen and standard specimen, respectively (in cps)

CUR^{UNK}, CUR^{STD}: total specimen currents of unknown specimen and standard specimen during measurement (A)

C^{STD}: weight concentration of standard specimen

At the beginning of calculation, the relative intensity measured is assumed as the mass concentration.

$$C_A^{UNK} = K_A^{UNK}$$

Initial assumption

$$K_A^{UNK\,\prime} = \frac{K_A^{UNK}}{K_A^{UNK} + K_B^{UNK} + \ldots K_N^{UNK}}$$

Normalization

$$C_A^{UNK\prime} = K_A^{UNK} \left\{ \left(\frac{f_A^{STD}(x_A)}{f_A^{UNK}(x_A)} \right) \times \left(\frac{R_A^{STD}}{R_A^{UNK}} \times \frac{P_A^{STD}}{P_A^{UNK}} \right) \times \left(\frac{1}{1 + I_A(f)/I_A(d)} \right) \right\}$$

First calculation

$$C_A^{UNK\prime\prime} = K_A^{UNK} \left\{ \left(\frac{f_A^{STD}(x_A)}{f_A^{UNK}(x_A)} \right) \times \left(\frac{R_A^{STD}}{R_A^{UNK}} \times \frac{P_A^{STD}}{P_A^{UNK}} \right) \times \left(\frac{1}{1 + I_A(f)/I_A(d)} \right) \right\}$$

Second calculation

The ZAF correction is iterated until the calculation converges. In this program, the calculation is iterated until the calculation error is reduced to within 0.001mass%, or until the tenth iteration.

6 OPERATION

To perform quantitative analysis, select **Quantitative Analysis** from the **Analysis** menu in the JEOL EPMA Menu. When you want to correct analysis data after measurement, select **Off-line Correction** from the **Process** menu. To print the summary of the results of quantitative analysis, select **Summary** from the **Process** menu. In each case, the function window opens. To terminate the function window, click on the **Exit** button.

6.1 Measurement

The general procedure for measurement is as follows.
After entering the sample name in which the data is to be saved, execute measurement by selecting the correction method, the elements to be measured, the conditions of the electron optical system and the coordinate position. You can save measurement conditions in advance and recall them when you execute measurement.

Before performing analysis of a sample whose elemental composition is unknown, it is important to determine what elements are contained in the sample by qualitative analysis, which scans the entire specimen and applies spectrometry. Also, by performing semi-quantitative analysis at this time, you can determine the approximate chemical composition. In order to obtain a highly precise quantification result, it is important to check whether or not interfering X-rays exist at peaks or the position of background signals. For this purpose, select **Quant. Background** from the Qualitative Analysis menu.

In order to acquire a good quantification result, it is important to perform prior measurement of a standard sample whose elemental composition is well known.

☞ For details, refer to the separate instruction manual of the Standard Sample Analysis Program.

By using the off-line correction method, you can obtain a new quantitative-analysis correction result through changing the correction method or the standard sample. Therefore, you do not need to perform measurement again.

6.1.1 Starting and terminating Quantitative Analysis Program

1. Open the EPMA Main Menu on the computer display and then click on the **Analysis** icon to display the pull-down menu.
   ☞ Refer to the instruction manual of the microanalyzer main unit to learn how to open the EPMA Main Menu.
2. Select **Quantitative Analysis** from the **Analysis** menu in the JEOL EPMA Menu.

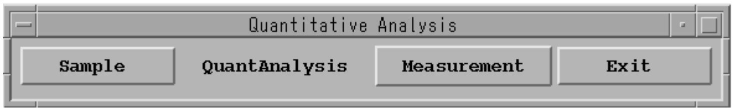


Fig. 1 Quantitative Analysis function window

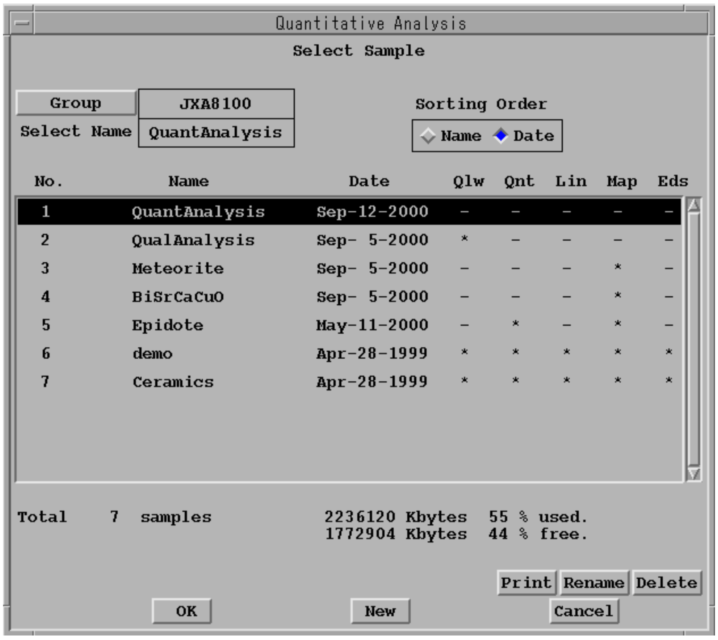
3. To terminate this program, click on the **Exit** button.

6.1.2 Specifying group name and sample name

Before starting measurement, it is necessary to decide the sample name in which data is to be stored. In the sample name, it is possible to store up to 10000 data. These data are managed with numbers. Data processing after measurement and data backup are executed for each sample. However, measurement data in the same sample name need not be subject to the same measurement conditions. Group name is used for controlling a sample group. If you assign the characteristics of a sample or operator name to the group name, you can later smoothly manage the sample group.

1. Click on the **Sample** button in the function window.

The Select Sample window opens.



Group: JXA8100
Select Name: QuantAnalysis

Sorting Order: ◇ Name ◆ Date

No.	Name	Date	Qlw	Qnt	Lin	Map	Eds
1	QuantAnalysis	Sep-12-2000	-	-	-	-	-
2	QualAnalysis	Sep- 5-2000	*	-	-	-	-
3	Meteorite	Sep- 5-2000	-	-	-	*	-
4	BiSrCaCuO	Sep- 5-2000	-	-	-	*	-
5	Epidote	May-11-2000	-	*	-	*	-
6	demo	Apr-28-1999	*	*	*	*	*
7	Ceramics	Apr-28-1999	*	*	*	*	*

Total 7 samples

2236120 Kbytes 55 % used.
1772904 Kbytes 44 % free.

Fig. 2 Select Sample window

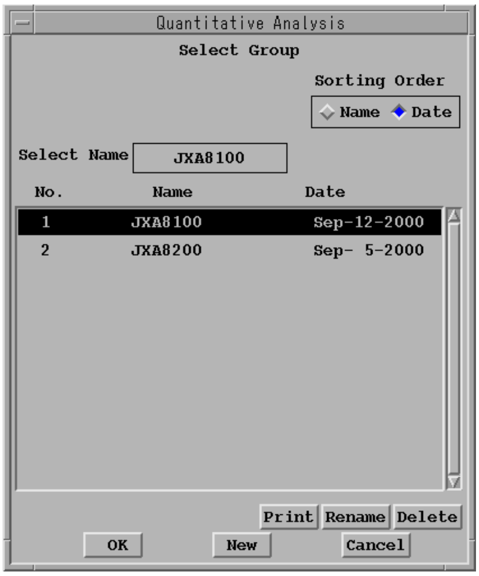
In this window, previously recorded sample names, measurement dates, and analysis methods applied to each sample are displayed.

✍ Here, analysis methods include the following: **Qlw**: qualitative analysis, **Qnt**: quantitative analysis, **Lin**: line analysis, **Map**: area analysis (mapping) and **Eds**: EDS analysis. Also, the hard disk space already used and the free space are displayed in kB.

If, before performing one of these analyses, you find that the hard disk space for storing new data is insufficient, back up the stored data using **File Utility** in **Utility** and delete the backed up data to make room.

2. Confirm the group name at the upper left of the Select Sample window.

To select an existing group name or a new one, click on the **Group** button to display the Select Group window, then select the desired group name or enter a new group name (using up to 14 characters) using the **New** button.



Select Name JXA8100

No.	Name	Date
1	JXA8100	Sep-12-2000
2	JXA8200	Sep- 5-2000

Fig. 3 Select Group window

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

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

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

Button	Function
OK	Click on the **OK** button in each window to finalize the Sample name and close the window. At this moment, if there are any measured data, a dialog box asking you “**Restore previous Conditions**?” appears. Then, if you want to continue measurements under the same measurement conditions, click on the **OK** button. If you want to perform measurements under different measurement conditions, click on the **Cancel** button.
Cancel	Click on the **Cancel** button in each window if you want to cancel the Sample name that was input and close the window.

6.1.3 Entering measurement conditions

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

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

The Measurement Conditions menu opens as shown in Fig. 4.

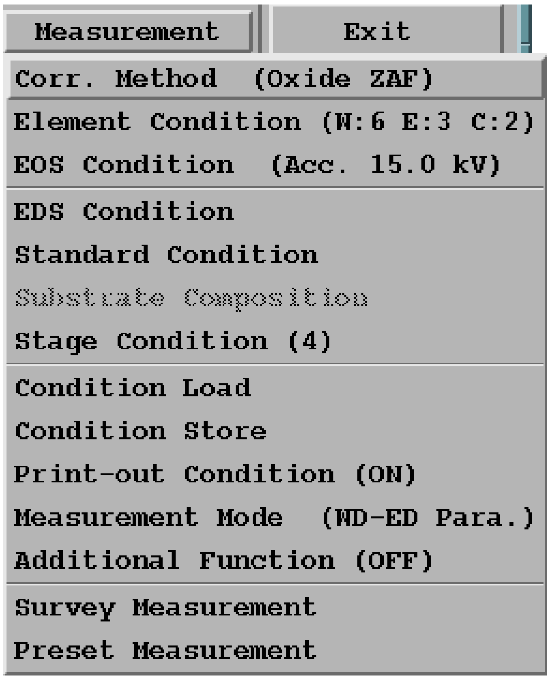


Fig. 4 Measurement Conditions menu

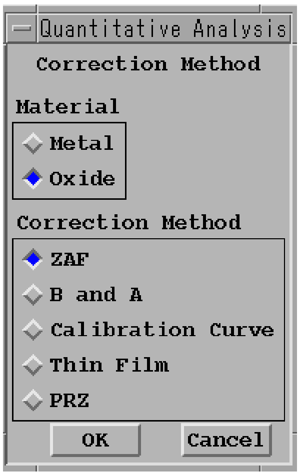
- When you first perform analysis using this program, it is recommended that you select each item in turn from Correction Method down to Stage Condition from the menu and enter appropriate information.
- When information has been already entered in this program, select the items that you want to modify and modify them.

✍ At the right of the menu items, basic settings are displayed so that you can see the outline of measurement conditions.

Correction Method

◆ Select **Measurement–Corr. Method** from the Quantitative Analysis function window.

The Correction Method window opens.



Correction Method

Material

○ Metal
● Oxide

Correction Method

● ZAF
○ B and A
○ Calibration Curve
○ Thin Film
○ PRZ

Fig. 5 Correction Method window

- **Material**

  By using the Correction Method window, select oxide material or metal material for substances to be analyzed, and select quantitative-analysis correction method. Here, in the correction method for oxide material, the quantity of oxide is automatically estimated from the valence. For metal material, this estimation is not executed.

- **Correction Method**

  Select the correction method from **ZAF**, **B and A** (Bence & Albee) (optional), **Calibration Curve**, **Thin Film** (optional), and **PRZ** (ϕ (ρz)) (optional).

✍ If you specify the Bence & Albee correction method, you can select only oxide materials.

Element Condition

◆ Select **Measurement–Element Condition** from the Quantitative Analysis function window.

The Element Condition window opens.

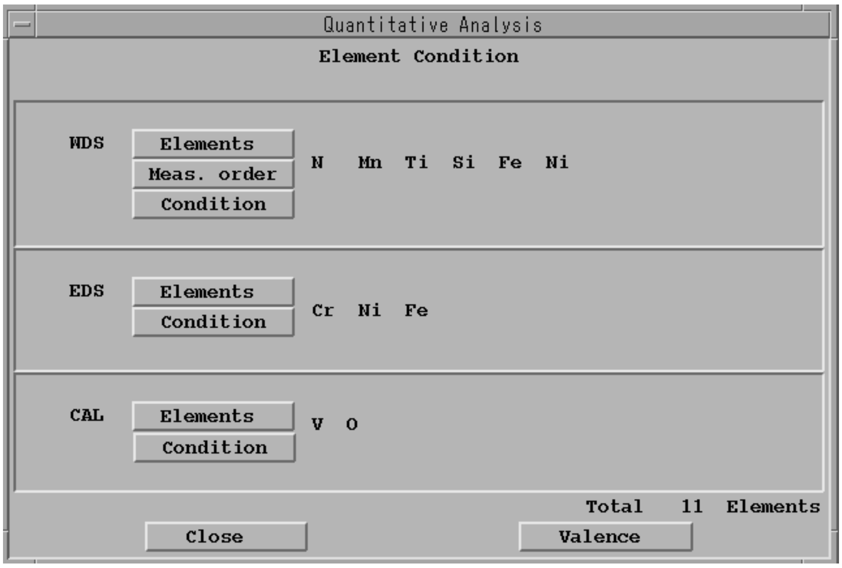


Fig. 6 Element Condition window

By using the Element Condition window, specify elements that are to be analyzed in **WDS** and **EDS** and elements whose concentration is to be calculated in **CAL –Elements**.

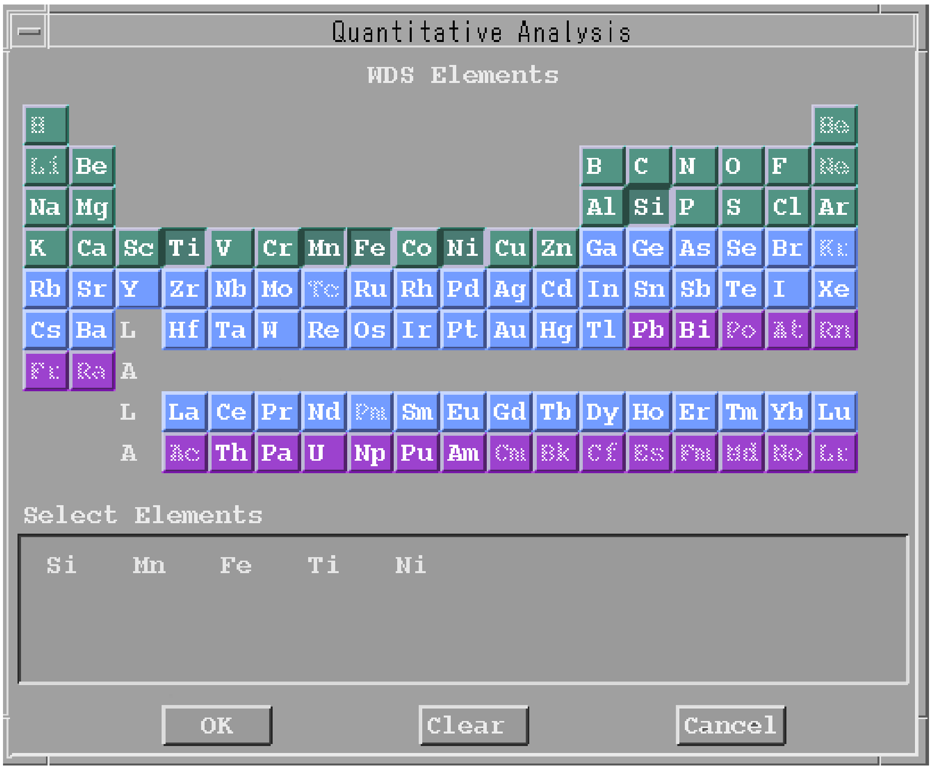
✍ When the same element is specified in WDS and EDS, measurement data of an element subject to WDS are used in correction.

✍ When the same elements are multiply specified in WDS, the average value of these measurement data is used in correction.

● Specifying elements for WDS

◆ Select **WDS–Elements** from the Element Condition window.

The WDS Elements window displaying the periodic table opens.



Si Mn Fe Ti Ni

Fig. 7 WDS Elements window

- When you select elements from the periodic table, their symbols are displayed in the **Select Elements** box under the periodic table.
- Selecting the same element again records it multiple times.
- To delete a selected element, click on the label of the selected element to highlight it and click on **Clear**.
- To delete selected multiple elements at the same time, drag the mouse to highlight the multiple elements and click on **Clear**.

Ordinarily, 76 elements can be selected from the WDS Elements window. The elements from Be to U, excluding the rare gases and radioactive elements, can be selected. Elements that cannot be selected are displayed dimmed. Each element that cannot be selected is listed, one line for each atomic number, in the file **/opt/epma/phys/elemprot.asc**. By adding or deleting atomic numbers in the file using the editor, you can modify the list of elements.

When the same element is selected multiple times, the average value of the multiple measurement data of the elements is used in correction calculation. This average value is used to contribute to increasing the count of X-rays if the same crystals exist in multiple channels and also these channels are not used in measurement of other elements.

● Performing WD/ED combined quantitative analysis in the JXA-8200 series EPMA

◆ Select **EDS–Elements** from the Element Condition window.

The EDS Elements window displaying the periodic table opens.

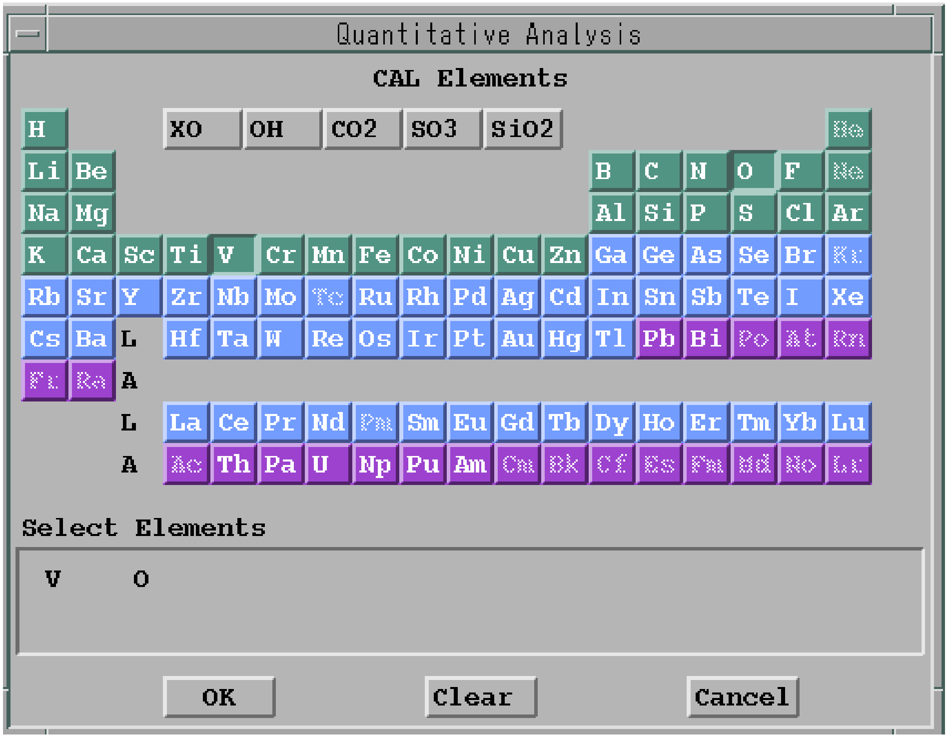
- Enter the elements to be measured in EDS in the EDS Elements window.
  The method for entering elements is the same as in WDS.

✍ In EDS, the reference spectrum is used in processing waveform data. At this time, processing is automatically executed by using the information from measured elements that are specified in WDS as a reference. Therefore, when you perform EDS analysis, you do not need to doubly record the measured elements that are already recorded in WDS. If the elements are doubly recorded in both EDS and WDS, the K-ratio of the elements recorded in WDS is used in the correction calculation, and the K-ratio of the elements recorded in EDS is neglected for the calculation.

● Determining the quantity of an element indirectly by using calculation without directly measuring it

◆ Select **CAL–Elements** from the Element Condition window.

The CAL Elements window displaying the periodic table opens.



Select Elements

V O

Fig. 8 CAL Elements (periodic table) window

Specify an element for calculation in the CAL Element Window. The method for setting it is the same as that in WDS. However, you cannot doubly record elements, which are recorded in WDS or EDS, in the CAL Elements. If you do, a warning window is displayed when you attempt to close the Element Condition window.

When you perform measurement in the oxide material mode, it is possible to specify the following in addition to ordinary elements.

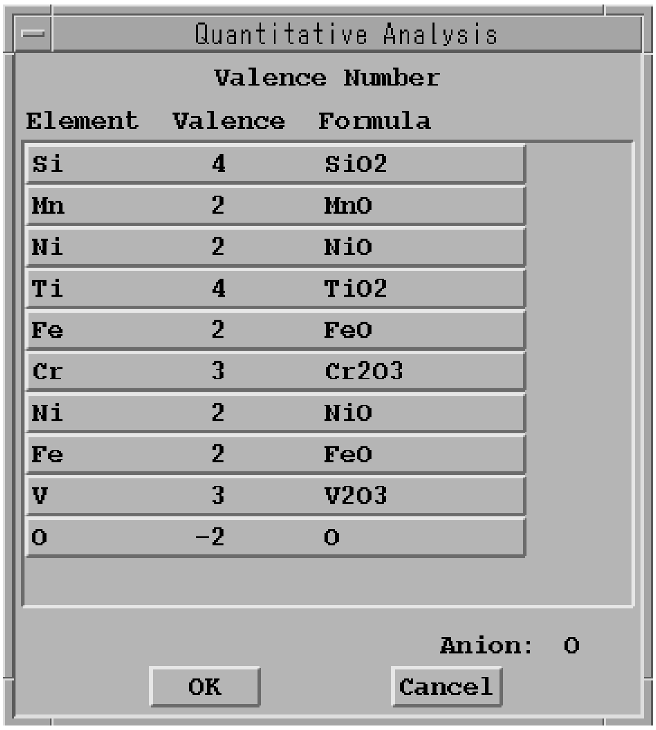
Button	Function
XO	Specify it when excess oxygen exists in the Bence & Albee correction method.
OH	Specify it when hydroxyl groups exist.
CO2	Specify it when you perform analysis of carbonates without measuring C.
SO3	Specify it when you perform analysis of hydrosulfates without measuring S.
SiO2	Specify it when you perform analysis of silicates without measuring Si.

✍ When the oxide material mode is specified, oxygen (O) is automatically recorded as CAL element, and the calculation mode for it becomes Anion.

● Setting valence

◆ Click on the **Valence** button in the Element Condition window.

The Valence Number window opens.



Quantitative Analysis

Valence Number

Element	Valence	Formula
Si	4	SiO2
Mn	2	MnO
Ni	2	NiO
Ti	4	TiO2
Fe	2	FeO
Cr	3	Cr2O3
Ni	2	NiO
Fe	2	FeO
V	3	V2O3
O	-2	O

Anion: 0

Fig. 9 Valence Number window

When you perform measurement in the oxide material mode, you can change valence. In the Valence Number window, **Element**, **Valence** and **Formula** (chemical formula of oxide material) are displayed.

When you want to change valence, select an element and enter the numerical value of the valence you want to change.
When you do not want to treat the target element in the form of oxide material, such as when measuring Cl and I, enter zero or a negative value as the valence.

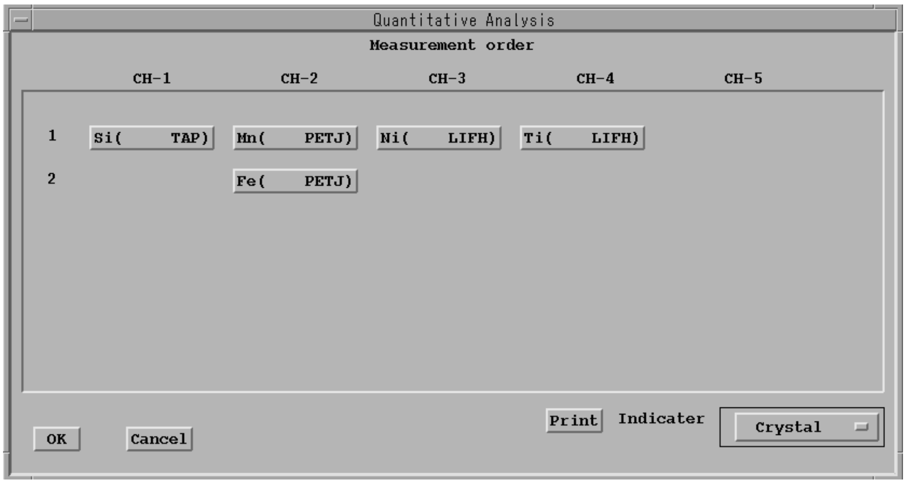
✍ The default value of each element is listed in the file **/opt/epma/phys/ atom.asc**.

● Setting measurement order

You select a channel to be used and the measurement order for each element. If you make the selection so that measurements are not concentrated in any one specific channel, you can shorten the entire measurement time.

◆ Select **WDS–Meas. order** from the Element Condition window.

The Measurement order window opens.



	CH-1	CH-2	CH-3	CH-4	CH-5
1	Si(TAP)	Mn(PETJ)	Ni(LIFH)	Ti(LIFH)	
2		Fe(PETJ)			

Indicater: Crystal

Fig. 10 Measurement order window

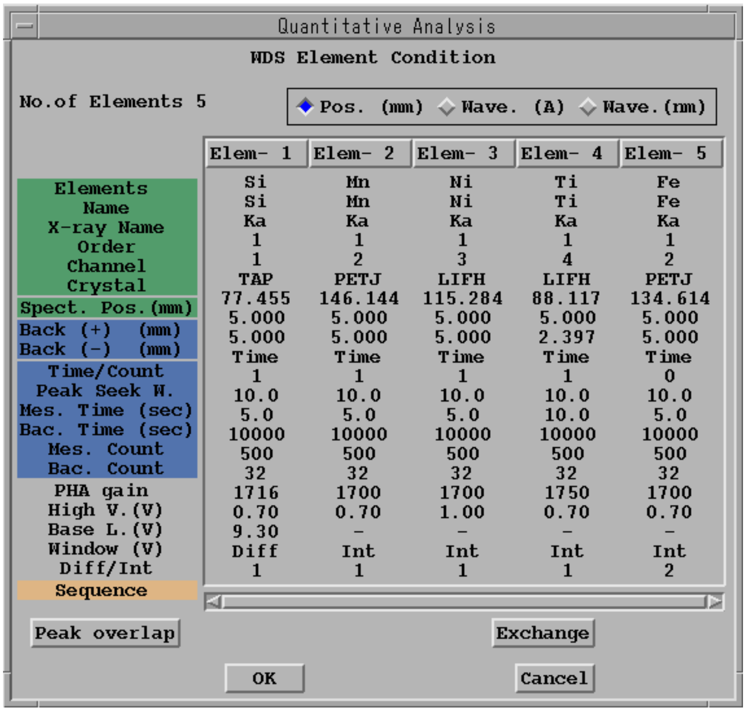
Drag an element label using the mouse to set the measurement order. Dragging up and down changes the measurement order and dragging left and right changes the channel number. If the measured X-rays are out of range of spectrometric analysis, a mouse cursor turns to a cross and you cannot select the channel for analyzing the X-rays.

Ordinarily, element name and X-ray analyzing crystal name are displayed in the element label. By selecting **Peak Pos.** for **Indicator** in the Measurement order window, it is possible to display wavelength instead of the crystal name. Doing this allows you to arrange elements in wavelength order. However, since you cannot change X-rays and analyzing crystals in this window, change them using the WDS Element Condition window (described later) and set the measurement order there.

● WDS Element Condition

◆ Select **WDS–Condition** from the Element Condition window.

The WDS Element Condition window opens.



Quantitative Analysis

WDS Element Condition

No.of Elements 5

◆ Pos. (mm) ◇ Wave. (A) ◇ Wave.(nm)

	Elem- 1	Elem- 2	Elem- 3	Elem- 4	Elem- 5
Elements	Si	Mn	Ni	Ti	Fe
Name	Si	Mn	Ni	Ti	Fe
X-ray Name	Ka	Ka	Ka	Ka	Ka
Order	1	1	1	1	1
Channel	1	2	3	4	2
Crystal	TAP	PETJ	LIFH	LIFH	PETJ
Spect. Pos.(mm)	77.455	146.144	115.284	88.117	134.614
Back (+) (mm)	5.000	5.000	5.000	5.000	5.000
Back (-) (mm)	5.000	5.000	5.000	2.397	5.000
Time/Count	Time	Time	Time	Time	Time
Peak Seek W.	1	1	1	1	0
Mes. Time (sec)	10.0	10.0	10.0	10.0	10.0
Bac. Time (sec)	5.0	5.0	5.0	10.0	5.0
Mes. Count	10000	10000	10000	10000	10000
Bac. Count	500	500	500	500	500
PHA gain	32	32	32	32	32
High V.(V)	1716	1700	1700	1750	1700
Base L.(V)	0.70	0.70	1.00	0.70	0.70
Window (V)	9.30	–	–	–	–
Diff/Int	Diff	Int	Int	Int	Int
Sequence	1	1	1	1	2

Fig. 11 WDS Element Condition window

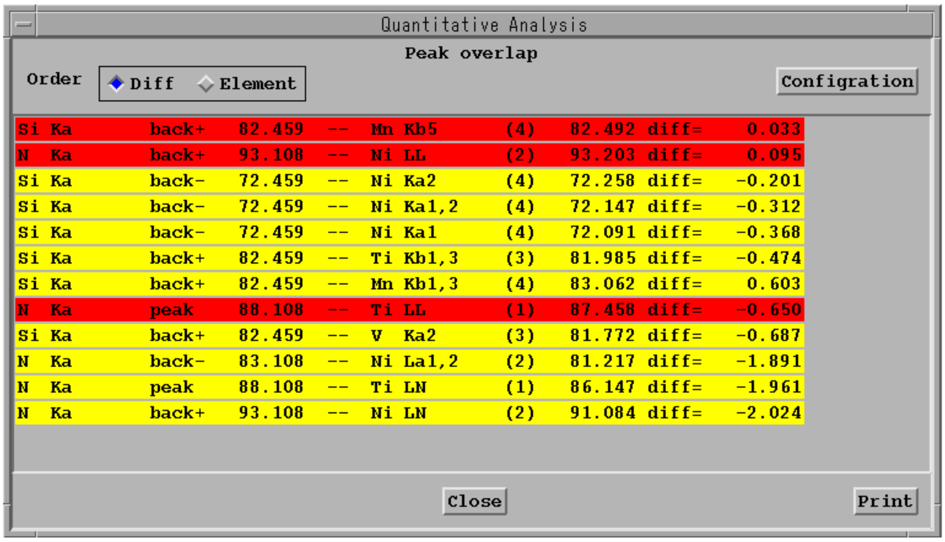
The measurement conditions of elements that are measured by WDS are displayed in the list in the WDS Element Condition window. To modify the measurement conditions, selecting **Elem-1**, **2**, **3** …, will display the WDS Element Data Table window (described later) where you can modify them.

This window has the following functions.

Button	Function
Wavelength display unit	Select the unit for displaying wavelength from mm, angstrom and nm.
Exchange	Changes the order in which the measurement conditions are displayed.
Peak overlap	Displays adjacent X-rays.

◆ Click on **Peak overlap** button in the WDS Element Condition window.

The Peak overlap window opens.



```
Si Ka      back+  82.459  --  Mn Kb5    (4)  82.492 diff=   0.033
N  Ka      back+  93.108  --  Ni LL     (2)  93.203 diff=   0.095
Si Ka      back-  72.459  --  Ni Ka2    (4)  72.258 diff=  -0.201
Si Ka      back-  72.459  --  Ni Ka1,2  (4)  72.147 diff=  -0.312
Si Ka      back-  72.459  --  Ni Ka1    (4)  72.091 diff=  -0.368
Si Ka      back+  82.459  --  Ti Kb1,3  (3)  81.985 diff=  -0.474
Si Ka      back+  82.459  --  Mn Kb1,3  (4)  83.062 diff=   0.603
N  Ka      peak   88.108  --  Ti LL     (1)  87.458 diff=  -0.650
Si Ka      back+  82.459  --  V  Ka2    (3)  81.772 diff=  -0.687
N  Ka      back-  83.108  --  Ni La1,2  (2)  81.217 diff=  -1.891
N  Ka      peak   88.108  --  Ti LN     (1)  86.147 diff=  -1.961
N  Ka      back+  93.108  --  Ni LN     (2)  91.084 diff=  -2.024
```

Fig. 12 Peak overlap window

In this window, the adjacent X-rays that interfere with the measured X-rays are displayed. The peak positions and background signal positions of all measured elements are compared with the wavelengths of all X-rays being generated from other elements in the sample. Then, the interfering X-rays are displayed in a list when they are judged to be in the vicinity of the measured X-rays.

In the Peak overlap window, one set of data is displayed from left to right in the following order: element name, measured X-ray name, peak or background signal, wavelength, interfering element name, interfering X-ray name, its order, interfering X-ray wavelength and difference of wavelength.

By default, data is displayed in ascending order according to the difference of wavelength. When you select **Element** for **Order**, the display changes to the element order.

When the difference of wavelength falls within the range of the full with at half maximum (FWHM) of the analyzing crystal, this data is displayed in red. When the difference corresponds to three times the FWHM or below, it is displayed in yellow.

You can set the criteria for judging these data and the maximum X-ray order to be checked using the **Configuration** menu.

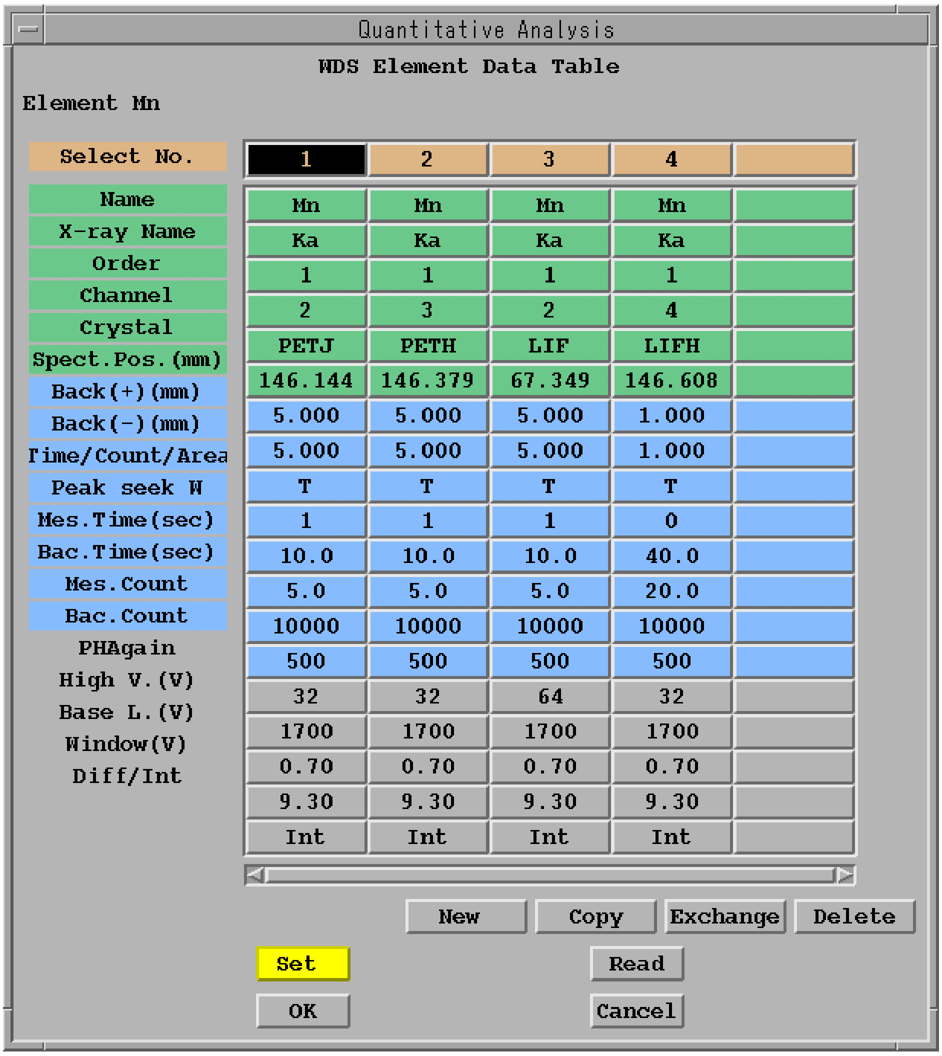
✍ You can set more precise measurement conditions by using these data and the functions of **Quant. Background**.

✍ In most cases, the interfering X-rays are minor X-rays or high-order X-rays; therefore, the interfering X-rays mostly do not cause problems. However, in the case of some element combination (for example, Ti Ka – V Kb, N Ka – Ti LL), the influence of the interfering X-rays cannot be neglected. In such a case, it is possible to correct this influence using **Overlap correction** in the **Additional Function**.

WDS Element Data Table

◆ Select **Elem-1**, **2**, **3** …, from the WDS Element Condition window.

The WDS Element Data Table window opens.



WDS Element Data Table

Element Mn

Select No.	1	2	3	4	
Name	Mn	Mn	Mn	Mn	
X-ray Name	Ka	Ka	Ka	Ka	
Order	1	1	1	1	
Channel	2	3	2	4	
Crystal	PETJ	PETH	LIF	LIFH	
Spect.Pos.(mm)	146.144	146.379	67.349	146.608	
Back(+)(mm)	5.000	5.000	5.000	1.000	
Back(-)(mm)	5.000	5.000	5.000	1.000	
Time/Count/Area	T	T	T	T	
Peak seek W	1	1	1	0	
Mes.Time(sec)	10.0	10.0	10.0	40.0	
Bac.Time(sec)	5.0	5.0	5.0	20.0	
Mes.Count	10000	10000	10000	10000	
Bac.Count	500	500	500	500	
PHAgain	32	32	64	32	
High V.(V)	1700	1700	1700	1700	
Base L.(V)	0.70	0.70	0.70	0.70	
Window(V)	9.30	9.30	9.30	9.30	
Diff/Int	Int	Int	Int	Int	

Fig. 13 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. These conditions are used in combination with analysis of the standard sample, line analysis and area analysis. 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.
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 conditions mentioned above are loaded from the EPMA basic unit. Use this button when you want to check the element conditions on the monitor screen and change the element table to reflect these conditions.
Copy	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.
Up data SCA	Sets the SCA conditions to the values of the hardware configuration.
OK	Finalizes the element conditions and changes the WDS Element Condition to reflect the element conditions of Select No.
Cancel	The element conditions are erased.

✍ Before 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 SCA conditions using the SCA Monitor.

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

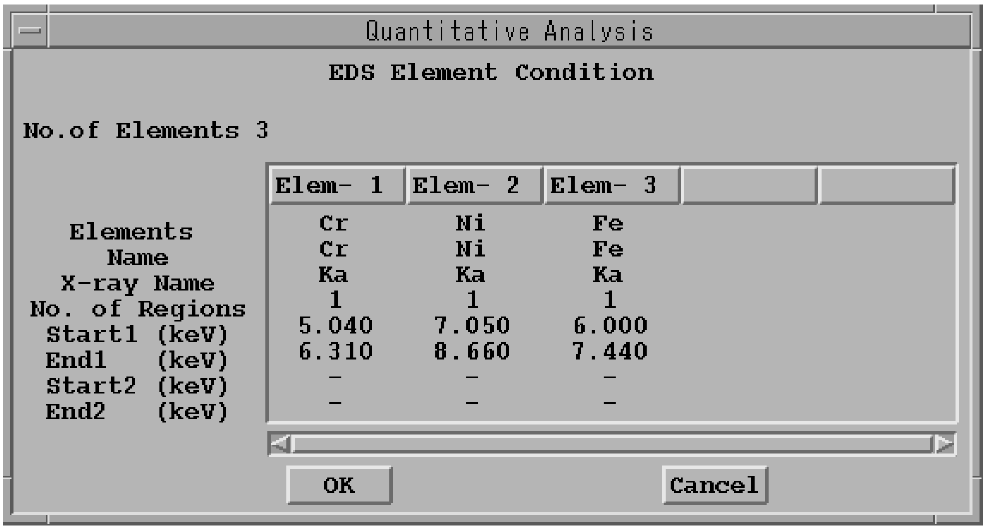
Button	Function
Name	Give any name by inputting up to 8 characters. The element name is shown as the default.
X-ray Name	Select an X-ray name from Ka, Kb, La, Lb, Ma, and Mb, where “a” and “b” mean “α” and “β” respectively.
Order	Select the order of X-rays from primary to tenth. Usually, use the primary X-rays. If you cannot use the primary X-rays by reason of interfering rays, select secondary X-rays.
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 to use are dimmed.

Button	Function
Spect. Pos.	Specify the position of the spectrometer. The default is the values calibrated to the theoretical position of the X-ray spectrometer. After the measurement of the standard sample, the position of the spectrometer corresponding to the standard sample is displayed. If you select **Save** by using the Peak Search Monitor, the peak position is displayed.
Back (+)	Specify the background offset on the higher angles. If you specify zero, the background measurement is not executed.
Back (–)	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. Fixed-time measurement is selected as a default. Selecting fixed-time measurement performs the peak/background measurement during the specified time. Selecting fixed-count measurement performs the measurement until you obtain the specified number of counts, but the measurement stops if the designated time is finished before the specified number of counts is reached. If you measure low intensity X-rays by the fixed-count method, specify an appropriately longer time. Furthermore, in the window, you can select the area method, which will be explained later.
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 the 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 for 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.

✍ The default values of the SCA parameters are those that were set in the hardware configuration. To set them more in detail, use the SCA monitor to select the SCA parameters at the peak position for loading.

EDS Element Condition

With the JXA-8200 Series EPMA, if you have specified element names in **EDS–Elements** in the Element Condition window, click on **EDS–Condition,** and then the EDS Element Condition window will open as shown in Fig. 14. In this window, you select measurement conditions for each element. However, since the ROIs for the standard sample are used as the ROIs for the EDS measurement, you cannot enter values using this EDS Element Condition window. You can enter only element names and X-ray names.



Quantitative Analysis

EDS Element Condition

No.of Elements 3

	Elem- 1	Elem- 2	Elem- 3		
Elements	Cr	Ni	Fe		
Name	Cr	Ni	Fe		
X-ray Name	Ka	Ka	Ka		
No. of Regions	1	1	1		
Start1 (keV)	5.040	7.050	6.000		
End1 (keV)	6.310	8.660	7.440		
Start2 (keV)	-	-	-		
End2 (keV)	-	-	-		

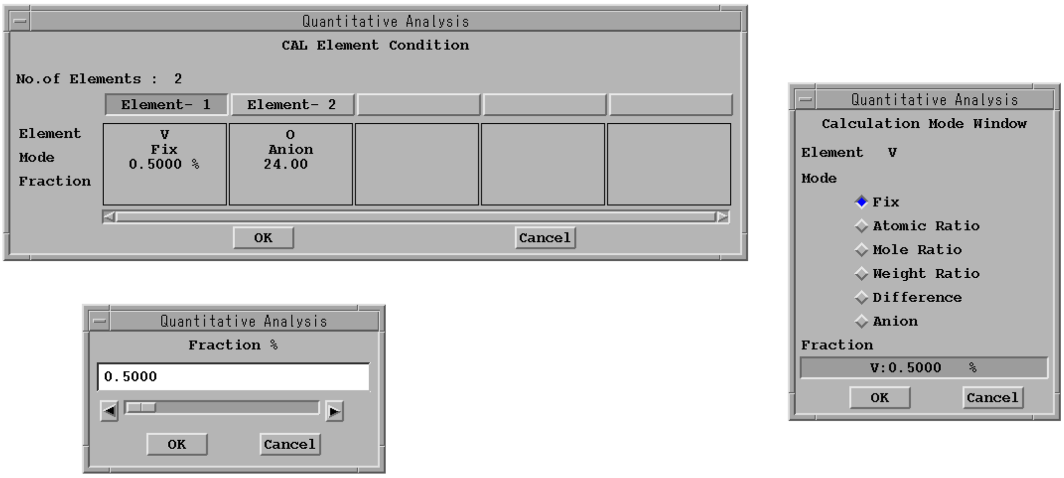
Fig. 14 EDS Element Condition window

CAL Element Condition

If you have specified element names in **CAL–Elements** in the Element Condition window, click on **CAL–Condition,** and then the CAL Element Condition window will open as shown in Fig. 15. In this window, you can specify the element calculation mode that you want to use. Select one of the six modes of element calculation: **Fix**, **Atomic Ratio**, **Mole Ratio**, **Mass Ratio**, **Difference**, and **Anion**. The **Mole Ratio** and **Anion** modes are used only for quantitative analysis of oxides.

Button	Function
Fix	If you know the concentration in advance, enter the value of mass percent in the Fraction % window for data processing.
Atomic Ratio	Input the atomic ratio of the specified element to the measured elements for data processing.
Mole Ratio	Input the molar ratio of the specified element to the measured elements for data processing. It can be applied only to oxide analysis.
Mass Ratio	Input the mass concentration ratio of the specified element to the measured elements for data processing.
Difference	Input the difference for data processing.
Anion	Input the valence for data processing.

Usually, the Mole Ratio and Anion are to be used for the quantitative analysis of oxides. In the quantitative analysis of metals, if you assign an element to Anion, the concentration of the desired element will be calculated from the valence ratio to the other elements.



No.of Elements : 2

Element	Element- 1	Element- 2
Element	V	O
Mode	Fix	Anion
Fraction	0.5000 %	24.00

Fraction %: 0.5000

Element V

Mode: ◆ Fix

Fraction: V:0.5000 %

Fig. 15 CAL Element Condition, Calculation Mode, and Fraction % windows

● Reference

◆ Special input method for the CAL elements

When the analysis mode is for the quantitative analysis of oxides, special elements, such as XO, OH, CO_2, SO_3, and SiO_2, can be used as the CAL elements.

Example

The chemical formula of carbonate is (Ca, Mg, Fe) CO_3. Decomposing this formula to (Ca, Mg, Fe) OCO_2, we can estimate the concentration from the chemical formula without direct quantification of CO_2.

The procedure is as follows:

Click on the **CO2** button in the CAL Elements window. Then, select **Mole Ratio** (or **Atomic Ratio**) from the Calculation Mode Window.

Click on the **Fraction** button, and enter * (asterisk) in the **Elem** input box, and the number of CO_2 per the chemical formula in the **Fraction** input box. This number can be determined from the value indicated on **No. of Oxygen** on Print-out Condition.

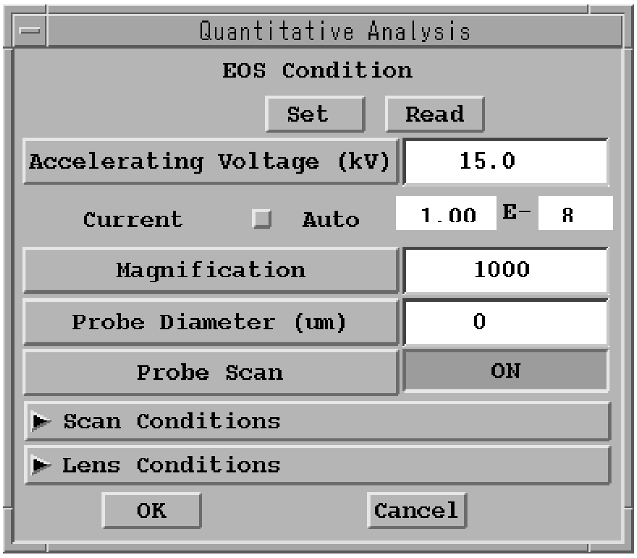
For carbonate, the chemical formula is (Ca, Mg, Fe) OCO_2. So, if O is 24, enter 24 in the Fraction input box.

EOS Condition

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

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

The EOS Condition window opens as shown in Fig. 16.



Accelerating Voltage (kV) 15.0

Current ☐ Auto 1.00 E- 8

Magnification 1000

Probe Diameter (um) 0

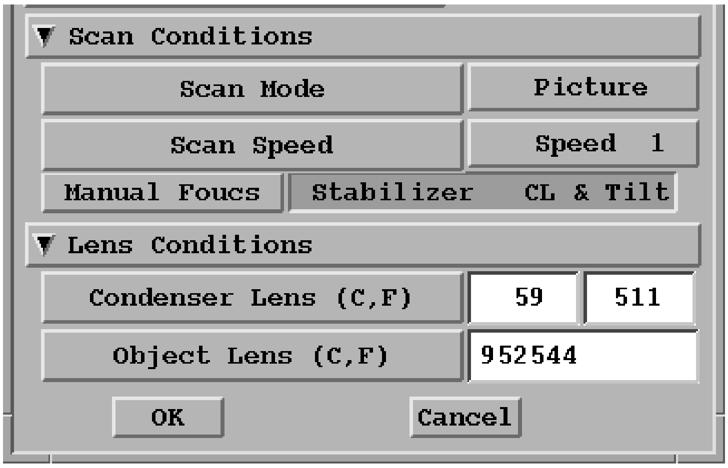
Probe Scan ON

Fig. 16 EOS Condition window

The EOS Condition window has the following objects.

Object	Function
Set	Sets the EOS to the present measurement conditions.
Read	Reads the present EOS conditions, and displays them in the EOS Condition window.
Accelerating Voltage	Sets the accelerating voltage (in kV).
Current	Displays the beam current. Click on the **Auto** button to specify the current; the specified current will be set automatically before measuring, and the automatic current-setting mode will be applied.
Magnification	Sets the scan magnification for measurement. This function is in effect only when the **Probe Scan** is **ON**. When you perform measurement at a very low magnification, the WDS elements will be out of the X-ray collecting conditions, and you cannot obtain good results.
Probe Diameter	Sets the probe diameter (in μm) for measurement. This function is in effect only when the **Probe Scan** is **OFF**.
Probe Scan	Turns the probe scan on or off for 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**.

Object	Function
Scan Mode	Specifies the scan mode for measurement. The choices are **Picture**, **Bup**, **Line**, **Spot** and **Area**.
Scan Speed	Selects the scan speed for measurement from S1 to S12. The larger the number is, the slower the speed is.
Auto Focus	Select automatic focusing (**Auto Focus**) or manual focusing (**Manual Focus**) before measurement.
Stabilizer	Specify whether to use the beam stabilizer (select from **CL & Tilt**, **CL**, and **Tilt**) or not (**Off**).
Lens Conditions	Clicking on the arrowhead of this button opens a pop-up menu for the following two items.
Condenser Lens	Sets the Condenser Lens to Coarse or Fine for measurement.
Object Lens	Sets the value of the Objective Lens for measurement.
OK	Finalizes the input EOS conditions, and closes the EOS Condition window.
Cancel	Cancels the input EOS conditions, and closes the EOS Condition window.


▼ Scan Conditions

Scan Mode: Picture

Scan Speed: Speed 1

Manual Foucs

Stabilizer: CL & Tilt

▼ Lens Conditions

Condenser Lens (C,F): 59 511

Object Lens (C,F): 952544

Fig. 17 EOS Condition pop-up menu

For the usual quantitative analysis using the WDS, set the probe current in the range of 1 to 5 × 10^{-8}A. For a sample sensitive to beam damage, it is recommended to make the beam diameter larger in analysis.

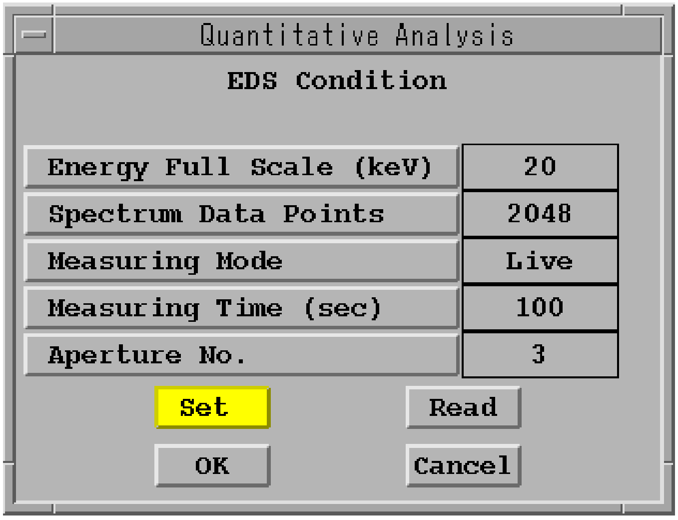
However, when you analyze elements by using both WDS and EDS spectrometers in the JXA-8200 Series, the intensity of X-rays is too high for the EDS. Therefore, you need to adjust the aperture of the EDS detector to reduce the intensity of X-rays that enter the detector. When you analyze samples by using the EDS only, fully open the aperture of the EDS detector and decrease the probe current to the range of 1 to 10 × 10^{-10}A. This minimizes the damage to the sample by electron beam irradiation during analysis.

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 Quantitative Analysis function window.

The EDS Condition window opens as shown in Fig. 18.



Energy Full Scale (keV)	20
Spectrum Data Points	2048
Measuring Mode	Live
Measuring Time (sec)	100
Aperture No.	3

Fig. 18 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	Is set to 2 K channels.
Measuring Mode	Selects **Live** Time or **Real** Time for the measurement mode.
Measuring Time	Specifies the measuring time in seconds.
Aperture No.	Selects the aperture number from 0 to 5. The value 0 is for open. The larger the number, the smaller the aperture diameter. The value 6 is for closed, and so it cannot be used for measurement.

To select an aperture number, first set the beam current, and then select the appropriate aperture number so that the Dead Time becomes 20 – 30%, while actually collecting EDS spectra.

Select Live and several tens of seconds for **Measuring Mode** and **Measuring Time** respectively.

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

Clicking on the **Read** button reads the EDS spectrum collection conditions that are presently set and displayed in the EDS Condition window.

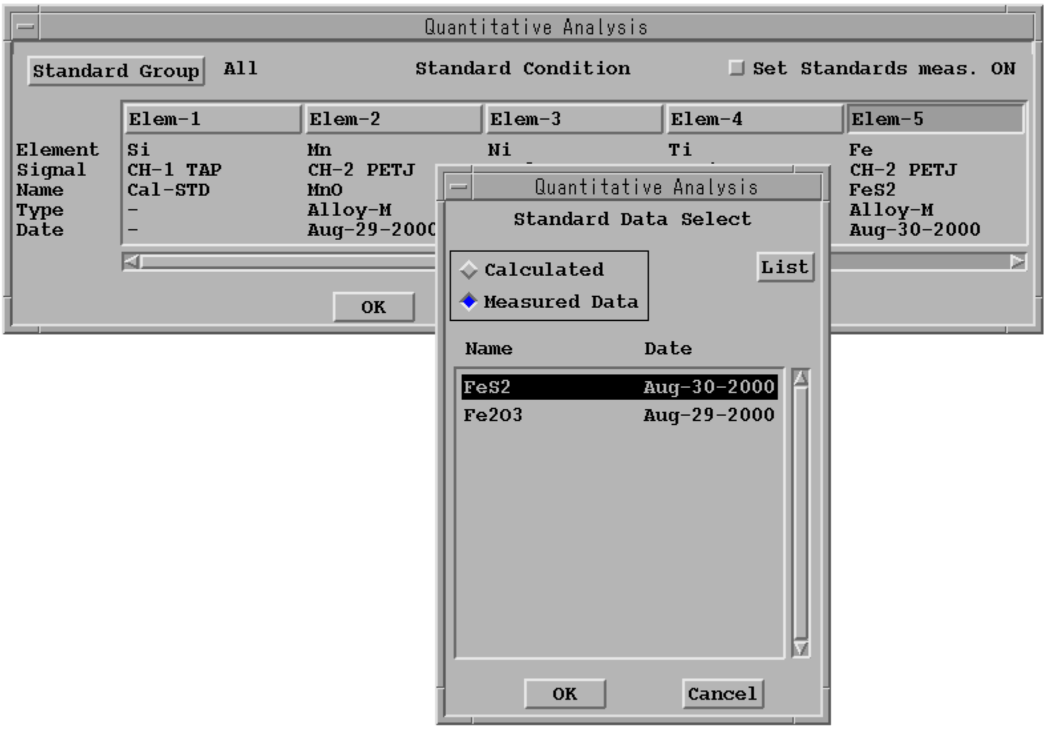
Note that the **Set** and **Read** buttons can be used only when the EDS Home Window is on the screen.

Standard Condition

The Standard Condition window allows you to specify standard specimen names required for correction calculation.

◆ Select **Measurement–Standard Condition** from the Quantitative Analysis function window.

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



	Elem-1	Elem-2	Elem-3	Elem-4	Elem-5
Element	Si	Mn	Ni	Ti	Fe
Signal	CH-1 TAP	CH-2 PETJ			CH-2 PETJ
Name	Cal-STD	MnO			FeS2
Type	-	Alloy-M			Alloy-M
Date	-	Aug-29-200			Aug-30-2000

Standard Data Select

○ Calculated
● Measured Data

Name	Date
FeS2	Aug-30-2000
Fe2O3	Aug-29-2000

Fig. 19 Standard Condition window

This window displays the group name of the presently selected standard samples (☞ for details, refer to the separate instruction manual of the Standard Sample Analysis Program), the names of the standard samples belonging to the selected group, the types of the standard samples (metal, oxide, or calibration curve), and the dates of measurements.

The names of standard samples will be selected automatically, if you specify an element in the Element Condition window. In the selection, the standard sample data collected under the same measurement conditions will be searched. The same measurement conditions signify element, X-rays, order, accelerating voltage, channel and analyzing crystal in the case of WDS; and element, X-rays, accelerating voltage and energy full-scale in the case of EDS. If there are multiple data, the priority of selection will be given to the latest measurement datum of the standard sample.

If standard sample data have not been measured for elements, the calculated data (CAL–STD mode) will be used as standard sample data. The calculated data will be the same as for the semi-quantitative analysis.

To change the selected standard samples, click on the **Elem-1**, **2**, **3**……buttons, and then the Standard Data Select window opens. In this window, you can select the desired standard samples. Selecting **Calculated** means that you have selected CAL–STD. If you do not find the elements that you want to use in this window, click on the **Search** button.

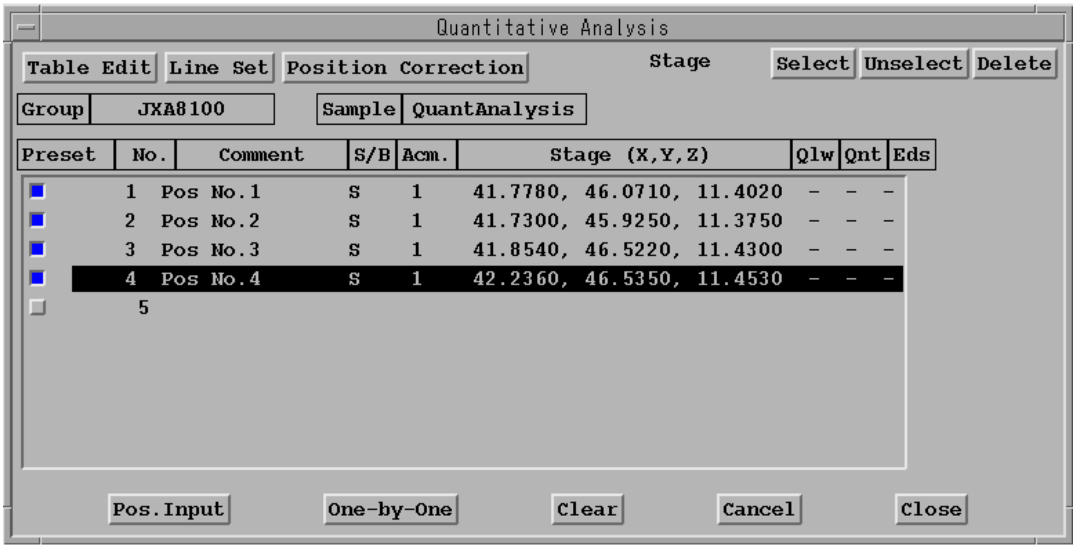
The data under any measurement conditions of all the standard samples including the elements to be measured will be displayed, and then you can easily search the standard samples already measured with different channels.
When you want to measure again the standard specimens that you selected in the quantitative analysis program, click on the **Set Standards Meas. ON** button; then the desired standard specimens will be selected in the Preset window of the Standard Sample Analysis Program.

Stage Condition

The Stage Condition window allows you to specify analysis points before measurement.

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

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



Group JXA8100

Sample QuantAnalysis

Preset	No.	Comment	S/B	Acm.	Stage (X,Y,Z)	Qlw	Qnt	Eds
☑	1	Pos No.1	S	1	41.7780, 46.0710, 11.4020	-	-	-
☑	2	Pos No.2	S	1	41.7300, 45.9250, 11.3750	-	-	-
☑	3	Pos No.3	S	1	41.8540, 46.5220, 11.4300	-	-	-
☑	4	Pos No.4	S	1	42.2360, 46.5350, 11.4530	-	-	-
☐	5							

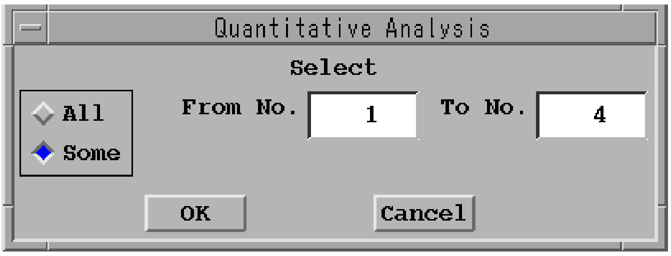
Fig. 20 Stage Condition window

You have to specify analysis points before you begin to perform measurement. To specify analysis points, there are two modes. One is the Stage mode in which you control the stage position, and the other is the Beam mode in which you specify the beam position.
When you open the Stage Condition window, the coordinates of the recorded analysis positions are displayed in it. The same list of coordinates is used in common by the quantitative analysis, qualitative analysis, and EDS qualitative analysis. Consequently, even when you perform the quantitative analysis for the first time, coordinates will already be in the list if you have previously performed other analyses.
However, using the same analysis points does not lead to any confusion, since the results of each analysis are recorded separately.
The list shows the **Preset** buttons that specify whether analysis is to be performed or not, coordinates number (**No.**), **Comment**, Stage/Beam mode (**S/B**) Accumulation (**Acm.**), **Stage (X, Y, Z)**, the kinds of analysis that have been executed (**Qlw**: qualitative analysis, **Qnt**: quantitative analysis, **Eds**: EDS analysis marked with asterisks, if already executed).
When this window is opened, a line is always highlighted, and it allows you both to enter values of coordinates in **Pos. Input**, and to perform **One-by One** analysis.

✍ If the optional Auto-focus Device is installed in the instrument, and **Auto Focus** is selected, the **A** mark will be labeled next to **Acm.** number.

✍ If you want to record a new analysis point, select the first unassigned line under the last recorded line of the list. If you want to alter a recorded analysis point, select the desired line from the list and execute **Pos. Input**. Select **One-by-One** for one-by-one analysis.

Button	Function
Pos. Input	Specifies analysis position. Clicking on this button displays the Stage Condition Input window, which will be explained later, and enables you to specify analisis position.
One-by One	Performs one point analysis of the highlighted position.
Clear	Clears the highlighted analysis point from the list. If there are other analysis points under the cleared point, they will advance upward by one line.
Cancel	Cancels the inputs, and does not enter them in the stage file. In the confirmation window that opens, select **OK** if you want to cancel the inputs.
Select/Unselect	To measure the position of the input coordinates in the Preset mode, which will be explained later, the **Preset** button should be selected in advance. Selecting **Select** turns **Preset** on for all or some of the coordinates. In the same way, selecting **Unselect** turns them off.



Quantitative Analysis

Select

○ All
● Some

From No. 1

To No. 4

Fig. 21 Select window

Button	Function
Delete	Deletes all or some of the input coordinates. If there are other analysis points under the deleted point, they will advance upward by one line.

✍ When you delete analysis points by using **Clear** or **Delete**, other analysis points under the deleted point will advance upward by one line, and then they will be recorded at the new positions. However, if analysis has been already executed at an analysis point, the result will remain without changing the position.

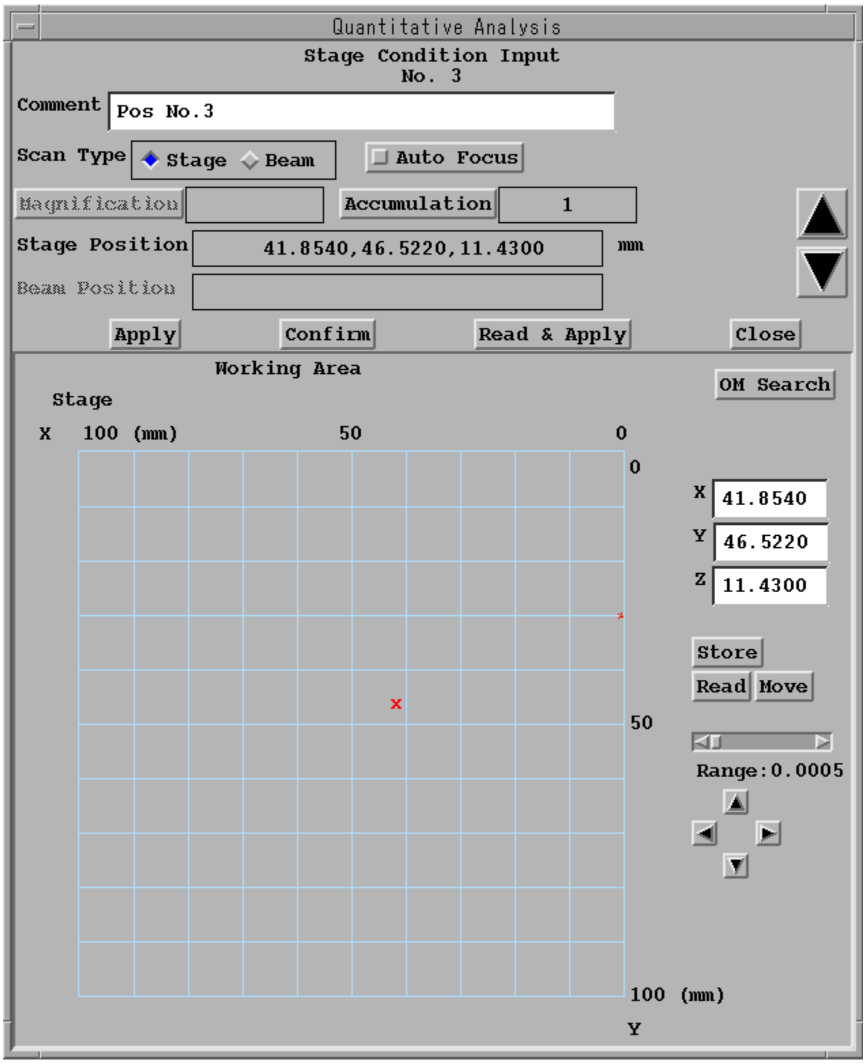
✍ The Stage Condition window has other functions such as **Table Edit**, **Line Set**, and **Position Correction** for your selection. These functions will be explained later.

● 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. 22.



No. 3

Comment: Pos No.3

Scan Type: ◆ Stage ◇ Beam

☐ Auto Focus

Accumulation: 1

Stage Position: 41.8540,46.5220,11.4300 mm

X: 41.8540

Y: 46.5220

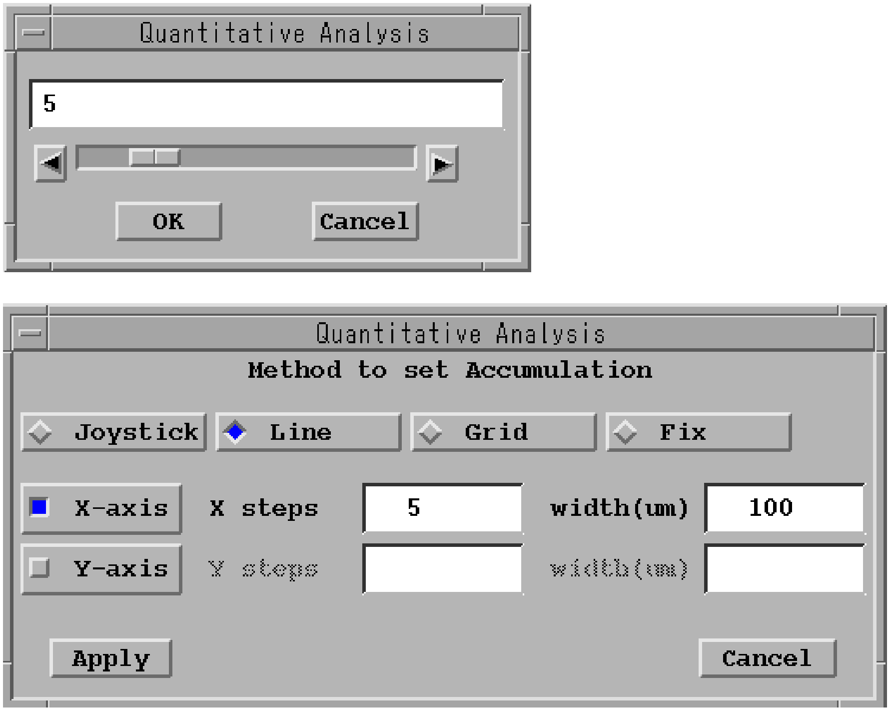
Z: 11.4300

Range:0.0005

Fig. 22 Stage Condition Input window with Working Area (Stage)

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

Button	Function
Comment	Lets you input a comment of up to 40 characters for the sample.
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 times to accumulate (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). When **Line** or **Grid** is selected, enter the number of steps and the width of scans (in μm). After specifying Accumulation, confirm each coordinate by using the following **Confirm** button.


Quantitative Analysis

5

Quantitative Analysis

Method to set Accumulation

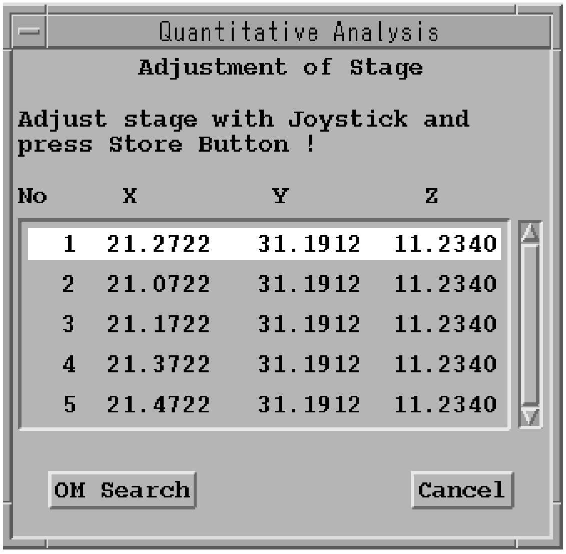
◇ Joystick ◆ Line ◇ Grid ◇ Fix

■ X-axis X steps 5 width(um) 100

☐ Y-axis Y steps width(um)

Fig. 23 Accumulation window

Button	Function
Confirm	After you have specified the number of times to accumulate, 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 up to the number of times to accumulate. If you select **Cancel** before finishing this step, the remaining points of accumulation will be neglected, and the number of times to accumulate will be reset to the number of points confirmed so far.



Adjustment of Stage

Adjust stage with Joystick and press Store Button !

No	X	Y	Z
1	21.2722	31.1912	11.2340
2	21.0722	31.1912	11.2340
3	21.1722	31.1912	11.2340
4	21.3722	31.1912	11.2340
5	21.4722	31.1912	11.2340

Fig. 24 Adjustment of Stage window

Button	Function
Stage Position	Displays the present recorded position of the stage.
Beam Position	Displays the present position of the beam (effective only if **Beam** is selected).
Apply	Enters the point of analysis, and records it in the list of coordinates.
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, a 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 current stage position.
X, **Y**, **Z**	Display 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 current stage position and displays it in the **X**, **Y**, **Z** indication boxes.
Move	Moves the stage to the coordinates specified in **X**, **Y** and **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, observing the OM image by using the joystick of the Joystick Controller of the EPMA main unit; then after focusing on the position, eliminate backlash by using the TEST button of the Joystick Controller.

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

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

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

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

Entering the analysis position in the Beam mode

1. Confirm that Scan Type is **Beam**.

   If it is not, click on the **Beam** button.

2. Display an image of the analysis position on the Viewing Display.

   ☞ Refer to the instruction book of the EPMA main unit.

3. Once you have decided on the analysis position, set the image on the Viewing Display to the analysis mode; change the cross cursors to green, and then select analysis points.

4. Click on the **Read** button.

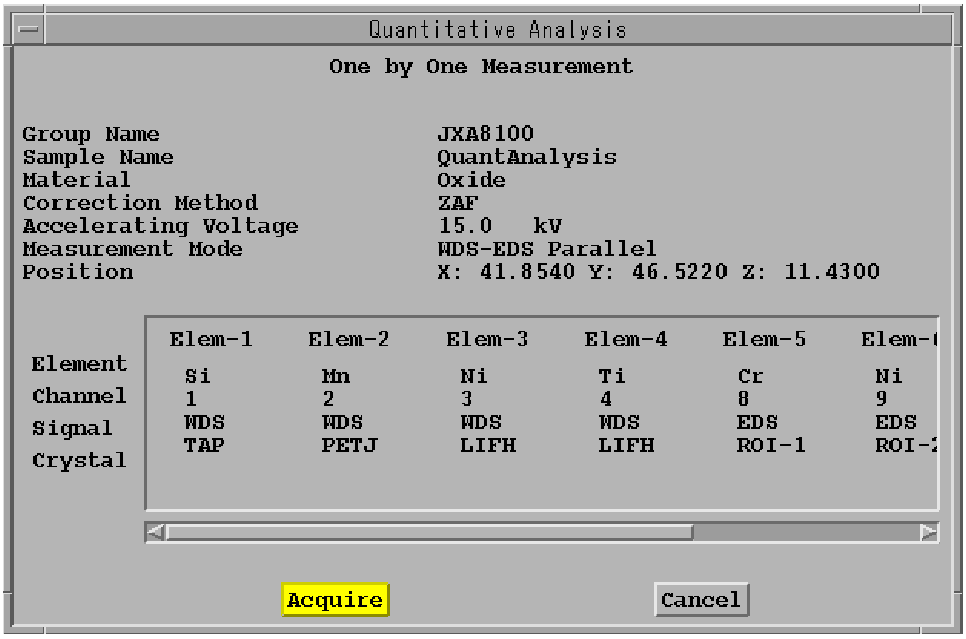
   Stage Position (**X**, **Y**, **Z**), **Magnification**, and **Beam Position** (**X**, **Y**) will be read.

5. To enter the analysis position, click on the **Store** button.

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. 25.



One by One Measurement

Group Name	JXA8100
Sample Name	QuantAnalysis
Material	Oxide
Correction Method	ZAF
Accelerating Voltage	15.0 kV
Measurement Mode	WDS-EDS Parallel
Position	X: 41.8540 Y: 46.5220 Z: 11.4300

	Elem-1	Elem-2	Elem-3	Elem-4	Elem-5	Elem-
Element	Si	Mn	Ni	Ti	Cr	Ni
Channel	1	2	3	4	8	9
Signal	WDS	WDS	WDS	WDS	EDS	EDS
Crystal	TAP	PETJ	LIFH	LIFH	ROI-1	ROI-

Fig. 25 One by One Measurement window

2. Click on the **Acquire** button.

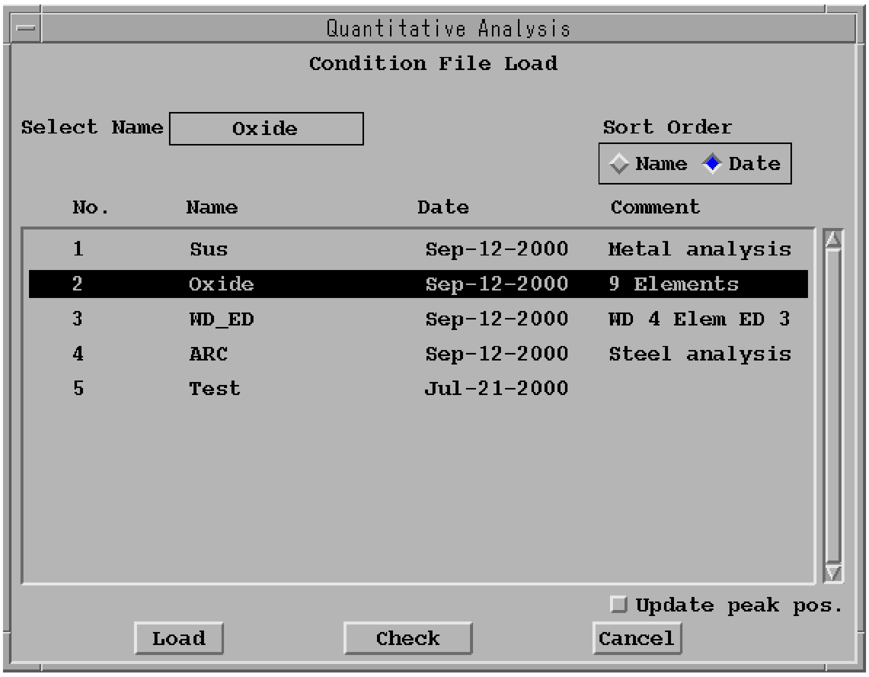
The program executes a single measurement at the highlighted analysis position.

Condition Load

The Condition Load function is used to read out the quantitative analysis conditions that you recorded in advance. Then, you can carry out measurements, using the conditions, by simply selecting samples and inputting the stage position.

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

The Condition File Load window opens as shown in Fig. 26. This window displays **Name** (file names), **Date** (the dates when they were recorded) and **Comment**.



Quantitative Analysis

Condition File Load

Select Name: Oxide

Sort Order: ○ Name ● Date

No.	Name	Date	Comment
1	Sus	Sep-12-2000	Metal analysis
2	Oxide	Sep-12-2000	9 Elements
3	WD_ED	Sep-12-2000	WD 4 Elem ED 3
4	ARC	Sep-12-2000	Steel analysis
5	Test	Jul-21-2000	

☐ Update peak pos.

Fig. 26 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 **Corr. Method**, **Element Condition**, **EOS Condition**, **EDS Condition**, **Standard Condition**, **Print-out Condition**, **Measurement Mode**, and **Additional Function**.

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

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

Condition Store

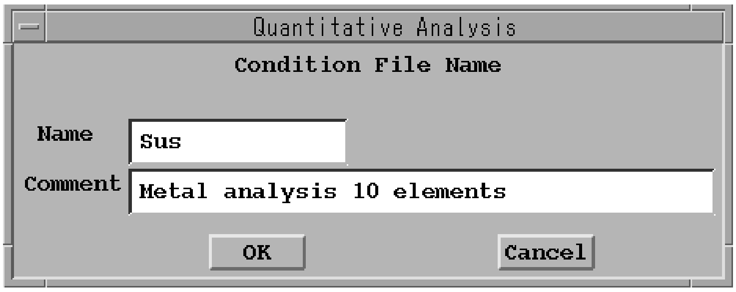
The Condition Store function allows you to save the measurement conditions.

1. Select **Measurement–Condition Store** from the Quantitative Analysis function window.

The Condition File Store window opens.

2. Click on the **New** button of the Condition File Store window.

The Condition File Name window opens as shown in Fig. 27.



Name: Sus
Comment: Metal analysis 10 elements

Fig. 27 Condition File Name window

3. Enter the desired file name (up to 14 characters) and 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 quantitative analysis conditions you have set. The conditions that will be stored are **Corr. Method**, **Element Condition**, **EOS Condition**, **EDS Condition**, **Standard Condition**, **Print-out Condition**, **Measurement Mode**, and **Additional Function**.

To overwrite the conditions in an existing file, click on the name of the file, which becomes highlighted, and then click on the **Store** button.

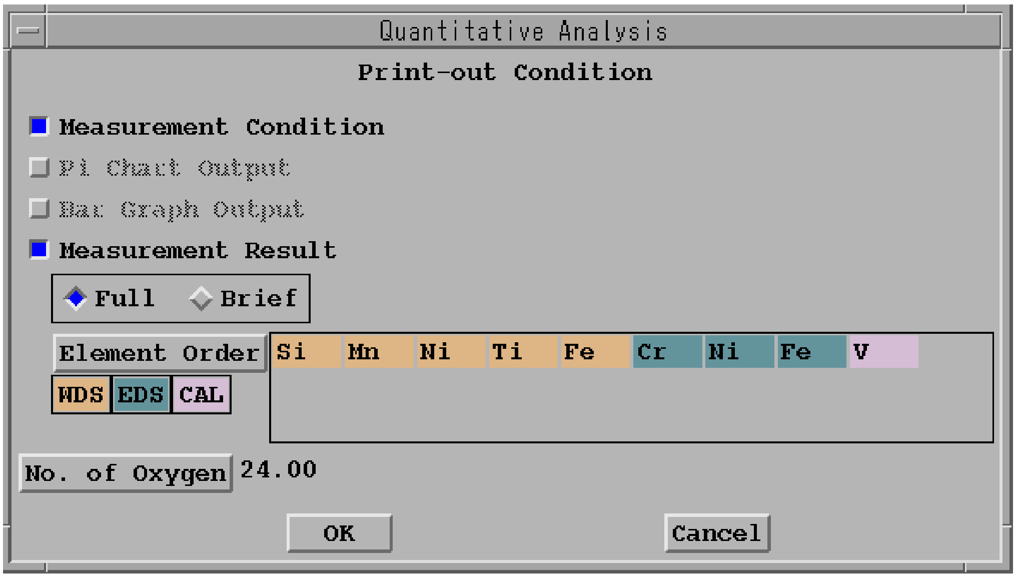
For **Standard Condition**, the program stores only the name of a standard sample, without storing its X-ray intensity. Therefore, the X-ray intensity of the standard sample will be changed when it is measured again.

Printout Condition

The Printout Condition function allows you to turn on and off the output to a printer during measurement, specify the information to be printed out, and control the sequence of the elements to be output.

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

The Print-out Condition window opens as shown in Fig. 28.



Quantitative Analysis

Print-out Condition

☑ Measurement Condition

☐ Pi Chart Output

☐ Bar Graph Output

☑ Measurement Result

◆ Full ◇ Brief

Element Order: Si Mn Ni Ti Fe Cr Ni Fe V

No. of Oxygen 24.00

Fig. 28 Print-out Condition window

Button	Function
Measurement Condition	Prints out the measurement conditions when the measurement results of the first analysis point are output.
Measurement Result	When this item is selected, the measurement results will be output to the printer. When it is not selected, the measurement results will not be output to the printer. When **Full** is selected, detailed contents of the results will be output. When **Brief** is selected, some results will be omitted from the output.
Element Order	Changes the output order of the elements. Select the output order of the elements in the Element Order window that is opened after you select **Element Order**.
No. of Oxygen	Specifies the number of oxygen that is required when the cation is to be calculated in the analysis of oxides.

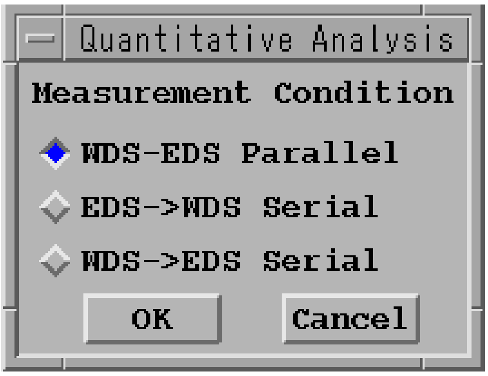
✍ The output to the printer is controlled by turning the **Measurement Result** button on or off. However, the results of analysis will be displayed in the Listing window during analysis regardless of the setting of the **Measurement Result** button. You can also print out the results of analysis by using the **Summary** program.

Measurement Mode

With the JXA-8200 Series, the Measurement Mode function allows you to specify the sequence of measurement when you use both the WDS and the EDS. For measurement using only one (not both) of the WDS and the EDS, any measurement mode specified here will be invalid in an actual measurement.

◆ Select **Measurement–Measurement Mode** from the Quantitative Analysis function window.

The Measurement Mode window opens as shown in Fig. 29.



Quantitative Analysis

Measurement Condition

● WDS-EDS Parallel

○ EDS->WDS Serial

○ WDS->EDS Serial

Fig. 29 Measurement Mode window

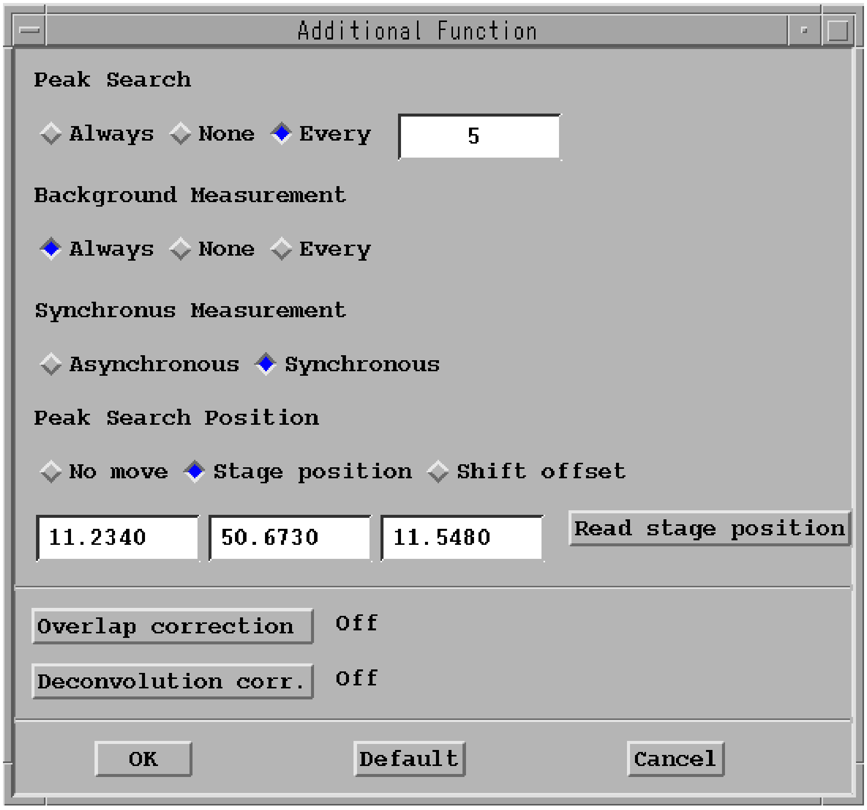
Button	Function
WDS–EDS Parallel	Performs measurements using both the WDS and EDS simultaneously.
EDS–>WDS Serial	Performs measurements first with the EDS and then with the WDS.
WDS–>EDS Serial	Performs measurements first with the WDS and then with the EDS.

Additional Function

In WDS measurement, this function allows you to set up conditions for Peak Search and Background Measurement, to select Synchronous or Asynchronous, and to set up conditions for interference correction.

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

The Additional Function window opens as shown in Fig. 30.



Additional Function

Peak Search

◇ Always ◇ None ◆ Every 5

Background Measurement

◆ Always ◇ None ◇ Every

Synchronus Measurement

◇ Asynchronous ◆ Synchronous

Peak Search Position

◇ No move ◆ Stage position ◇ Shift offset

11.2340 50.6730 11.5480

Off

Off

Fig. 30 Additional Function window

Peak Search

The Peak Search function enables you to set up conditions for peak search in WDS measurement.

Button	Function
Always	Performs peak search (default).
None	Does not perform peak search.
Every	Performs peak search once for every specified number of measurements. Enter an integer in the input box to specify the number of measurements. For example, if you enter 5, the peak search will be executed for every 5 samples. In the event that the number of the peak search assigned to the spectrometer is 0 in the WDS Element Condition window (☞ Fig. 11), peak searches will not be performed even if **Every** is specified here.

Background Measurement

The Background Measurement function enables you to set up conditions for background measurement in WDS measurement.

Button	Function
Always	Performs background measurement (default).
None	Does not perform background measurement.
Every	Performs background measurement once for every specified number of measurements. If you do not perform background measurement, the latest background intensity will be applied. In the event that 0 is specified as the positive or negative distance of background in the WDS Element Condition window (☞ Fig. 11), background measurements will not be performed even if **Every** is specified here.

Synchronous Measurement

The Synchronous Measurement function enables you to set up conditions for spectrometer driving in WDS measurement.

Button	Function
Asynchronous	Each spectrometer moves and performs measurement independently after the sample stage has moved to the measurement position. When measurement is completed under all the measurement conditions of the WDS Element Condition window (☞ Fig. 11), the stage moves to the subsequent measurement position. **Asynchronous** is a default.
Synchronous	This mode is also called the round method. Measurement is carried out by moving the stage, with the spectrometer locked. If this mode is specified, the position for peak search measurement can be specified.

Peak Search Position

The Peak Search Position function enables you to specify the stage position for performing peak search in the **Synchronous** mode.

Button	Function
No move	Peak search is carried out at the first stage position in the stage conditions.
Stage position	The absolute position where you want to perform peak search is specified If you have clicked on the **Read stage position** button, the current position will be set (unit: mm).
Shift offset	The relative position where you want to perform peak search is specified as a position change to the first stage position in the stage conditions (unit: mm).

● Overlap Correction

When the interfering X-rays are in the vicinity of the peaks of the measured X-rays and they influence the peak intensity of the measured X-rays, calibrate peak overlap before executing correction calculation and utilize the result of this calibration for executing conversion on peak intensity in the correction calculation.

The conversion formula is as follows.

Corrected intensity = Measured intensity−Correction value × Intensity of interfering X-rays

◆ Click on the **Overlap Correction** button in the Additional Function window.

The Peak overlap correction window opens as shown in Fig. 32.

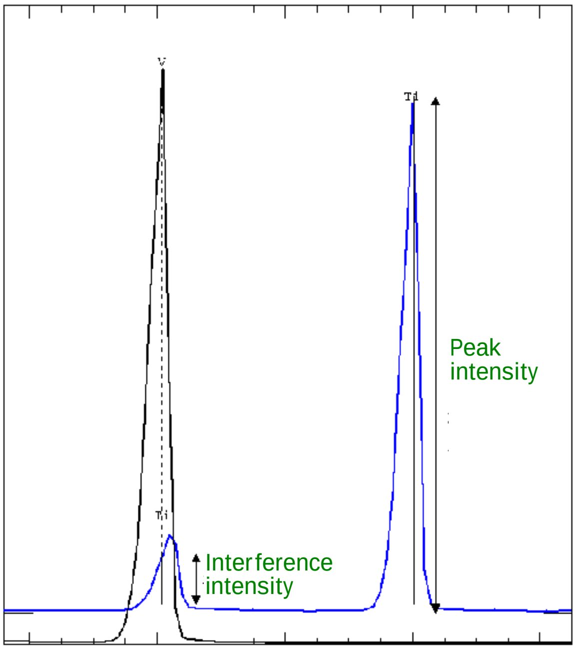


Fig. 31 Peak overlap correction window

- Specify the name of the element that suffers from the interfering X-rays in the first element name. Specify the name of the element that generates the interfering X-rays in the second element name. When you click on the **Peak overlap** button, the list indicating the combination of elements that generates the adjacent peak is displayed.
- When you know the correction value beforehand, enter the value in the **Coefficient** column. When you do not know it, click on the **Calibrate** button and execute calibration.

Calibration is executed in the following order.

1. Ordinary peak measurement and background signal measurement are performed on the standard sample that contains the first element.

   Peak position is obtained.

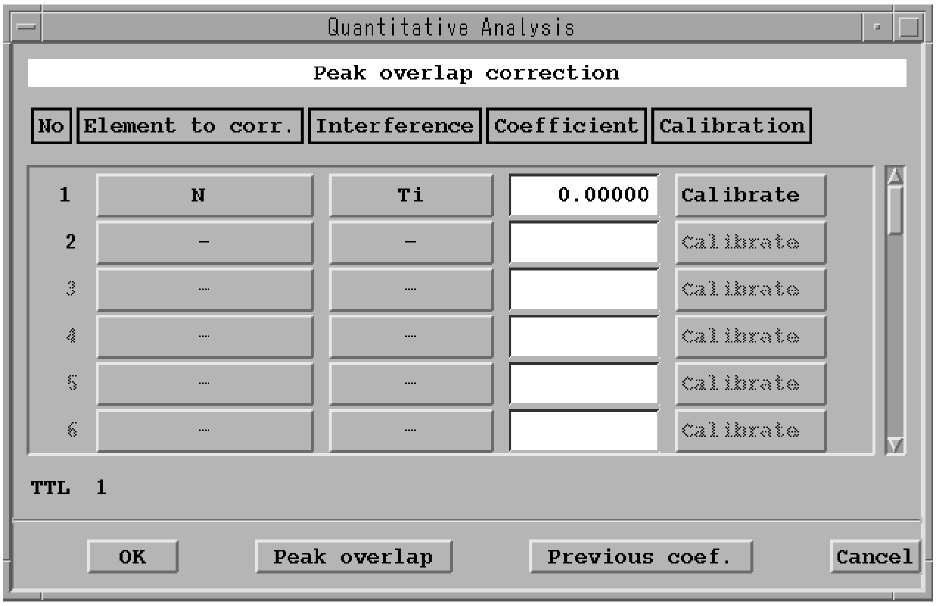
2. Measurement of the intensity of the X-rays is performed on the standard sample that contains the second element under the conditions mentioned above.

   The intensity of the interfering X-rays is obtained.

3. Ordinary peak measurement and background signal measurement are performed on the standard sample that contains the second element under the conditions for the second element.

   The intensity of the X-rays of the element that generates the interfering X-rays is obtained. The correction value is obtained by dividing the actual intensity of Step 2 by that of Step 3.

   ✍ Before executing calibration, it is necessary to align the coordinate position using the Standard Sample Analysis Program.



Peak overlap correction

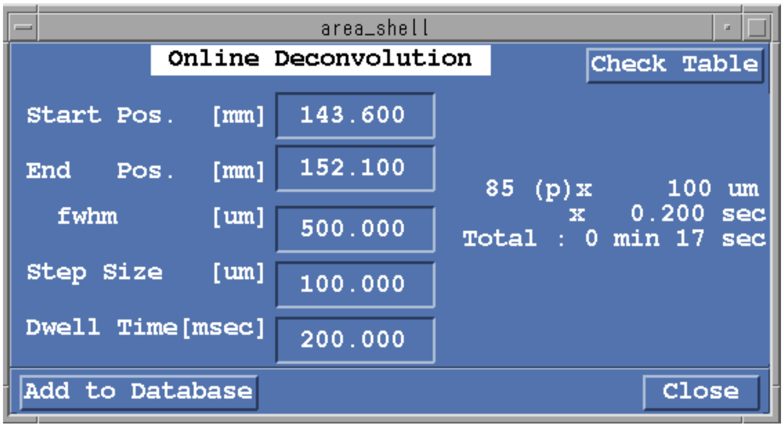
No	Element to corr.	Interference	Coefficient	Calibration
1	N	Ti	0.00000	Calibrate
2	-	-		Calibrate
3	....	....		Calibrate
4	....	....		Calibrate
5	....	....		Calibrate
6	....	....		Calibrate

TTL 1

Fig. 32 Peak overlap correction window

Deconvolution correction

The Deconvolution correction function allows you to obtain peak intensity by deconvolution. Although this method resembles the Peak overlap method, it is used when it is difficult to separate peaks using the Peak overlap method due to the fact that the peak position is very close to the other peak positions. In the X-ray measurement mode by which the deconvolution correction is to be executed on the elements, you need to select **Area**. (For the interfering X-rays, using the ordinary-constant time mode is acceptable.) When you set the scan conditions of **Area**, it is necessary to select the Area mode in the peak search monitor and set the conditions using the **Online Deconvolution** in **Utility** in the peak search monitor. In this window, set the scan start and end points, step size and sampling time.



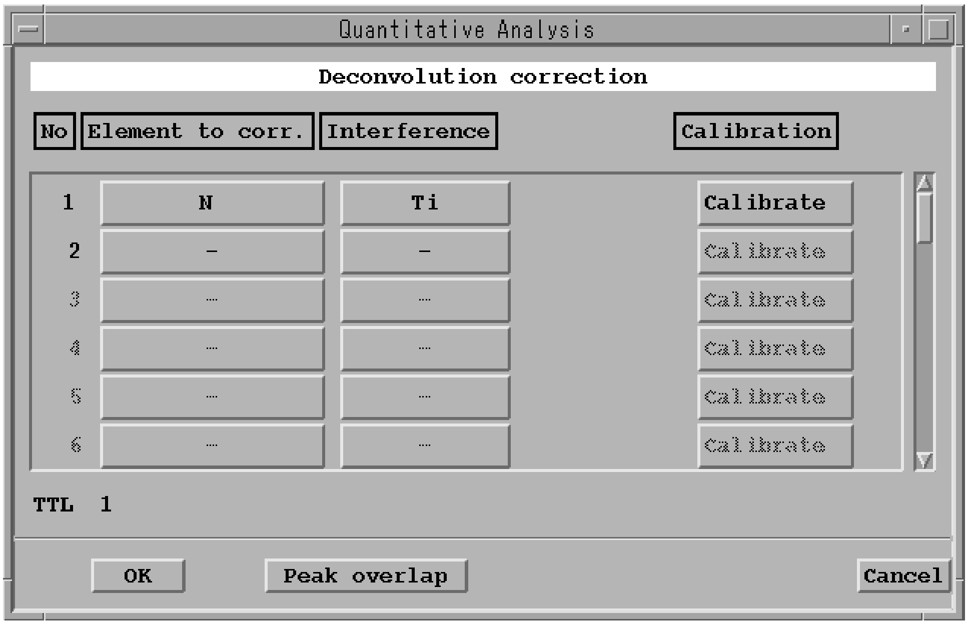
Start Pos. [mm] 143.600
End Pos. [mm] 152.100
fwhm [um] 500.000
Step Size [um] 100.000
Dwell Time[msec] 200.000

85 (p)x 100 um
x 0.200 sec
Total : 0 min 17 sec

Fig. 33 Online Deconvolution window in the peak search monitor

◆ Click on the **Deconvolution Corr.** button in the Additional Function window.

The Deconvolution correction window opens as shown in Fig. 34.



No	Element to corr.	Interference	Calibration
1	N	Ti	Calibrate
2	-	-	Calibrate
3	...	...	Calibrate
4	...	...	Calibrate
5	...	...	Calibrate
6	...	...	Calibrate

TTL 1

Fig. 34 Deconvolution correction window

Specify the name of the element that suffers from the interfering X-rays in the first element name. Specify the name of the element that generates the interfering X-rays in the second element name. When you click on the **Peak overlap** button, the list indicating the combination of elements that generates the adjacent peak is displayed.

To execute the online deconvolution, it is necessary to execute calibration one time beforehand. Click on the **Calibrate** button to execute it.

Calibration is executed in the following order.

1. Peak measurement is performed on the standard sample that contains the first element according to the Area method. Then, the measured data is saved as qualitatively analyzed data with the same standard sample name under the group named Peak_Area.

   Four-digit numerical values are assigned to the saved data in the following order: accelerating voltage, channel number, and crystal holder number. Only accelerating voltage is expressed using two digits. For example, if accelerating voltage is 15 kV, channel number is 4, and crystal holder number is 2, the number 1542 is assigned to the saved data.

2. Peak measurement is performed on the standard sample that contains the element generating the interfering X-rays according to the same procedure of the Area method as mentioned in 1. Then, the measured data is saved as qualitatively analyzed data in the same way as mentioned in 1.

   ✍ Before executing calibration, it is necessary to align the coordinate position using the Standard Sample Analysis Program.

Measurement of the unknown specimen is executed in the following order.

1. Spectrum measurement is performed on the unknown sample according to the Area method. Then, the measured data is saved as qualitatively analyzed data with the unknown sample name and number under the group named Peak_Area.
2. After measurement ends, deconvolution is executed using the spectrum from the standard sample subjected to calibration, as a reference spectrum. Then, the calculated K-ratio is transferred to the correction routine.
3. The correction routine executes correction calculation by using the K-ratio (mentioned in 2) after printing the result of the deconvolution.

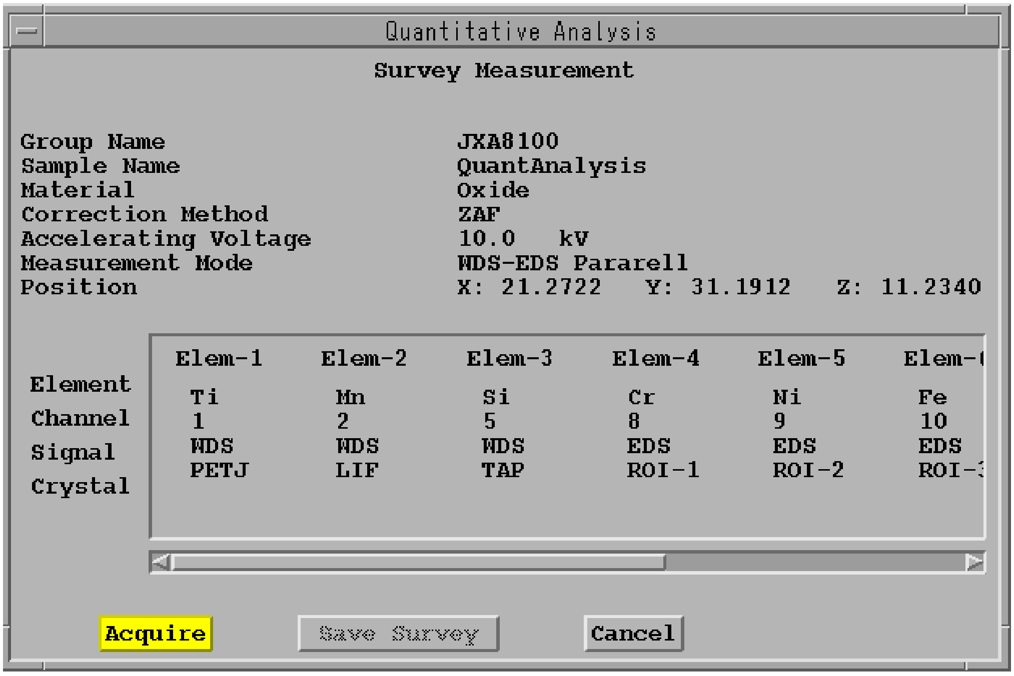
   If you have the optional deconvolution program, you can perform deconvolution after measurement, and check how far deconvolution has proceeded.

Survey Measurement

The Survey Measurement function allows you to perform measurements at the present stage position under the present EOS conditions, though the analysis conditions have been already set up. This measurement is used for your reference before you perform precise measurement.

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

The Survey Measurement window opens as shown in Fig. 35.



Quantitative Analysis

Survey Measurement

Group Name	JXA8100
Sample Name	QuantAnalysis
Material	Oxide
Correction Method	ZAF
Accelerating Voltage	10.0 kV
Measurement Mode	WDS-EDS Pararell
Position	X: 21.2722 Y: 31.1912 Z: 11.2340

	Elem-1	Elem-2	Elem-3	Elem-4	Elem-5	Elem-
Element	Ti	Mn	Si	Cr	Ni	Fe
Channel	1	2	5	8	9	10
Signal	WDS	WDS	WDS	EDS	EDS	EDS
Crystal	PETJ	LIF	TAP	ROI-1	ROI-2	ROI-

Fig. 35 Survey Measurement window

2. Click on the **Acquire** button in this window.

Survey measurement will be performed at the present stage position under the present EOS conditions. The data that you obtained are always stored at the stage No. 99999, being overwritten with every measurement.

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

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

Preset Measurement

The Preset Measurement function allows you to perform measurements in succession by using stored measurement conditions.

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

The Preset Measurement window opens. Its format is similar to the Survey Measurement window shown in Fig. 35.

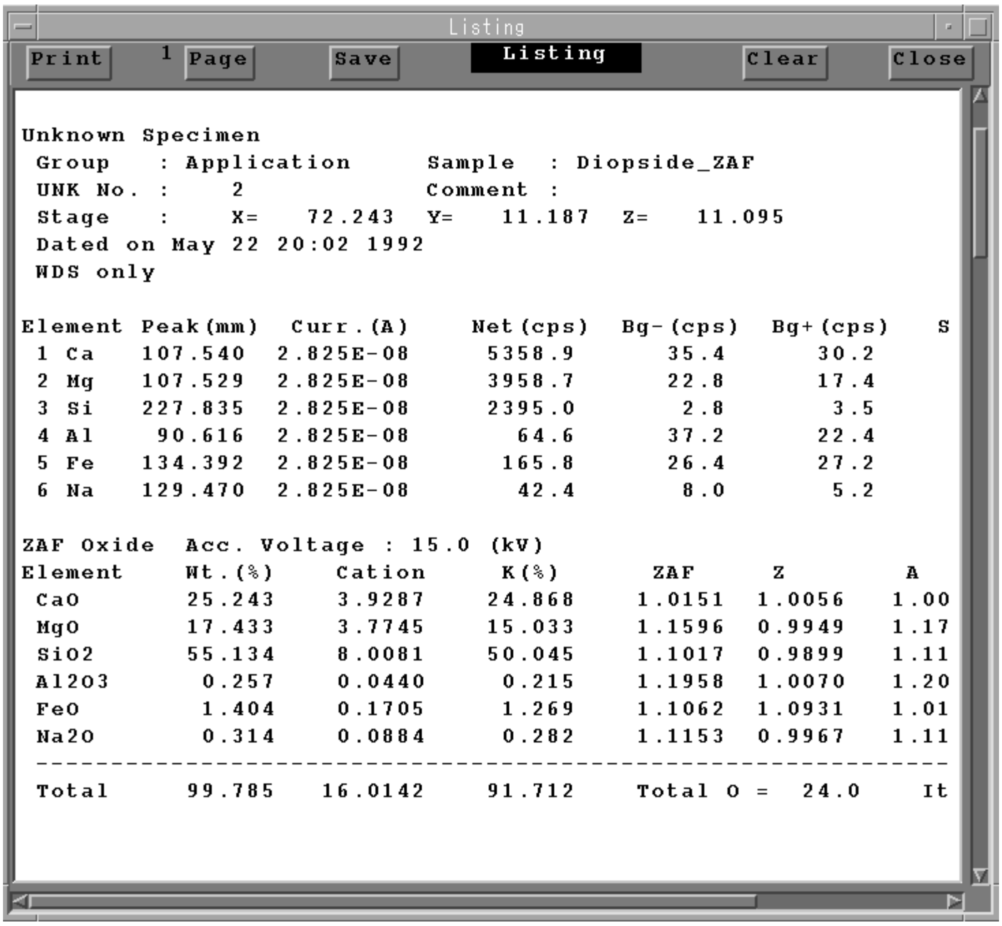
2. Click on the **Acquire** button in the Preset Measurement window.

The preset measurement conditions and the analysis positions in the list of the Stage Condition window whose **Preset** check boxes are turned on will be loaded. Then the measurement will be carried out in succession at the analysis positions, being started with the smallest number. Finally, the correction calculations will be performed.

● Quant Analysis Measurement window

During measurement, the Quant Analysis Measurement window in Fig. 37 will be displayed with the Listing window shown in Fig. 36, displaying the progress for the measurement being made.

When the **Measurement Result** button is turned on in the Print-out window (☞ Fig. 28), the data will be printed.

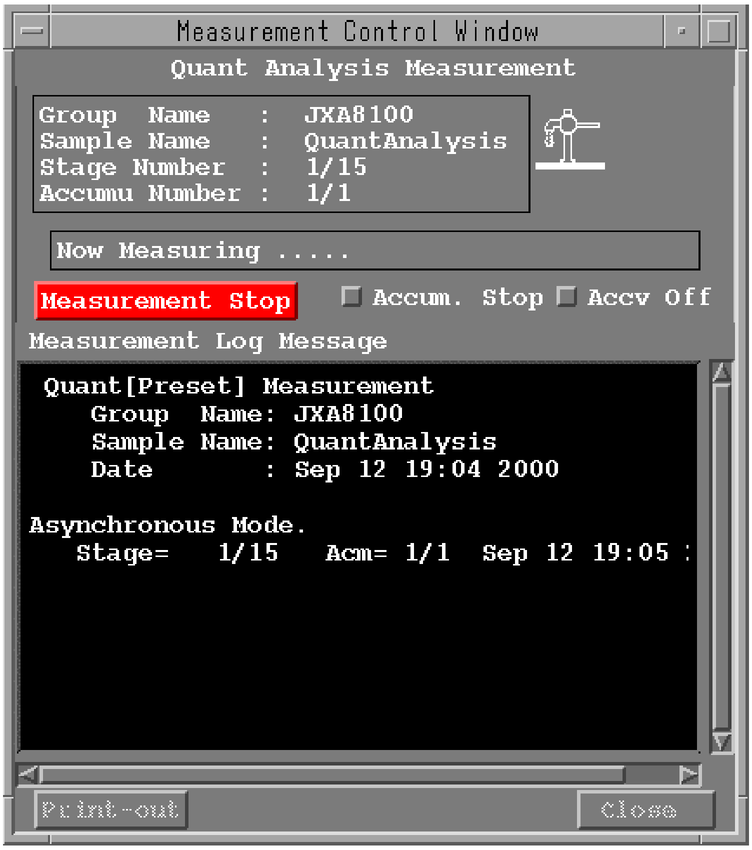


```
Unknown Specimen
 Group    : Application     Sample   : Diopside_ZAF
 UNK No.  :     2           Comment  :
 Stage    :     X=    72.243   Y=    11.187   Z=    11.095
 Dated on May 22 20:02 1992
 WDS only

Element  Peak(mm)   Curr.(A)     Net(cps)   Bg-(cps)   Bg+(cps)    S
 1 Ca    107.540   2.825E-08     5358.9       35.4       30.2
 2 Mg    107.529   2.825E-08     3958.7       22.8       17.4
 3 Si    227.835   2.825E-08     2395.0        2.8        3.5
 4 Al     90.616   2.825E-08       64.6       37.2       22.4
 5 Fe    134.392   2.825E-08      165.8       26.4       27.2
 6 Na    129.470   2.825E-08       42.4        8.0        5.2

ZAF Oxide   Acc. Voltage : 15.0 (kV)
Element     Wt.(%)    Cation       K(%)       ZAF      Z         A
 CaO        25.243    3.9287      24.868    1.0151   1.0056    1.00
 MgO        17.433    3.7745      15.033    1.1596   0.9949    1.17
 SiO2       55.134    8.0081      50.045    1.1017   0.9899    1.11
 Al2O3       0.257    0.0440       0.215    1.1958   1.0070    1.20
 FeO         1.404    0.1705       1.269    1.1062   1.0931    1.01
 Na2O        0.314    0.0884       0.282    1.1153   0.9967    1.11
 --------------------------------------------------------------
 Total      99.785   16.0142      91.712    Total O =   24.0     It
```

Fig. 36 Listing window



Quant Analysis Measurement

Group Name : JXA8100
Sample Name : QuantAnalysis
Stage Number : 1/15
Accumu Number : 1/1

Now Measuring .....

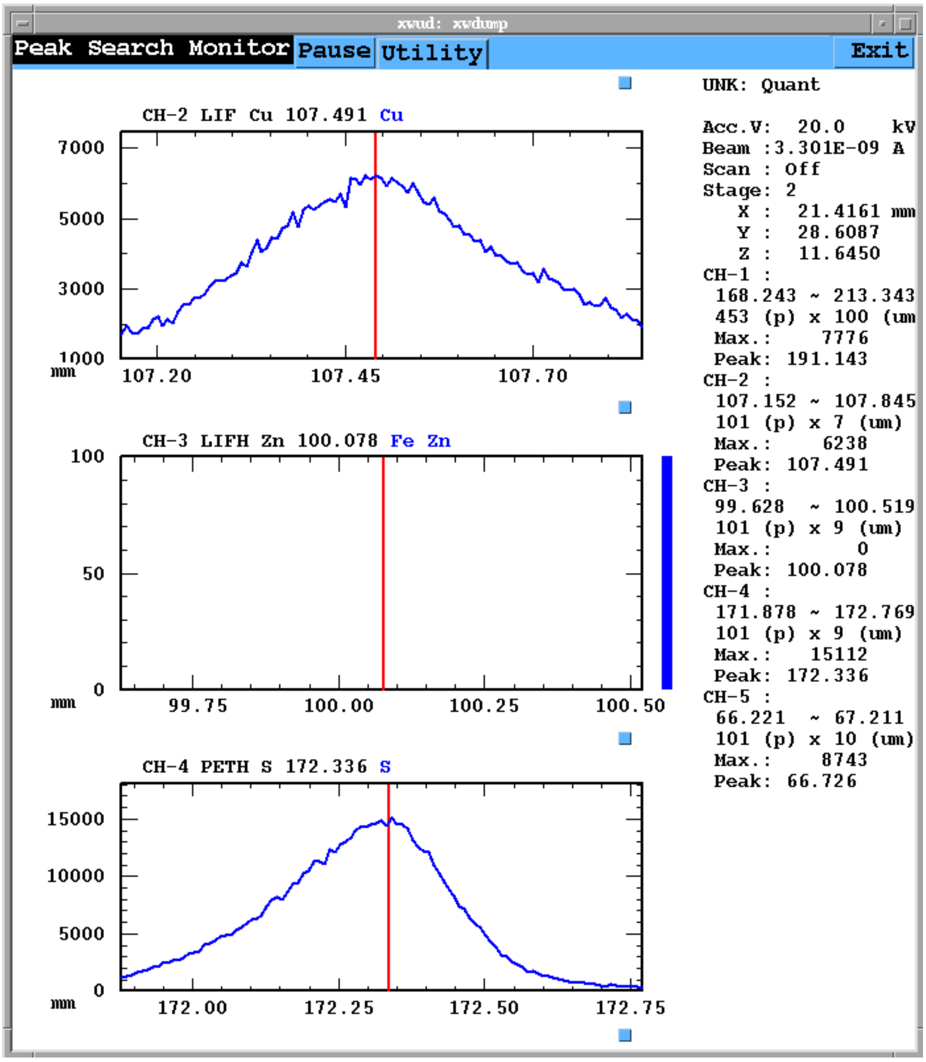
Measurement Log Message

```
 Quant[Preset] Measurement
     Group  Name: JXA8100
     Sample Name: QuantAnalysis
     Date       : Sep 12 19:04 2000

Asynchronous Mode.
   Stage=   1/15   Acm= 1/1  Sep 12 19:05 :
```

Fig. 37 Measurement-under-way window

- ✍ Turn the accelerating voltage on before measurement. If the accelerating voltage is off, a warning message appears, and the measurement will not be performed.
- ✍ 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 displayed on the screen, or minimized. If you execute EDS control or EDS measurement when the EDS Home Window is not active, the instrument will stop measurement due to an error.
- ✍ If you have selected **Analysis–Peak Search Monitor** from the EMPA 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 (☞ Fig. 32). However, never open the Peak Search Monitor window during peak search; otherwise peaks may not be searched accurately.
- ✍ During measurement, do not change the measurement conditions; changing them will seriously influences the measurement. Especially, never change the element conditions.



```
UNK: Quant

Acc.V:  20.0    kV
Beam :3.301E-09 A
Scan : Off
Stage: 2
   X :  21.4161 mm
   Y :  28.6087
   Z :  11.6450
CH-1 :
 168.243 ~ 213.343
 453 (p) x 100 (um
 Max.:    7776
 Peak: 191.143
CH-2 :
 107.152 ~ 107.845
 101 (p) x 7 (um)
 Max.:    6238
 Peak: 107.491
CH-3 :
 99.628  ~ 100.519
 101 (p) x 9 (um)
 Max.:       0
 Peak: 100.078
CH-4 :
 171.878 ~ 172.769
 101 (p) x 9 (um)
 Max.:   15112
 Peak: 172.336
CH-5 :
 66.221  ~ 67.211
 101 (p) x 10 (um)
 Max.:    8743
 Peak: 66.726
```

Fig. 38 Peak Search Monitor window

6.2 Summary

The Summary function mainly allows you to edit and print the results of the Quantitative Analysis. Furthermore, you can print the elements identified by the WDS Qualitative Analysis, elements identified by the EDS Qualitative Analysis, results of the WDS Semi-Quantitative Analysis, results of the EDS Semi-Quantitative Analysis, and results of the Particle Analysis (optional).

6.2.1 Starting and terminating Summary program

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

   The Process menu opens.

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

2. Select **Quantitative Analysis**–**Summary** from the **Process** menu.

   The Process function window opens as shown in Fig. 39.

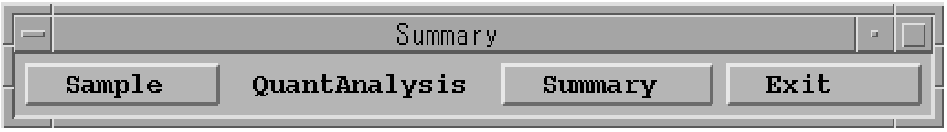


Fig. 39 Process function window

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

6.2.2 Specifying group name and sample name

Specify group name and sample name according to the same procedure as for Sect. 6.1.2 of this instruction manual. However, you can also specify the desired analysis method in the Select Analysis window, which is opened by clicking on the **Analysis** button in the Summary window as shown in Fig. 41.

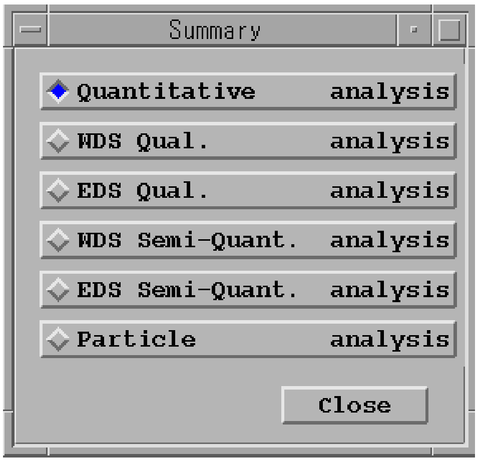
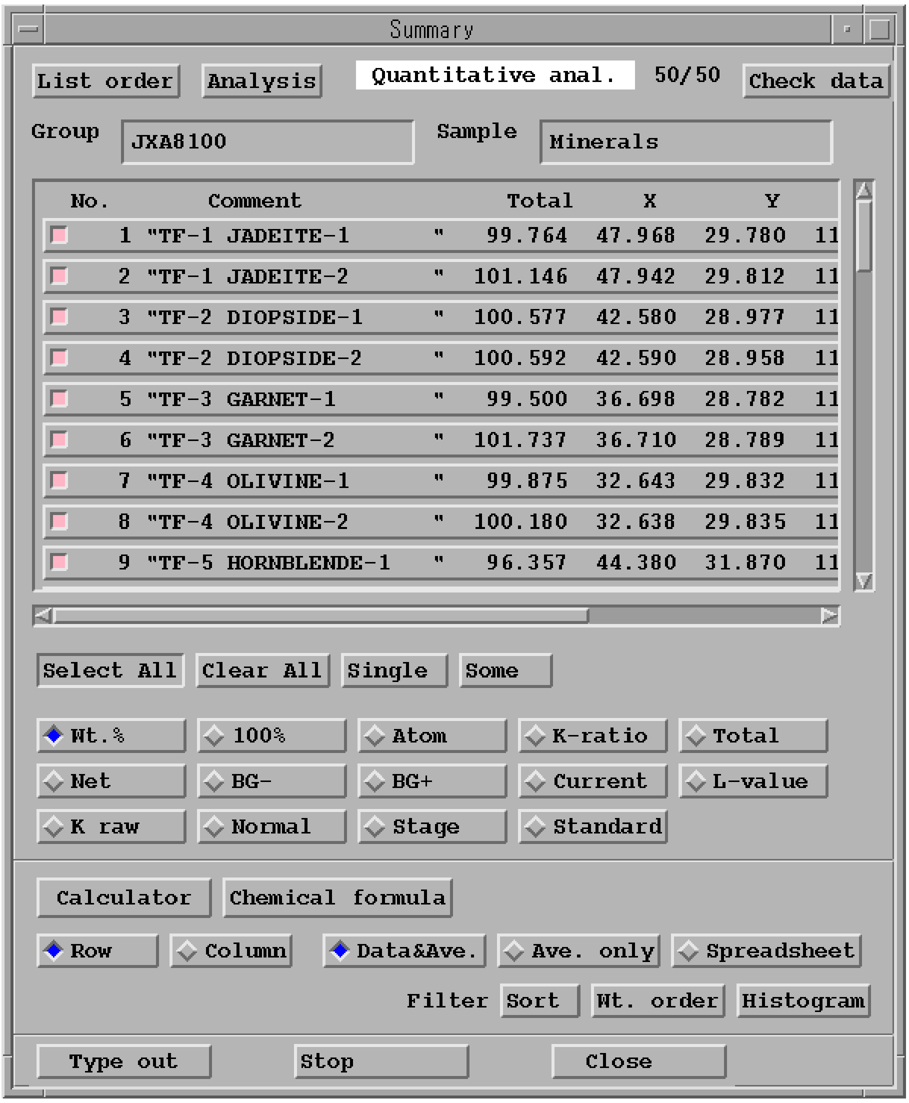


Fig. 40 Select Analysis window

6.2.3 Entering Summary conditions

◆ Click on the **Summary** button of the Process function window.

The Summary window opens as shown in Fig. 41.



Quantitative anal. 50/50

Group JXA8100

Sample Minerals

	No.	Comment	Total	X	Y	
☐	1	"TF-1 JADEITE-1 "	99.764	47.968	29.780	11
☐	2	"TF-1 JADEITE-2 "	101.146	47.942	29.812	11
☐	3	"TF-2 DIOPSIDE-1 "	100.577	42.580	28.977	11
☐	4	"TF-2 DIOPSIDE-2 "	100.592	42.590	28.958	11
☐	5	"TF-3 GARNET-1 "	99.500	36.698	28.782	11
☐	6	"TF-3 GARNET-2 "	101.737	36.710	28.789	11
☐	7	"TF-4 OLIVINE-1 "	99.875	32.643	29.832	11
☐	8	"TF-4 OLIVINE-2 "	100.180	32.638	29.835	11
☐	9	"TF-5 HORNBLENDE-1 "	96.357	44.380	31.870	11

● Wt.% ○ 100% ○ Atom ○ K-ratio ○ Total ○ Net ○ BG- ○ BG+ ○ Current ○ L-value ○ K raw ○ Normal ○ Stage ○ Standard

● Row ○ Column ● Data&Ave. ○ Ave. only ○ Spreadsheet

Fig. 41 Summary window

This window displays all the sample numbers, comments, total values of the results of quantitative analysis carried out under the specified Group/Sample names, coordinates and measurement dates for all the measured samples.

Clicking on the **Analysis** button in the Summary window opens the Select Analysis window, in which you can select the desired analysis method again.

Clicking on the **Select All** button in this window specifies the data for all stage positions, and clicking on the **Clear All** button clears all the data. Clicking on the **Single** button specifies data for a particular stage position. Clicking on the **Some** button selects or cancels data in a certain range.

The following can be printed. Select one for printing.

In the Quantitative Analysis, WDS Semi-Quantitative Analysis, and EDS Semi-Quantitative Analysis, you can select from the following items for printing.

Button	Function
Mass %	Prints mass concentration value.
100%	Prints mass concentration value standardized to 100%.
Atom	Prints atomic ratio or cation in oxides.
K-ratio	Prints K-ratio, which is necessary for calculation for correction.
Mole%	Prints Mole % for each element in oxides.
Mass & Atom	Prints mass concentration and atomic ratio.
100% & Atom	Prints mass concentration standardized to 100% and atomic ratio.
Total	Prints only the total mass concentration values.
Net	Prints the actual intensity of the X-rays in cps/μA.
BG–	Prints the intensity of the low-angle background signal in cps/μA.
BG+	Prints the intensity of the high-angle background signal in cps/μA.
Current	Prints the beam current.
L-value	Prints the peak position in the unit of mm.
K raw	Prints the intensity ratio between an unknown sample and the standard sample without current correction.
Normal	Prints the result of quantitative analysis for each unknown sample in the standard format.
Stage	Prints the list of the stage numbers in the Summary window.
Standard	Prints the data of the standard samples.

In the WDS Qualitative Analysis and EDS Qualitative Analysis, you can select from the following items for printing.

Button	Function
A, B rank	Prints the values of the identified elements in both A and B ranks.
A rank	Prints only the values in the A rank of the identified elements.
B rank	Prints only the values in the B rank of the identified elements.
All	Prints the comments, Stage positions and identified elements.

✍ In Particle Analysis (optional), you can select parameters and chemical types for printing.

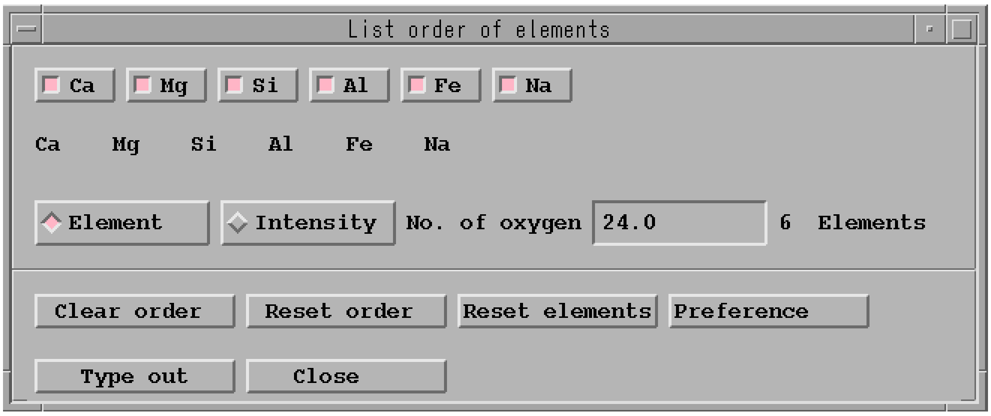
You can select from the following printing formats.

Button	Function
Row	Prints each data in a row. However, if the number of elements is more than eight, the ninth data will be printed in the following row. Up to 50 pieces of data can be printed on each page. Following the last data, maximum, minimum, average and variance will be printed.
Column	Prints eight data in a column. Following the last data, maximum, minimum, average and variance will be printed.
Data & Ave.	Prints each data and maximum, minimum, average and variance. This setting is the default.
Ave. only	Prints only maximum, minimum, average and variance.
Spreadsheet	Outputs the measured data in a format to allow a personal computer to process the data, using a spreadsheet program. In this format, data are output continuously. Each row ends with <CR><LF> codes. In a row, each data is separated by a <Tab> code. No other special code is used in the format.
Sort	Ordinarily prints data in ascending order. If you specify ascending or descending order for an element, all the data will be printed accordingly.
Mass order	Used for printing the results of Semi-Quantitative Analysis. Since in this Analysis the quantitative correction is executed based on identified elements, it is very hard to distinguish a lot of printed elements. Therefore, if you print data up to the specified number of elements in descending order by mass concentration using this window, you can easily grasp the characteristics of each element. Select **Row** for printing.
Histogram	Prints frequency of occurrence in a specified element. It is useful for statistical processing when a lot of samples are measured.

6.2.4 Specifying the order for printing elements

◆ Click on the **List order** button in the Summary window.

The List order of elements window opens as shown in Fig. 42.



No. of oxygen 24.0

6 Elements

Fig. 42 List order of elements window

Using the List order of elements window, specify the order of elements to be displayed and printed for both **Element** and **Intensity** respectively. Before specifying elements, first, click on the **Clear order** button to clear the previous order, and then click on the element buttons in the desired order.
This window has the following functions.

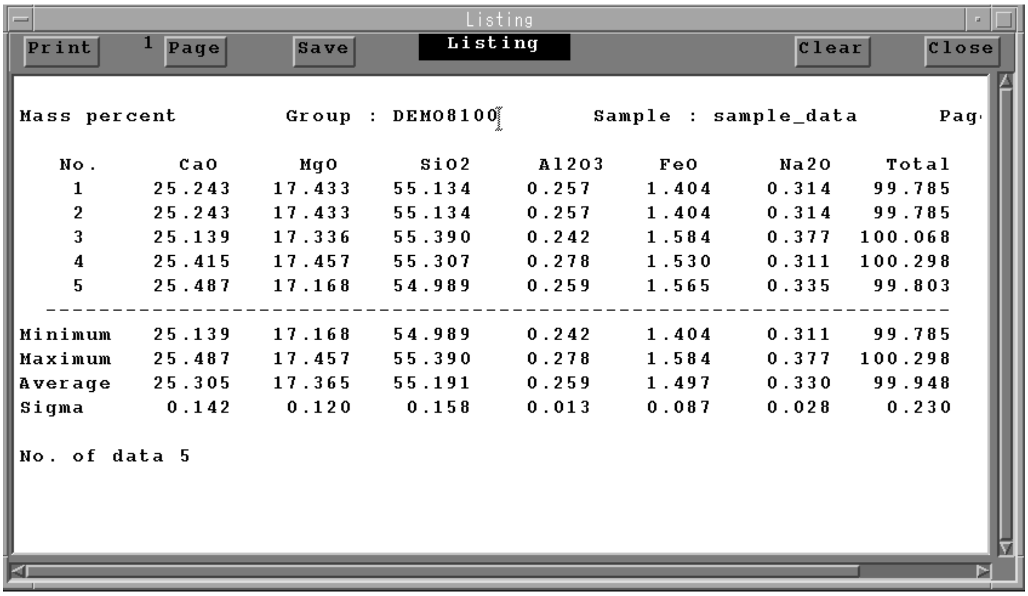
Button	Function
Element	Specifies the order of elements whose concentration is to be printed.
Intensity	Specifies the order of elements whose intensity is to be printed.
Total atom	Specifies the number of atoms to be standardized for printing atomic ratio. In oxides, specify the number of oxygen.
Clear order	Clears all the settings of the order of elements.
Reset order	Resets the order of elements to be printed to the default order.
Reset elements	Reads again the element table from the files of measured data, and enables you to update it. This function is used to update the information in the summary that was printed during measurement.
Preference	Allows you to set further detailed conditions in the summary for printing data. However, only experienced users with enough knowledge can use this function since changing the preset conditions sometimes provokes unexpected troubles in printing data.
Type out	Opens the Listing window. To print data, click on the **Print** button in this window.

Selecting **Reset elements** opens the Editor window in which you can set the following items.

Button	Function
No of Data in Row	Specifies the number of data in a page in row format. By default, 50 data are written in a page.
No of Data in Column	Specifies the number of data in a column in column format. By default, 8 data are written in a column.
No of Line in Page	Specifies the number of lines on a page. If the number of lines exceeds the specified number, a page break code will be inserted. By default, 55 lines are on a page.
Type NULL data	Specify 0 (zero) to display a **–** (dash) if the value of data is zero (default). Specify 1, to display **0.0**.
Min Max	Specify **True** if you want variance to be printed in addition to maximum, minimum and average. If you specify **False**, average will not be printed. **False** exists so that the data obtained by this model can be made compatible with those by the previous models.
Form feed	Specify **True** so that page break codes can be inserted. If you specify **False**, page break codes will not be inserted. **False** exists so that tractor-fed paper can be saved if it is used.
Spreadsheet	Specifies the spreadsheet format for printing. You can select **Excel** or **Lotus**. In **Lotus**, character strings are enclosed in two quotation marks (“ ”).

Button	Function
Spreadsheet TAB	Separates data by a tab or a comma when spreadsheets are printed. Select **TAB** or **COMMA** for the tab or comma marks respectively.
Spreadsheet CR	When data are printed in the spreadsheet format, lines are separated by a backslash n (\ **n**) if you have selected **New_line** (default of Unix). Lines are separated by a <CR> code if you have selected **Return**. If you load data of this spreadsheet on another PC using its spreadsheet software, the data will be displayed on every other line. To avoid this phenomenon, select **Return**.
Block row	Used for processing data in the row format. When the number of elements is more than 8, data will be processed as block if you have selected **True**. If you have selected **False**, first, all the element names are printed, and then data for each item are printed for all the elements until all the items have been printed.
Show comment column	Prints a comment at the same time (if the comment exists), when you have selected **True**. In the row format, a comment of up to 8 characters can be printed. This comment is added after the last character of the data.

To open the Listing window, click on the **Type out** button in the List order of elements window. To print data, click on the **Print** button in this window.



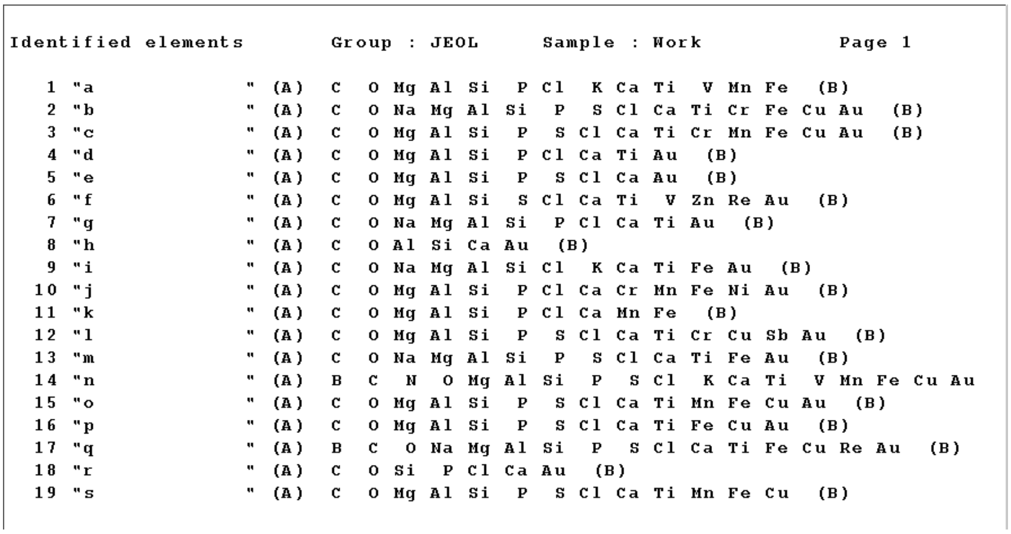
```
Mass percent         Group : DEMO8100       Sample : sample_data      Pag

   No.       CaO      MgO      SiO2     Al2O3     FeO      Na2O    Total
    1      25.243   17.433   55.134    0.257    1.404    0.314   99.785
    2      25.243   17.433   55.134    0.257    1.404    0.314   99.785
    3      25.139   17.336   55.390    0.242    1.584    0.377  100.068
    4      25.415   17.457   55.307    0.278    1.530    0.311  100.298
    5      25.487   17.168   54.989    0.259    1.565    0.335   99.803
  ---------------------------------------------------------------------
Minimum    25.139   17.168   54.989    0.242    1.404    0.311   99.785
Maximum    25.487   17.457   55.390    0.278    1.584    0.377  100.298
Average    25.305   17.365   55.191    0.259    1.497    0.330   99.948
Sigma       0.142    0.120    0.158    0.013    0.087    0.028    0.230

No. of data 5
```

Fig. 43 Listing window

Fig. 44 shows an example of the identified results in the Qualitative Analysis.

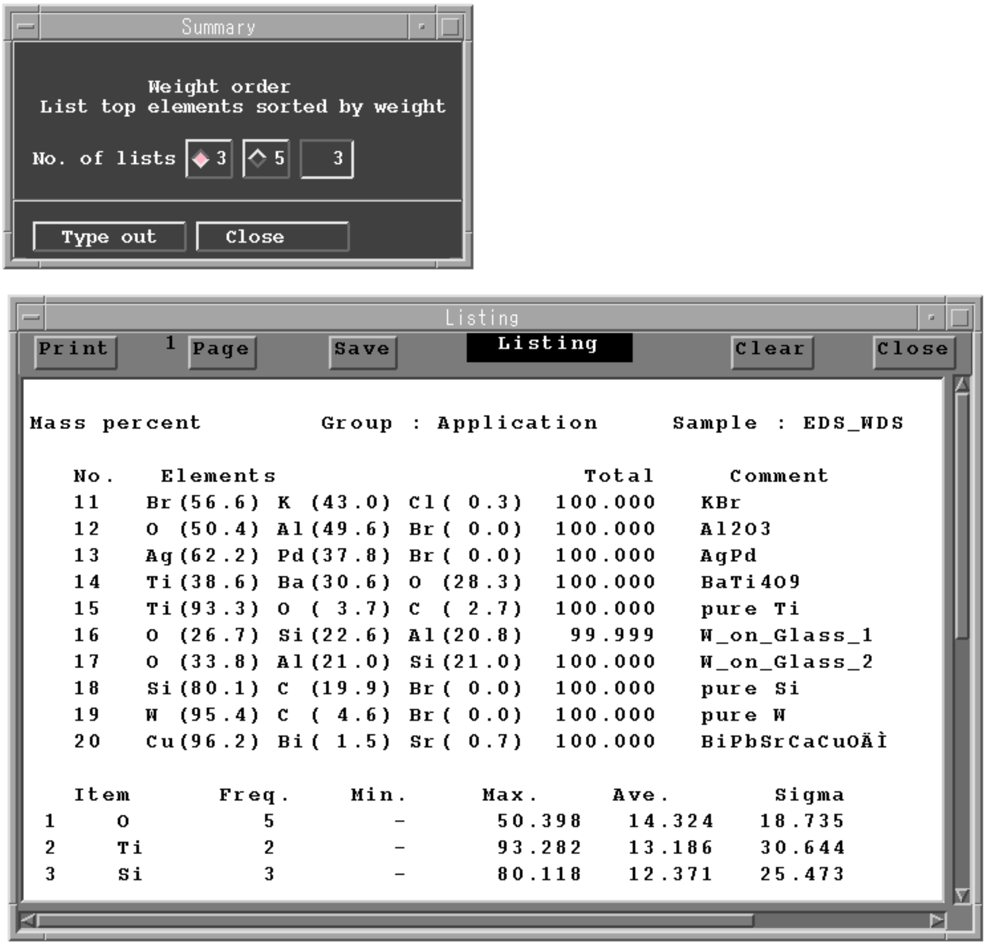


```
Identified elements        Group : JEOL      Sample : Work            Page 1

   1 "a                " (A)  C  O Mg Al Si  P Cl  K Ca Ti  V Mn Fe  (B)
   2 "b                " (A)  C  O Na Mg Al Si  P  S Cl Ca Ti Cr Fe Cu Au  (B)
   3 "c                " (A)  C  O Mg Al Si  P  S Cl Ca Ti Cr Mn Fe Cu Au  (B)
   4 "d                " (A)  C  O Mg Al Si  P Cl Ca Ti Au  (B)
   5 "e                " (A)  C  O Mg Al Si  P  S Cl Ca Au  (B)
   6 "f                " (A)  C  O Mg Al Si  S Cl Ca Ti  V Zn Re Au  (B)
   7 "g                " (A)  C  O Na Mg Al Si  P Cl Ca Ti Au  (B)
   8 "h                " (A)  C  O Al Si Ca Au  (B)
   9 "i                " (A)  C  O Na Mg Al Si Cl  K Ca Ti Fe Au  (B)
  10 "j                " (A)  C  O Mg Al Si  P Cl Ca Cr Mn Fe Ni Au  (B)
  11 "k                " (A)  C  O Mg Al Si  P Cl Ca Mn Fe  (B)
  12 "l                " (A)  C  O Mg Al Si  P  S Cl Ca Ti Cr Cu Sb Au  (B)
  13 "m                " (A)  C  O Na Mg Al Si  P  S Cl Ca Ti Fe Au  (B)
  14 "n                " (A)  B  C  N  O Mg Al Si  P  S Cl  K Ca Ti  V Mn Fe Cu Au
  15 "o                " (A)  C  O Mg Al Si  P  S Cl Ca Ti Mn Fe Cu Au  (B)
  16 "p                " (A)  C  O Mg Al Si  P  S Cl Ca Ti Fe Cu Au  (B)
  17 "q                " (A)  B  C  O Na Mg Al Si  P  S Cl Ca Ti Fe Cu Re Au  (B)
  18 "r                " (A)  C  O Si  P Cl Ca Au  (B)
  19 "s                " (A)  C  O Mg Al Si  P  S Cl Ca Ti Mn Fe Cu  (B)
```

Fig. 44 An example of the identified results in the Qualitative Analysis

Fig. 45 shows an example of the results measured in the Semi-Qualitative Analysis.



Weight order
List top elements sorted by weight

No. of lists ◆ 3 ◇ 5 3

```
Mass percent          Group : Application      Sample : EDS_WDS

   No.   Elements                             Total      Comment
   11    Br(56.6) K (43.0) Cl( 0.3)  100.000    KBr
   12    O (50.4) Al(49.6) Br( 0.0)  100.000    Al2O3
   13    Ag(62.2) Pd(37.8) Br( 0.0)  100.000    AgPd
   14    Ti(38.6) Ba(30.6) O (28.3)  100.000    BaTi4O9
   15    Ti(93.3) O ( 3.7) C ( 2.7)  100.000    pure Ti
   16    O (26.7) Si(22.6) Al(20.8)   99.999    W_on_Glass_1
   17    O (33.8) Al(21.0) Si(21.0)  100.000    W_on_Glass_2
   18    Si(80.1) C (19.9) Br( 0.0)  100.000    pure Si
   19    W (95.4) C ( 4.6) Br( 0.0)  100.000    pure W
   20    Cu(96.2) Bi( 1.5) Sr( 0.7)  100.000    BiPbSrCaCuOÃÌ

   Item       Freq.     Min.      Max.      Ave.      Sigma
 1    O          5        -       50.398    14.324    18.735
 2    Ti         2        -       93.282    13.186    30.644
 3    Si         3        -       80.118    12.371    25.473
```

Fig. 45 An example of Mass order in the Semi-Qualitative Analysis

6.2.5 Confirming data

Click on the **Check data** button in the Summary window (☞ Fig. 41) to check each unknown sample data.

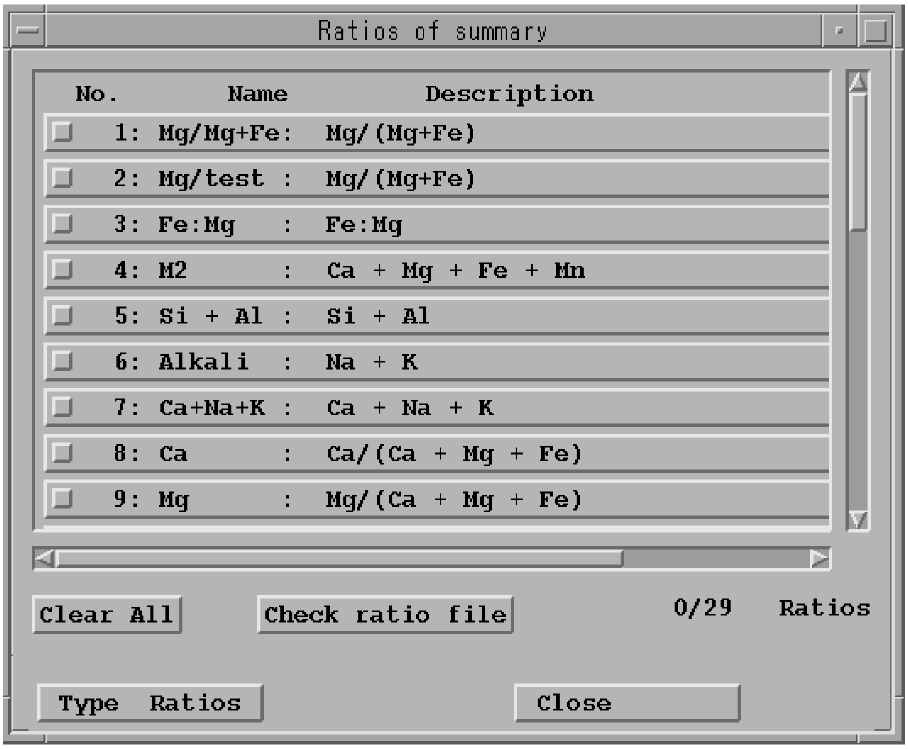
The selected unknown sample data will be displayed one by one every time you click on this button. The display itself is an editor screen, so you can check values of data and correct them as necessary. For instance, you may correct a comment after completion of a measurement.

6.2.6 Calculation mode

Calculator is a button in the Summary window (☞ Fig. 41). Use this button to check the stoichiometric relationship between measured elements.

◆ Click on the **Calculator** button.

☞ The Ratios of summary (Calculator) window opens as shown in Fig. 46. Already recorded conditions are listed in this window.



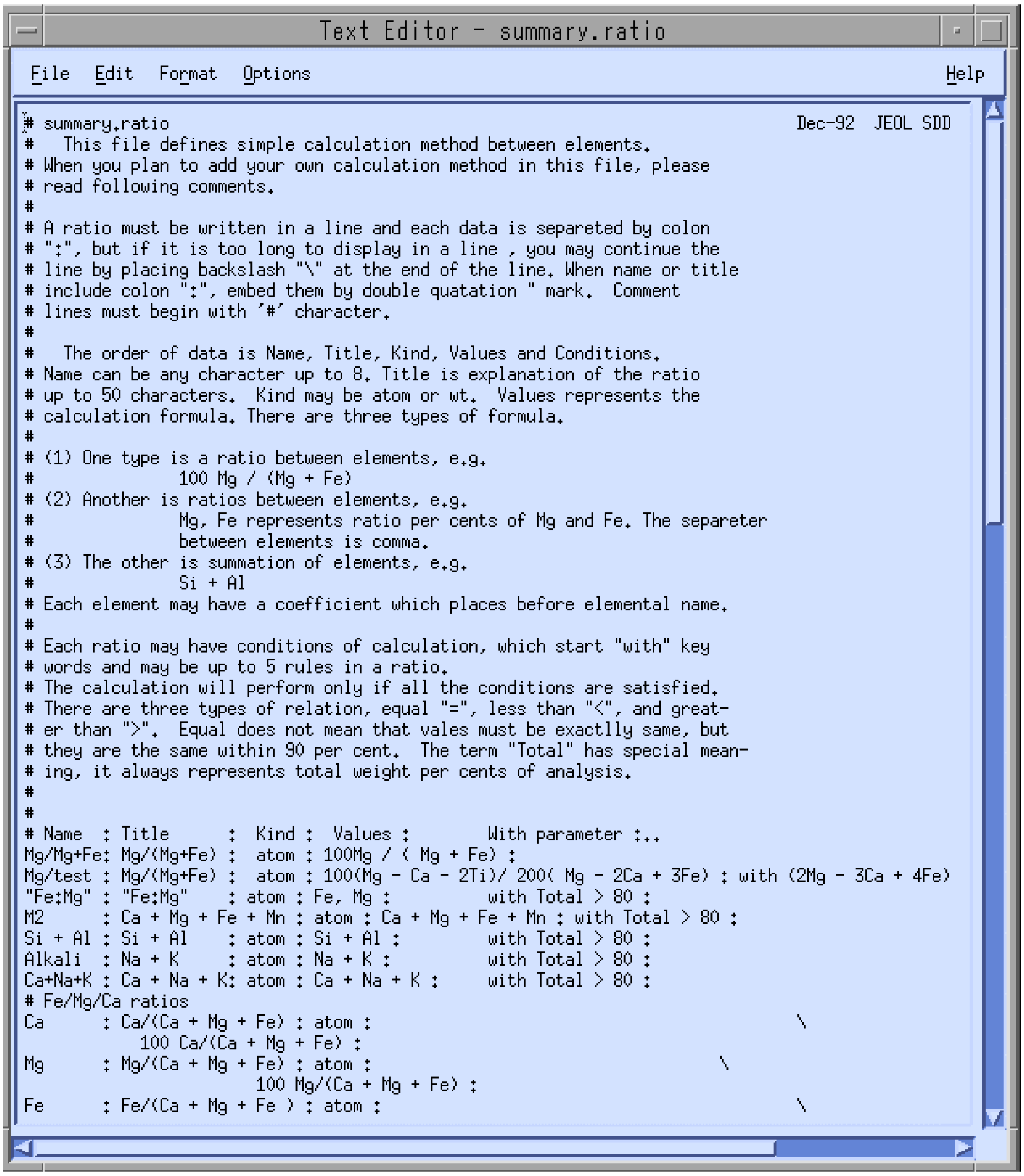
	No.	Name		Description
☐	1:	Mg/Mg+Fe	:	Mg/(Mg+Fe)
☐	2:	Mg/test	:	Mg/(Mg+Fe)
☐	3:	Fe:Mg	:	Fe:Mg
☐	4:	M2	:	Ca + Mg + Fe + Mn
☐	5:	Si + Al	:	Si + Al
☐	6:	Alkali	:	Na + K
☐	7:	Ca+Na+K	:	Ca + Na + K
☐	8:	Ca	:	Ca/(Ca + Mg + Fe)
☐	9:	Mg	:	Mg/(Ca + Mg + Fe)

Fig. 46 Ratios of summary (Calculator) window

Select a condition to be applied and click on the **Type Ratios** button; then the calculated result will be displayed.

◆ If you cannot find the desired condition in the window, click on the **Check ratio file** button.

The Text Editor (Check ratio file) window opens as shown in Fig. 47. In this window you can enter a new condition by yourself.



```
# summary.ratio                                                                 Dec-92  JEOL SDD
#   This file defines simple calculation method between elements.
# When you plan to add your own calculation method in this file, please
# read following comments.
#
# A ratio must be written in a line and each data is separeted by colon
# ":", but if it is too long to display in a line , you may continue the
# line by placing backslash "\" at the end of the line. When name or title
# include colon ":", embed them by double quatation " mark.  Comment
# lines must begin with '#' character.
#
#   The order of data is Name, Title, Kind, Values and Conditions.
# Name can be any character up to 8. Title is explanation of the ratio
# up to 50 characters.  Kind may be atom or wt.  Values represents the
# calculation formula. There are three types of formula.
#
# (1) One type is a ratio between elements, e.g.
#               100 Mg / (Mg + Fe)
# (2) Another is ratios between elements, e.g.
#               Mg, Fe represents ratio per cents of Mg and Fe. The separeter
#               between elements is comma.
# (3) The other is summation of elements, e.g.
#               Si + Al
# Each element may have a coefficient which places before elemental name.
#
# Each ratio may have conditions of calculation, which start "with" key
# words and may be up to 5 rules in a ratio.
# The calculation will perform only if all the conditions are satisfied.
# There are three types of relation, equal "=", less than "<", and great-
# er than ">".  Equal does not mean that vales must be exactlly same, but
# they are the same within 90 per cent.  The term "Total" has special mean-
# ing, it always represents total weight per cents of analysis.
#
#
# Name  : Title      :  Kind :  Values :        With parameter :..
Mg/Mg+Fe: Mg/(Mg+Fe) :  atom : 100Mg / ( Mg + Fe) :
Mg/test : Mg/(Mg+Fe) :  atom : 100(Mg - Ca - 2Ti)/ 200( Mg - 2Ca + 3Fe) : with (2Mg - 3Ca + 4Fe)
"Fe:Mg" : "Fe:Mg"    : atom : Fe, Mg :          with Total > 80 :
M2      : Ca + Mg + Fe + Mn : atom : Ca + Mg + Fe + Mn : with Total > 80 :
Si + Al : Si + Al    : atom : Si + Al :         with Total > 80 :
Alkali  : Na + K     : atom : Na + K :          with Total > 80 :
Ca+Na+K : Ca + Na + K: atom : Ca + Na + K :     with Total > 80 :
# Fe/Mg/Ca ratios
Ca      : Ca/(Ca + Mg + Fe) : atom :                                           \
           100 Ca/(Ca + Mg + Fe) :
Mg      : Mg/(Ca + Mg + Fe) : atom :                                   \
                        100 Mg/(Ca + Mg + Fe) :
Fe      : Fe/(Ca + Mg + Fe ) : atom :                                          \
```

Fig. 47 Text Editor (Check ratio file) window

Specify each item, such as name, comment, concentration or atomic ratio, calculation method and condition, separating by colons (:).

If the information is too long for one line, terminate a line with a backslash (＼) and continue on the next line.

The sort of data to be treated is mass concentration (wt) or atomic ratio (atom).

The equation to be used for calculations consists of four operations (+, –, ×, /) and parentheses. Up to five conditions (parameters following “with”) can be applied to a calculation. These conditions are applied to eliminate improper analysis results.

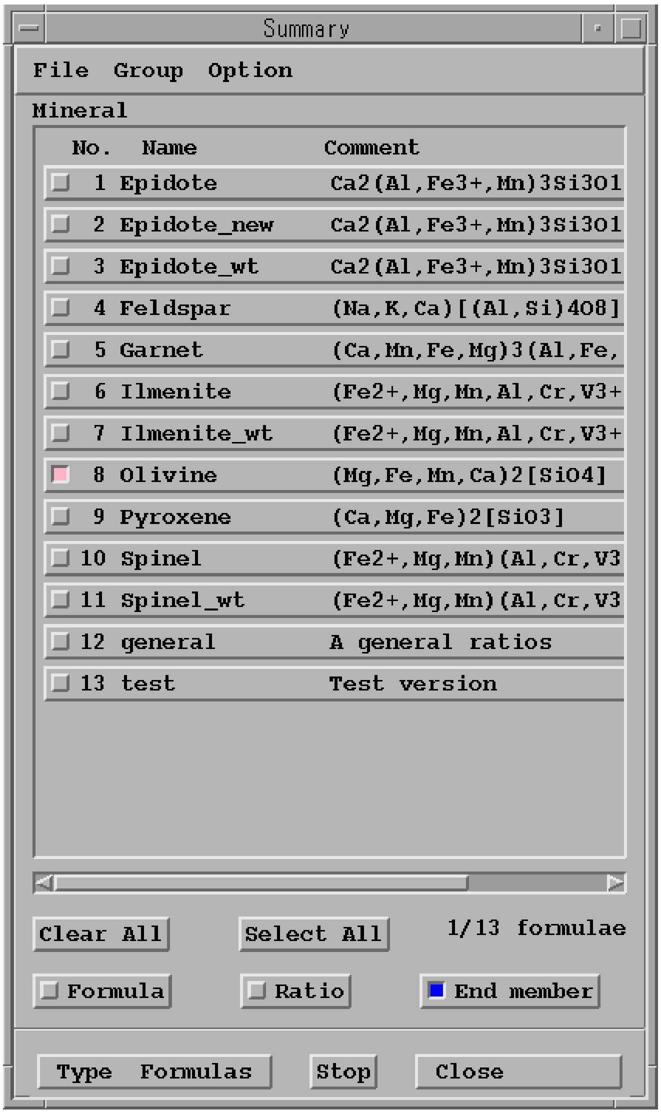
☞ For other details, refer to the explanation displayed in the Text Editor (Check ratio file) window (☞ Fig. 47).

6.2.7 Calculation by using chemical formula

You can obtain the ratio of elements whose chemical formula is known in the result of analysis by using **Chemical formula** in the Summary window.

◆ Click on the **Chemical formula** button in the Summary window.

The Chemical formula window opens.



	No.	Name	Comment
☐	1	Epidote	Ca2(Al,Fe3+,Mn)3Si3O1
☐	2	Epidote_new	Ca2(Al,Fe3+,Mn)3Si3O1
☐	3	Epidote_wt	Ca2(Al,Fe3+,Mn)3Si3O1
☐	4	Feldspar	(Na,K,Ca)[(Al,Si)4O8]
☐	5	Garnet	(Ca,Mn,Fe,Mg)3(Al,Fe,
☐	6	Ilmenite	(Fe2+,Mg,Mn,Al,Cr,V3+
☐	7	Ilmenite_wt	(Fe2+,Mg,Mn,Al,Cr,V3+
☑	8	Olivine	(Mg,Fe,Mn,Ca)2[SiO4]
☐	9	Pyroxene	(Ca,Mg,Fe)2[SiO3]
☐	10	Spinel	(Fe2+,Mg,Mn)(Al,Cr,V3
☐	11	Spinel_wt	(Fe2+,Mg,Mn)(Al,Cr,V3
☐	12	general	A general ratios
☐	13	test	Test version

Fig. 48 Chemical formula window

In this window, the list of stored minerals is displayed. By default, **Mineral** is selected in the chemical group in which Feldspar, Garnet, Olivine, Pyroxene and other minerals are stored.

Click on the desired mineral, and click on the **Type Formulas** button; then you can obtain the printed results that were calculated based on the chemical formula corresponding to the selected mineral. Before printing, select **Formula**, **Ratio**, or **End member**.

The printing algorithm is the following.

- First, the conditional expression is evaluated to judge whether the calculation is to be performed based on the chemical composition of each unknown sample or not. If it is judged so, the calculation will be performed for the sample. This is because to avoid unnecessary calculation for the sample that contains too many kinds of minerals.
- If **Formula** is selected for printing, the calculation for the chemical formula will be performed and its result will be printed.
- If **Ratio** is selected for printing, the calculation for the element ratio will be performed and its result will be printed.
- If **End member** is selected for printing, the calculation for the end member ratio will be performed and its result will be printed.
- If the calculation is judged to be useless, it will not be performed in any selection for printing.

Before printing, select one of the following.

- **Print in single line**

  When this object has been selected, both chemical formulas and calculated values are printed in a line even if multiple items are selected for printing.

  ✍ When this object has not been selected, chemical formulas and calculated values are printed separately if multiple items are selected for printing.
- **Don’t check condition**

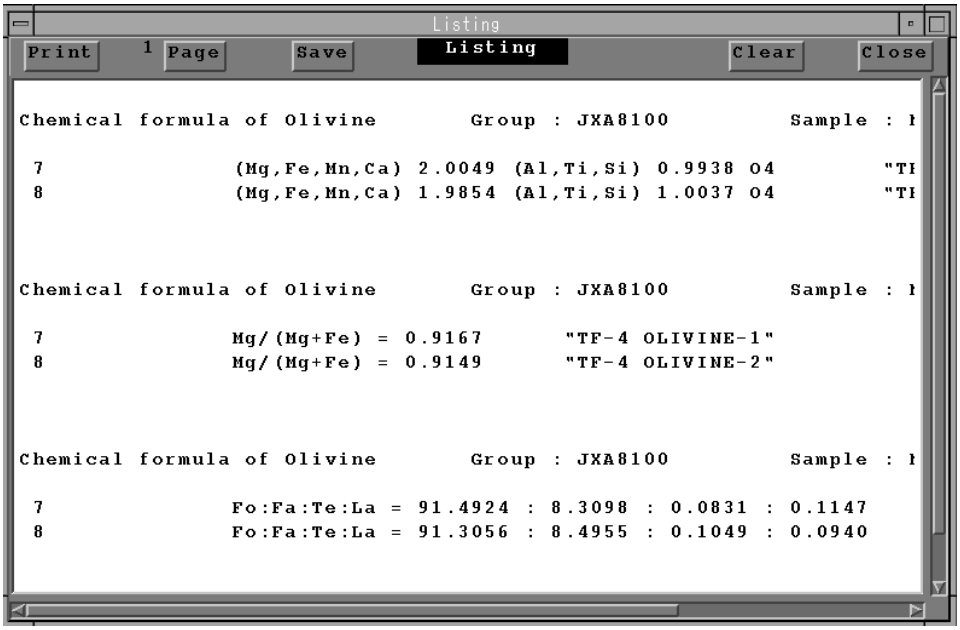
  Usually, the conditional expression is evaluated to judge whether the calculation is to be performed based on the chemical composition of each unknown sample or not. When this object has been selected, the calculation for all the unknown samples is performed without doing the judgment.
- **Print reason if failed**

  If a sample is judged to be improper as mineral, the reason for the judgment will be displayed.
- Sort by formula

  When this object has been selected, data are printed in the preset order of minerals if multiple minerals are selected for printing.

  ✍ When this object has not been selected, data are printed in the selected order of unknown samples if multiple items are selected for printing.

  ☞ For how to store and change minerals, refer to the Help window in the Chemical formula window.



```
Chemical formula of Olivine        Group : JXA8100        Sample : I

 7              (Mg,Fe,Mn,Ca) 2.0049 (Al,Ti,Si) 0.9938 O4        "TI
 8              (Mg,Fe,Mn,Ca) 1.9854 (Al,Ti,Si) 1.0037 O4        "TI



Chemical formula of Olivine        Group : JXA8100        Sample : I

 7              Mg/(Mg+Fe) = 0.9167      "TF-4 OLIVINE-1"
 8              Mg/(Mg+Fe) = 0.9149      "TF-4 OLIVINE-2"



Chemical formula of Olivine        Group : JXA8100        Sample : I

 7              Fo:Fa:Te:La = 91.4924 : 8.3098 : 0.0831 : 0.1147
 8              Fo:Fa:Te:La = 91.3056 : 8.4955 : 0.1049 : 0.0940
```

Fig. 49 Example of Chemical Formula printing

6.3 Off-line Correction

The Off-line Correction program enables you to correct the data of WDS and EDS Quantitative Analyses, WDS Semi-Quantitative Analysis, EDS Semi-Quantitative Analysis. This function is used to compare analysis results between correction methods or to correct them again by changing the data of the standard samples.

6.3.1 Starting and terminating Off-line Correction program

1. Open the EPMA Main Menu on the computer display and then click on the **Process** icon to display the pull-down menu.
   ☞ Refer to the instruction manual of the microanalyzer main unit to learn how to open the EPMA Main Menu.
2. Select **Quantitative Analysis–Off-line Correction** from the **Process** pull-down menu.
   The Off-line Correction function window opens as shown in Fig. 50.

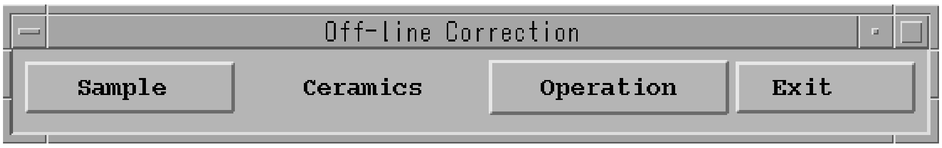


Fig. 50 Off-line Correction function window

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

6.3.2 Specifying group name and sample name

Specify group name and sample name according to the same procedure as for Sect. 6.1.2 of this instruction manual. However, to recall the previous measurement conditions automatically, select the desired sample and finalize the selection by clicking on the **OK** button.

You can set the conditions for off-line correction and those for on-line measurement separately, and so you can execute off-line correction even during quantitative analysis.

6.3.3 Entering conditions for off-line correction

◆ Click on the **Operation** button of the Off-line Correction function window.

The Off-line Correction menu opens as shown in Fig. 51. From this menu, select the items necessary to execute off-line calculation.

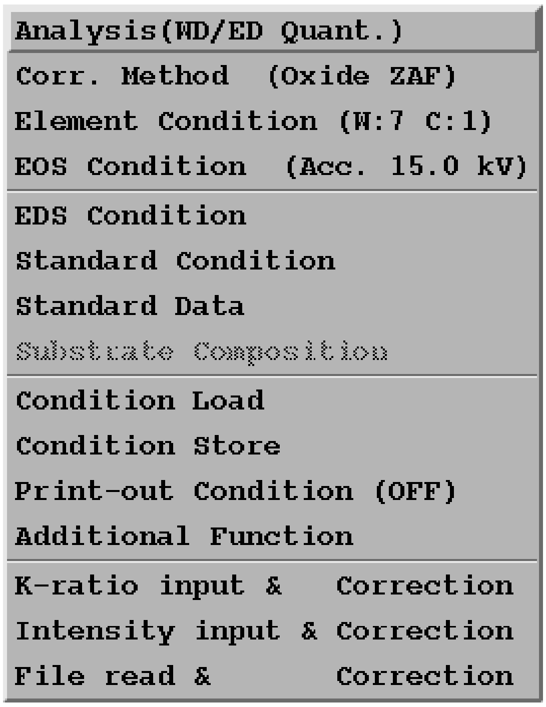


Fig. 51 Off-line Correction menu

As for the following items in the Off-line Correction menu, refer to the Measurement Conditions menu used for entering measurement conditions (☞ Sect. 6.1.3): **Corr. Method**, **Element Condition**, **EOS Condition**, **EDS Condition**, **Standard Condition**, **Condition Load**, **Condition Store**, and **Print-out Condition**. The description below deals with other items: **Analysis** type, **Standard Data**, **K-ratio input & Correction**, **Intensity input & Correction**, and **File read & Correction**.

■ **Analysis** type

Select the desired analysis type for the correction of quantitative analysis.

◆ Select **Analysis** from the Off-line Correction menu.

The Analysis type window opens.

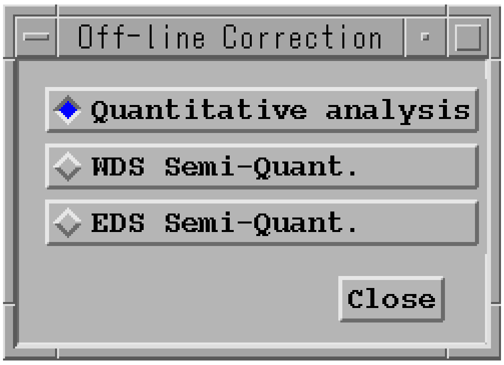


Fig. 52 Analysis type window

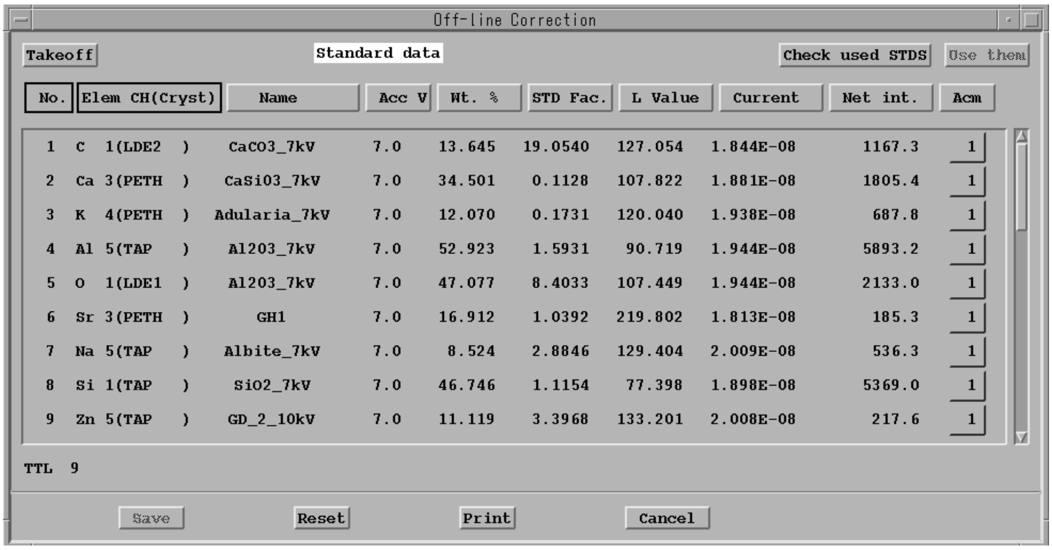
Button	Function
Quantitative analysis	Selects WDS/EDS combined quantitative analysis.
WDS Semi-Quant.	Selects WDS semi-quantitative analysis.
EDS Semi-Quant.	Selects EDS semi-quantitative analysis.

Standard Data

This function allows you to check and correct the data of each standard sample.

◆ Select **Standard Data** from the Off-line Correction menu.

The Standard data window opens as shown in Fig. 53.



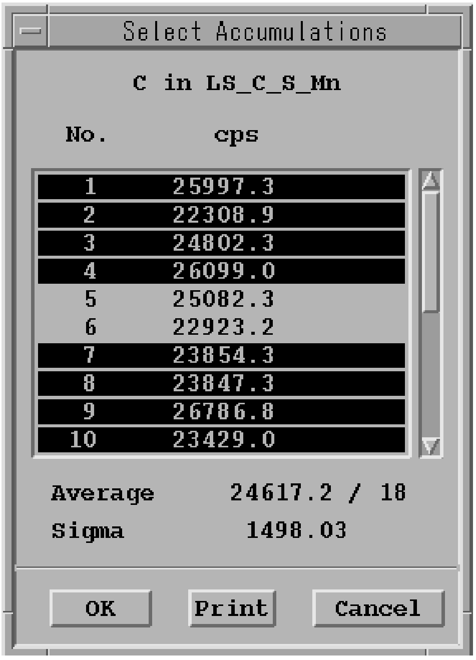
No.	Elem CH(Cryst)	Name	Acc V	Wt. %	STD Fac.	L Value	Current	Net int.	Acm
1	C 1(LDE2)	CaCO3_7kV	7.0	13.645	19.0540	127.054	1.844E-08	1167.3	1
2	Ca 3(PETH)	CaSiO3_7kV	7.0	34.501	0.1128	107.822	1.881E-08	1805.4	1
3	K 4(PETH)	Adularia_7kV	7.0	12.070	0.1731	120.040	1.938E-08	687.8	1
4	Al 5(TAP)	Al2O3_7kV	7.0	52.923	1.5931	90.719	1.944E-08	5893.2	1
5	O 1(LDE1)	Al2O3_7kV	7.0	47.077	8.4033	107.449	1.944E-08	2133.0	1
6	Sr 3(PETH)	GH1	7.0	16.912	1.0392	219.802	1.813E-08	185.3	1
7	Na 5(TAP)	Albite_7kV	7.0	8.524	2.8846	129.404	2.009E-08	536.3	1
8	Si 1(TAP)	SiO2_7kV	7.0	46.746	1.1154	77.398	1.898E-08	5369.0	1
9	Zn 5(TAP)	GD_2_10kV	7.0	11.119	3.3968	133.201	2.008E-08	217.6	1

TTL 9

Fig. 53 Standard Data window

In the window of Fig. 53, you find details of the standard samples that you have selected: element name, channel, analyzing crystal, standard sample name, accelerating voltage, mass concentration, ZAF correction factor, spectrometer position (in mm), beam current, net X-ray intensity, and number of accumulations.

- Click on one of the following buttons: **Name**, **Acc.V**, **Mass(%)**, **STD Fac.**, **L Value**, **Current**, **Net int.**, and **Acm**. This enables you to alter the presently displayed value. However, the four items, **L Value**, **Current**, **Net int.**, and **Acm**, are only in effect for the elements measured with the WDS; they are not in effect for data measured with the EDS.
- Clicking on the **Takeoff** button at the upper left of the window allows you to change the X-ray take-off angle and perform off-line calculation.
- If you carried out accumulations on a standard sample measurement and want to obtain another intensity of X-rays by using some of the accumulated data, select the desired accumulated data, and click on the **Acm** button (☞ Fig. 54). The same operation can be done instead by using the Standard Sample Program.



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 24617.2 / 18

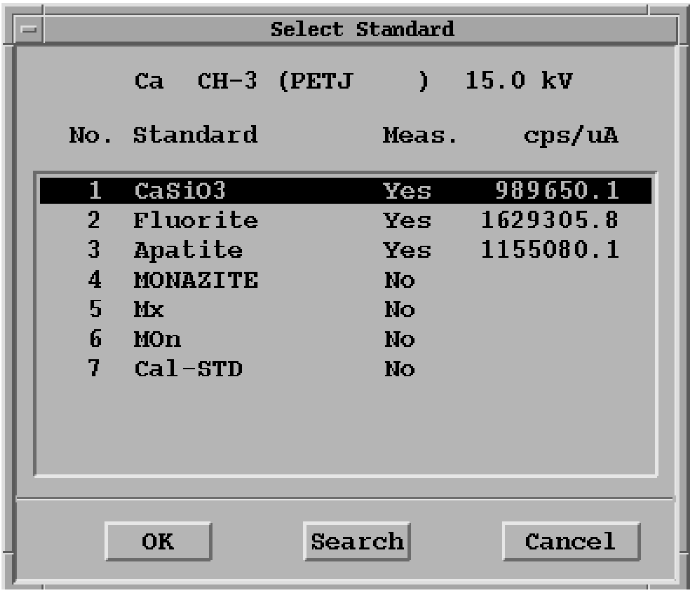
Sigma 1498.03

Fig. 54 Select Accumulations (Acm) window

- Clicking on the **Check used STDS** button displays the data of a standard sample that was used for an unknown sample measurement in the editor screen, allowing you to correct the data directly. When you click on this button once again, the unknown sample data whose standard samples were changed are searched for and if any are found, they are displayed. This function is used when different standard samples are used for different unknown samples. If you want to use the displayed standard sample data, first, click on the **Use them** button, then click on the **Save** button.
  Clicking on the **Save** button stores the information on the screen. However, the data on **Mass %** and **STD Fac**. are temporarily stored. Thus, note that the data will be deleted the next time you select a standard sample. Also, when using the metal mode, click on the **Save** button, and then the X-ray intensity for a 100% standard sample will be recorded as the intensity of CAL–STD, which can be used as the data of the standard samples in the correction of the semi-quantitative analysis.

  ✍ If a metal standard sample is selected in oxide analysis, the **Mass %** may exceed 100 %, in some cases (e.g., when you convert metal Al to Mass % in the form of Al_2O_3). This is just a way of indicating the concentration of the metal standard sample in terms of an oxide and has no influence on correction.

Fig. 55 shows the Select Standard window that is displayed by clicking on the **Name** button and then clicking on the desired name in the list under the button. If there are multiple sets of standard sample data (for the same accelerating voltage, channel, and analyzing crystal) satisfying the conditions, as shown in the figure, any one of them can be selected anew.



Ca CH-3 (PETJ) 15.0 kV

No.	Standard	Meas.	cps/uA
1	CaSiO3	Yes	989650.1
2	Fluorite	Yes	1629305.8
3	Apatite	Yes	1155080.1
4	MONAZITE	No	
5	Mx	No	
6	MOn	No	
7	Cal-STD	No	

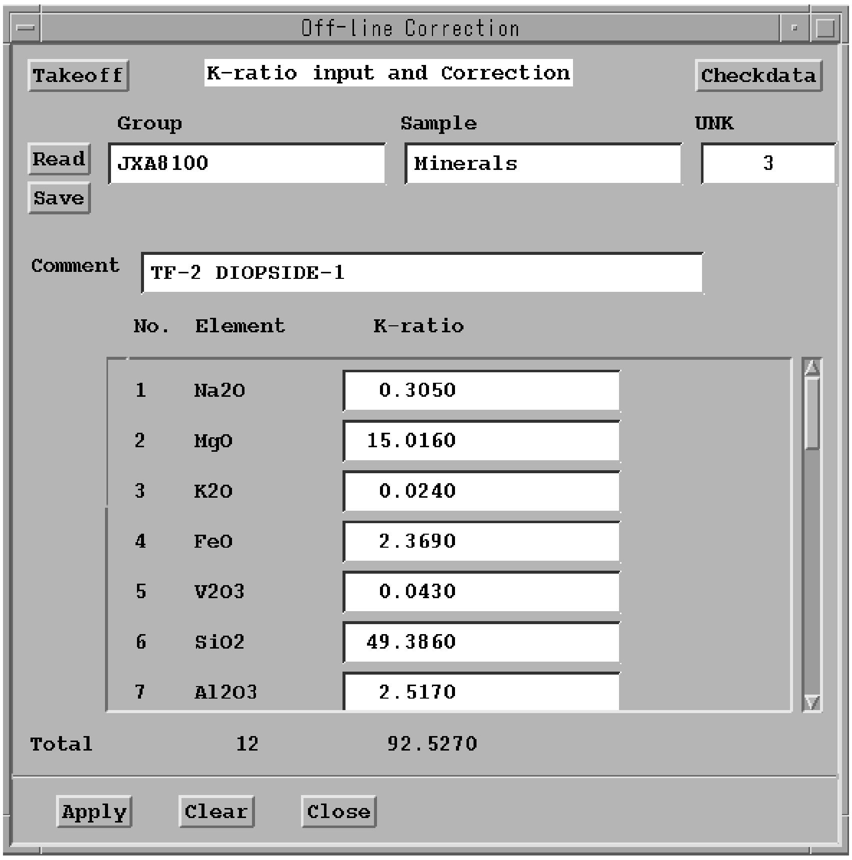
Fig. 55 Select Standard window

K-ratio Input & Correction

Enter K-ratio to perform the correction of data.

◆ Select **K-ratio Input & Correction** from the Off-line Correction menu.

The K-ratio Input & Correction window opens.



Group: JXA8100
Sample: Minerals
UNK: 3

Comment: TF-2 DIOPSIDE-1

No.	Element	K-ratio
1	Na2O	0.3050
2	MgO	15.0160
3	K2O	0.0240
4	FeO	2.3690
5	V2O3	0.0430
6	SiO2	49.3860
7	Al2O3	2.5170
Total	12	92.5270

Fig. 56 K-ratio Input & Correction window

- When this window is opened, the **Comment** input box and **K-ratio** input column are ready to be input. Type values there and click on the **Apply** button; then correction calculation will be performed.
- To read the K-ratios that are stored in a measured data file, click on the **Read** button, and enter a Group name, Sample name, and the number of an unknown sample; then a K-ratio, which is obtained from a measurement data, will be displayed. Correct the values, if necessary; then click on the **Apply** button to re-calculate.
  To store the results of re-correction, click on the **Save** button, and then specify the Group name, Sample name, and stage number.
- Click on the **Takeoff** button in the upper left corner of the window; then you can perform correction calculation by changing the take-off angle. This function is used to correct the data obtained with other models.
- Click on the **Check data** button in the upper right corner of the window; then the data of an unknown sample indicated in the **Read** boxes will be displayed.

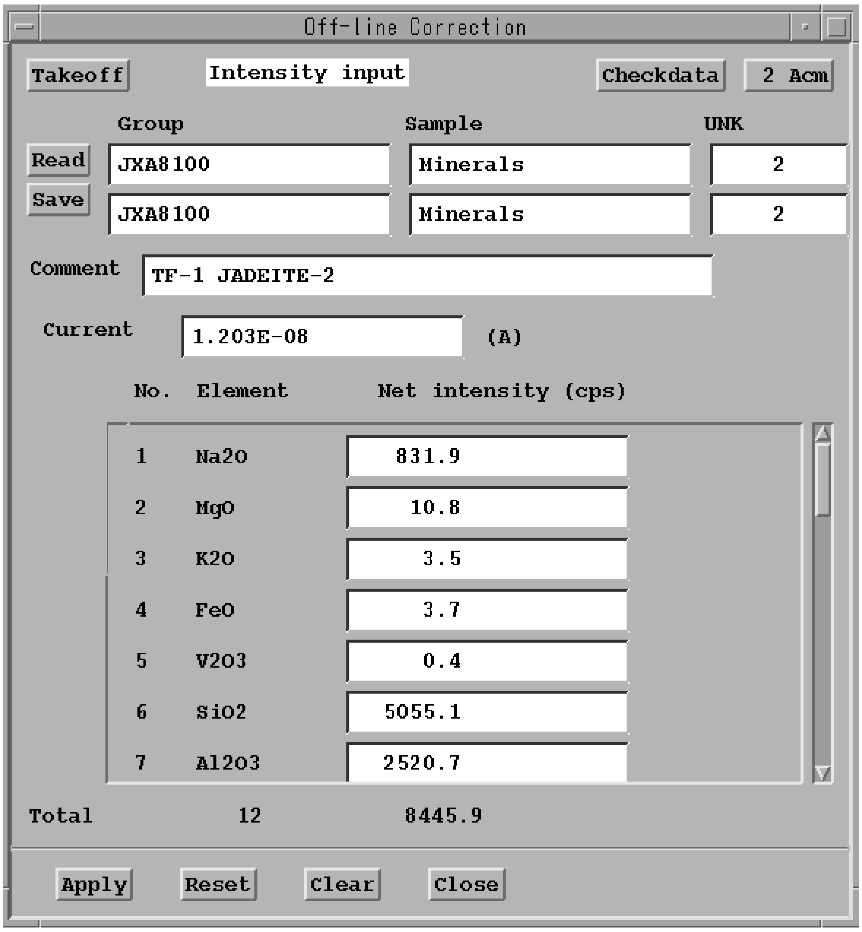
✍ When you want to change the correction method and perform re-correction, use **Intensity Input & Correction** as described in the next section, since the definition of K-ratio is different between the ZAF and the $\phi(\rho Z)$ methods.

Intensity Input & Correction

Enter the net X-ray intensity in this window to perform the correction of the data.

◆ Select **Intensity Input & Correction** from the Off-line Correction menu.

The Intensity Input & Correction window opens.



	Group	Sample	UNK
Read	JXA8100	Minerals	2
Save	JXA8100	Minerals	2

Comment: TF-1 JADEITE-2

Current: 1.203E-08 (A)

No.	Element	Net intensity (cps)
1	Na2O	831.9
2	MgO	10.8
3	K2O	3.5
4	FeO	3.7
5	V2O3	0.4
6	SiO2	5055.1
7	Al2O3	2520.7
Total	12	8445.9

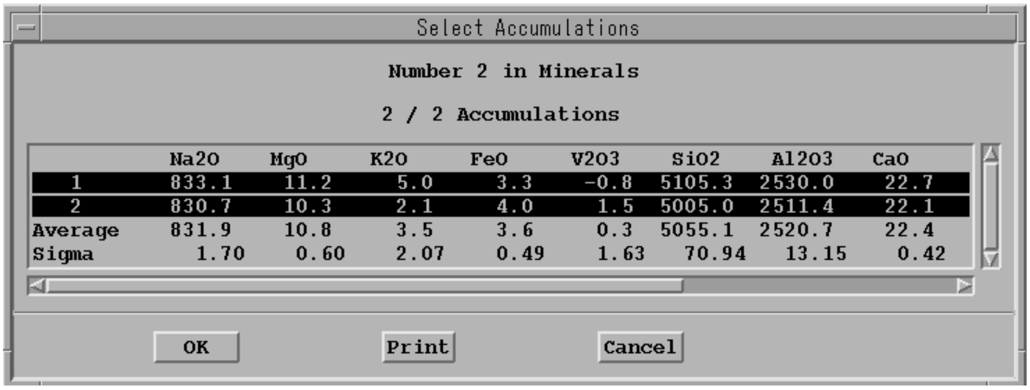
Fig. 57 Intensity Input & Correction window

This window shows, by default, the probe current and net X-ray intensity obtained from the data measured with the WDS.

- To perform correction calculation, type values in the **Net intensity** column, and then click on the **Apply** button.
- You can write or read data into or from a file by specifying the Group name, Sample name, and the number of an unknown sample in the input boxes at the right of the **Read** and **Save** buttons.

  If you want to keep previous measurement data, save the present results of re-correction after entering different names in the **Save** input boxes.

  If you do not want to save the present data, click on the **Save** button and make its input boxes blank. The data will not be written.
- If you carried out accumulations on an unknown sample measurement, click on the **Acm** button; then the Select Accumulations window opens as shown in Fig. 58.



Number 2 in Minerals

2 / 2 Accumulations

	Na2O	MgO	K2O	FeO	V2O3	SiO2	Al2O3	CaO
1	833.1	11.2	5.0	3.3	-0.8	5105.3	2530.0	22.7
2	830.7	10.3	2.1	4.0	1.5	5005.0	2511.4	22.1
Average	831.9	10.8	3.5	3.6	0.3	5055.1	2520.7	22.4
Sigma	1.70	0.60	2.07	0.49	1.63	70.94	13.15	0.42

Fig. 58 Select Accumulations (Acm) window

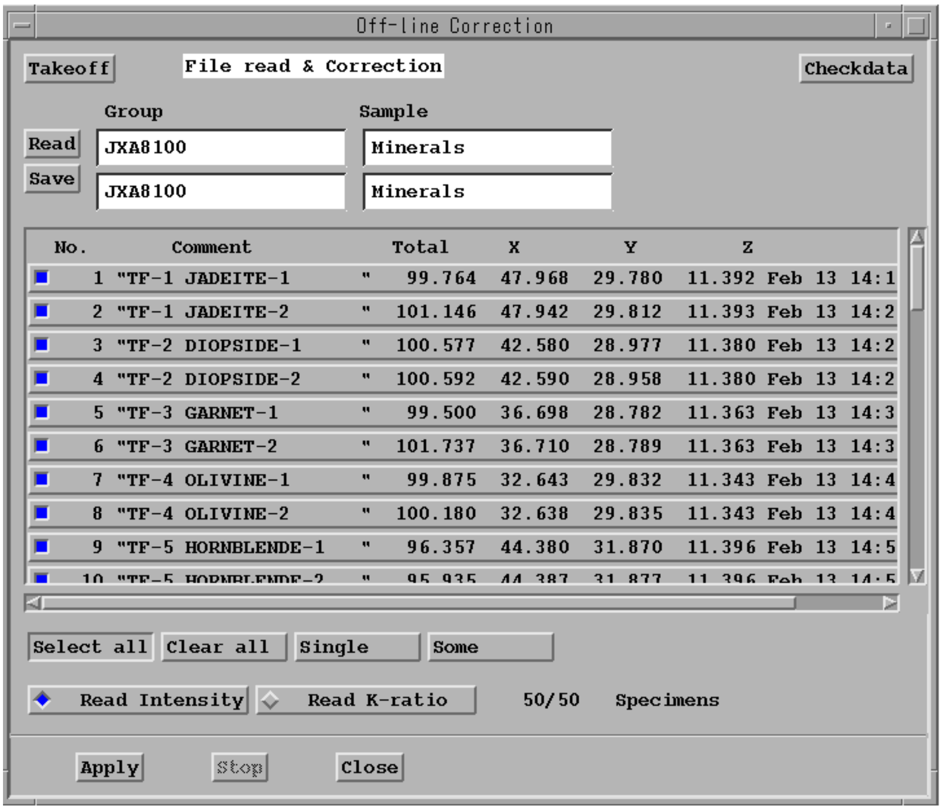
In this window, you can eliminate the intensities of X-rays that are unnecessary for the desired average calculation by selecting necessary accumulation values.

File Read & Correction

The File Read & Correction function allows you to perform re-correction on a series of data as a unit.

◆ Select **File Read & Correction** from the Off-line Correction menu.

The File Read & Correction window opens.



	Group	Sample
Read	JXA8100	Minerals
Save	JXA8100	Minerals

	No.	Comment	Total	X	Y	Z	
■	1	"TF-1 JADEITE-1 "	99.764	47.968	29.780	11.392	Feb 13 14:1
■	2	"TF-1 JADEITE-2 "	101.146	47.942	29.812	11.393	Feb 13 14:2
■	3	"TF-2 DIOPSIDE-1 "	100.577	42.580	28.977	11.380	Feb 13 14:2
■	4	"TF-2 DIOPSIDE-2 "	100.592	42.590	28.958	11.380	Feb 13 14:2
■	5	"TF-3 GARNET-1 "	99.500	36.698	28.782	11.363	Feb 13 14:3
■	6	"TF-3 GARNET-2 "	101.737	36.710	28.789	11.363	Feb 13 14:3
■	7	"TF-4 OLIVINE-1 "	99.875	32.643	29.832	11.343	Feb 13 14:4
■	8	"TF-4 OLIVINE-2 "	100.180	32.638	29.835	11.343	Feb 13 14:4
■	9	"TF-5 HORNBLENDE-1 "	96.357	44.380	31.870	11.396	Feb 13 14:5
■	10	"TF-5 HORNBLENDE-2 "	95.935	44.387	31.877	11.396	Feb 13 14:5

◆ Read Intensity ◇ Read K-ratio 50/50 Specimens

Fig. 59 File Read & Correction window

- This window shows, as a list, the comments, total values, and stage coordinates obtained from the present measured data. The samples with the buttons turned on, under **No.**, will be re-corrected.

  To select the desired buttons, click on the lines of desired samples in the list, or use some of **the Select all**, **Clear all**, **Single**, **Some** buttons.

  To execute re-correction calculation for the data, click on the **Apply** button.
- If you want to keep previous measurement data, save the present results of re-correction after entering different names in the **Save** input boxes.

  If you do not want to save present data, click on the **Save** button and make its input boxes blank. The data will not be written.
- Clicking on the **Read Intensity** button re-calculates the K-ratio from the X-ray intensity. This function is used after re-measurement of the standard sample.

  Clicking on the **Read K-ratio** button re-calculates using the K-ratio that was used for the measurement.

✍ When you want to change the correction method and perform re-correction, use **Read Intensity** in place of **Read K-ratio**, since the definition of K-ratio is different between the ZAF and the $\phi(\rho Z)$ methods.

6.4 Measuring by Calibration Curve Method

If you have obtained the relationship between mass concentration and intensity by measuring multiple standard samples, the composition of which you know in advance, you can perform highly precise quantitative correction. The procedure for measuring unknown samples by this method is almost the same as the ordinary method. Therefore, the measurement menu items specific to the Calibration Curve Method will be explained in detail; they are Correction Method, Standard Condition, Measurement, and Check Data.

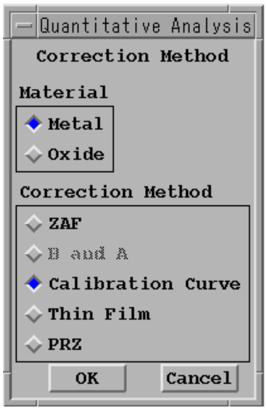
☞ For the measurement of standard samples, refer to the separate instruction manual of the Standard Sample Analysis Program.

6.4.1 Correction Method

The procedure for selecting the Calibration Curve Method is the following.

1. Select **Measurement–Corr. Method** from the Quantitative Analysis function window.

   The Correction Method window opens.



Quantitative Analysis

Correction Method

Material

● Metal
○ Oxide

Correction Method

○ ZAF
○ B and A
● Calibration Curve
○ Thin Film
○ PRZ

Fig. 60 Correction Method window

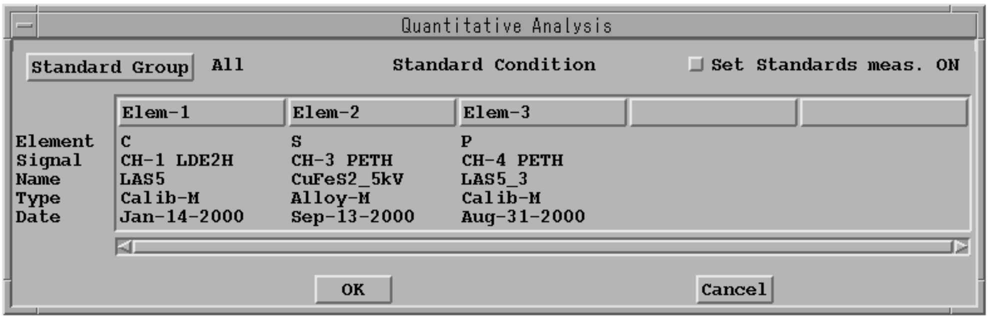
2. Click on **Calibration Curve** in the Correction Method window.

   The Calibration Curve has the orders from the first to the third. However, the order to be applied should be decided at the time of standard sample measurement.

6.4.2 Standard Condition

◆ Click on **Measurement–Standard Condition** from the Quantitative Analysis function window.

The Standard Condition window opens.



Standard Group All

☐ Set Standards meas. ON

	Elem-1	Elem-2	Elem-3		
Element	C	S	P		
Signal	CH-1 LDE2H	CH-3 PETH	CH-4 PETH		
Name	LAS5	CuFeS2_5kV	LAS5_3		
Type	Calib-M	Alloy-M	Calib-M		
Date	Jan-14-2000	Sep-13-2000	Aug-31-2000		

Fig. 61 Standard Condition window

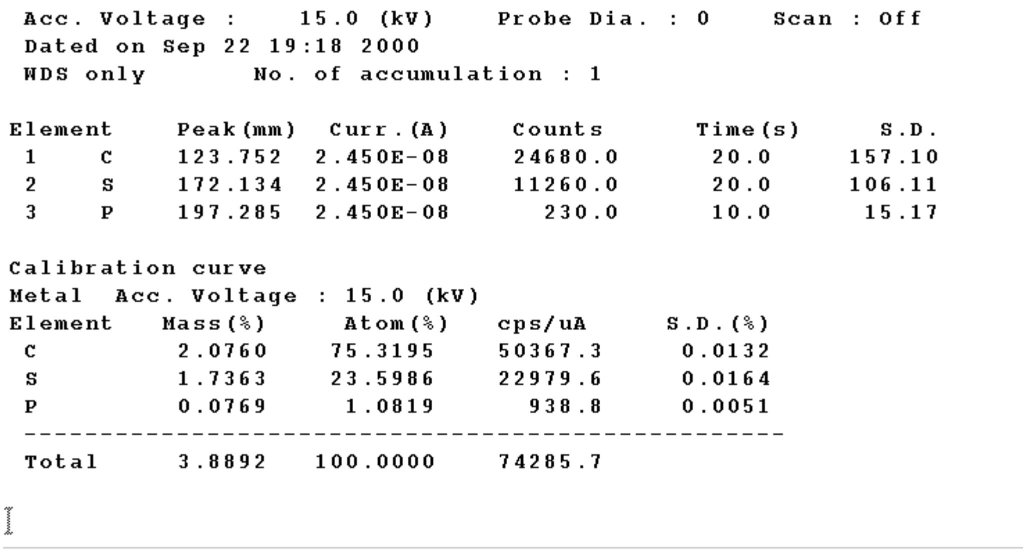
Select the standard sample that was measured using the calibration curve; then the type of the standard sample is displayed as **Calib-M** or **Calib-O**. If you select an ordinary standard sample, the A and B factors will be calculated by using the X-ray intensities of the peak and background.

✍ When you use an ordinary correction method that does not have calibration curve, such as ZAF, if you select the standard sample for calibration curve, mass concentration will be calculated based on calibration curve, even if you have selected another correction method that has no relationship with calibration curve. Furthermore, **Calibration** will be displayed as its calculation mode.

6.4.3 Measurement

Executing **Preset Measurement** or **One-by-One Measurement** lets you have the mass concentration calculated from X-ray intensity by Calibration Curve Correction Method. However, the background is neglected in the Calibration Curve Method, so select **Background Measurement–None** in the Additional Function window (☞ Fig. 30).

- Usually, the values of mass concentration (%) are displayed with up to three digits to the right of the decimal point.
- When you want to print data of a small quantity of constituents, first, correct a file of /opt/epma/phys/zaf.cnd by using the editor, and then increase the preset number 3 of the WT. Format item.



```
 Acc. Voltage :    15.0 (kV)    Probe Dia. : 0    Scan : Off
 Dated on Sep 22 19:18 2000
 WDS only       No. of accumulation : 1

Element     Peak(mm)  Curr.(A)    Counts      Time(s)     S.D.
 1    C     123.752  2.450E-08   24680.0      20.0     157.10
 2    S     172.134  2.450E-08   11260.0      20.0     106.11
 3    P     197.285  2.450E-08     230.0      10.0      15.17

Calibration curve
Metal  Acc. Voltage : 15.0 (kV)
Element    Mass(%)     Atom(%)    cps/uA     S.D.(%)
 C          2.0760    75.3195    50367.3     0.0132
 S          1.7363    23.5986    22979.6     0.0164
 P          0.0769     1.0819      938.8     0.0051
 --------------------------------------------------
 Total      3.8892   100.0000    74285.7
```

Fig. 62 An example of measurement results of Calibration Curve

6.4.4 Check Data

1. Select **Measurement–Corr. Method** from the Quantitative Analysis function window.

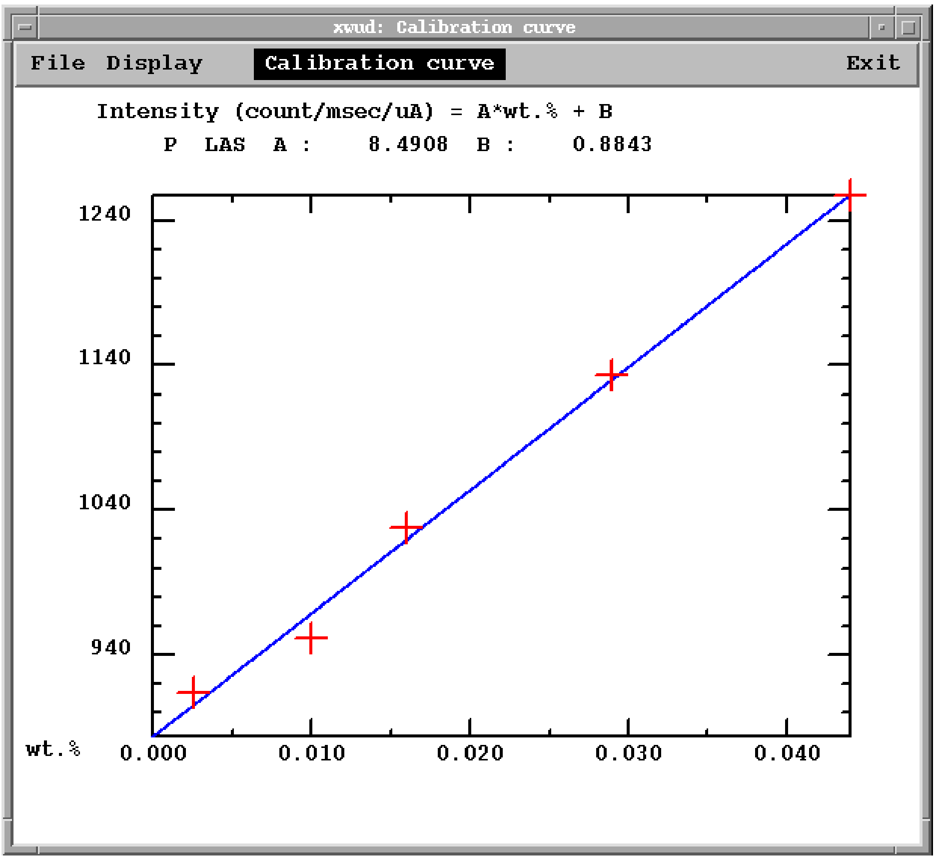
   The Correction Method window opens.

2. Select **Calibration Curve** from the Correction Method window.

3. Select **Standard Data** from the Off-line Correction menu.

   The Check Data window displaying a calibration curve opens as shown in Fig. 63. This window enables you to check the A and B factors. The procedure for this function is the same as that of **Check Data** of the Standard Sample Analysis Program.

   ☞ Refer to the separate instruction manual of the Standard Sample Analysis Program.



```
Intensity (count/msec/uA) = A*wt.% + B
     P  LAS  A :    8.4908  B :    0.8843
```

Fig. 63 Check Data window

7 SOPHISTICATED COORDINATE SETTING

A sophisticated method for setting coordinates is described. **Table Edit** displays the coordinates as a list in the Point Table Edit window. This window is used for modifying the coordinate table directly using the keyboard. **Line** Set is used when you want to set the coordinates sequentially in the form of line or map. When you want to convert the analysis results into line data or map data by using the ASCII conversion program, it is possible to display the data graphically. **Position Correction** is used when you want to utilize the coordinates that have already been set, and carry out coordinate conversion of up to 3 reference points. This method is useful for performing measurement of a sample taken out from the vacuum and for cyclic measurement.

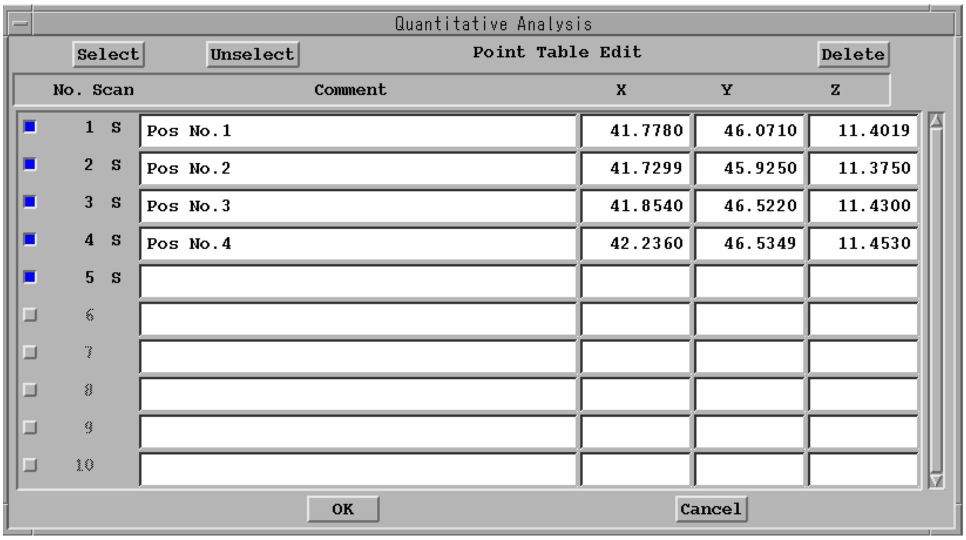
7.1 Table Edit

1. Select **Measurement–Stage Condition** from the Quantitative Analysis function window.

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

2. Click on the **Table Edit** button in the Stage Condition window.

The Point Table Edit window opens.



	No.	Scan	Comment	X	Y	Z
■	1	S	Pos No.1	41.7780	46.0710	11.4019
■	2	S	Pos No.2	41.7299	45.9250	11.3750
■	3	S	Pos No.3	41.8540	46.5220	11.4300
■	4	S	Pos No.4	42.2360	46.5349	11.4530
■	5	S				
☐	6					
☐	7					
☐	8					
☐	9					
☐	10					

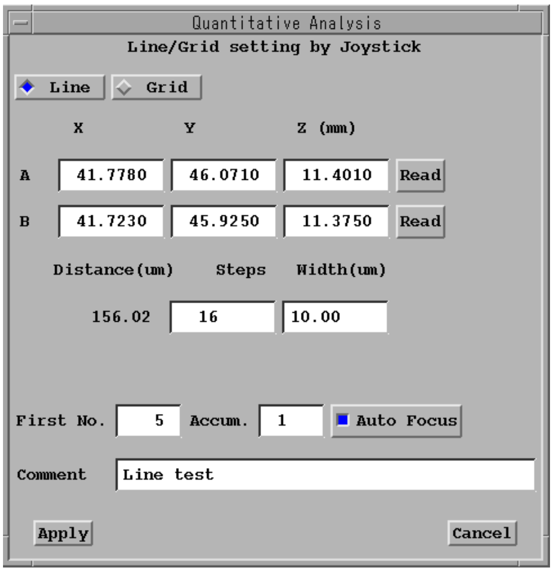
Fig. 64 Point Table Edit window

The Point Table Edit window is used for the following purposes: to modify the comment on the analysis point, to enter X, Y, Z coordinates using the keyboard, and to turn all measurements on or off at the same time. Clicking on the button at the left end of the stage position line turns measurement on or off. Also, comments and X, Y, Z coordinates are directly modified using the keyboard.

The **Select**, **Unselect** and **Delete** buttons allow you to turn measurement on or off and delete all points at the same time, by entering the desired first and last numbers and clicking on the **OK** button. When you modify the coordinates and click on the **OK** button, the modified coordinates are saved. Clicking on the **Cancel** button discards the modifications.

7.2 Line Set

1. Select **Measurement–Stage Condition** from the Quantitative Analysis function window.
   ☞ The Stage Condition window opens as shown in Fig. 20.
2. Click on the **Line Set** button in the Stage Condition window.
   The Line/Grid setting by Joystick window opens.



Line/Grid setting by Joystick

● Line ○ Grid

	X	Y	Z (mm)
A	41.7780	46.0710	11.4010
B	41.7230	45.9250	11.3750

Distance(um)	Steps	Width(um)
156.02	16	10.00

First No. 5 Accum. 1 ☑ Auto Focus

Comment Line test

Fig. 65 Line/Grid setting by Joystick window

Line Set is used to set the coordinate points in the form of a line or a grid. In this window, enter the start point and end point using the joystick, and specify the number of points to be measured or the distance between measurements. You can obtain the Z-coordinate of each point from comparison calculation. Also, it is possible to check and modify this Z-coordinate by using the **Pos. Input** afterwards.

Object	Function
Line/Grid	Select the method for setting the coordinate table from **Line** or **Grid**.
Read	When the coordinate points are in the form of a line, specify the start point and end point (A point and B point) using the joystick, and click on the **Read** button to read the coordinates. When the coordinate points are in the form of a grid, specify two diagonally opposite corners (points A and C) using the joystick, and click on the **Read** button to read the coordinates.. When the two points are determined, the distance between them is displayed and entering the values of **Steps** and **Width** becomes possible.
Steps	Specify the number of points to be measured. After you enter a number in the **Steps** input box, the distance between measurements is automatically calculated again.
Width	Specify the distance between measurements (in µm). After you enter a number in the **Width** input box, the number of points to be measured is automatically calculated again.

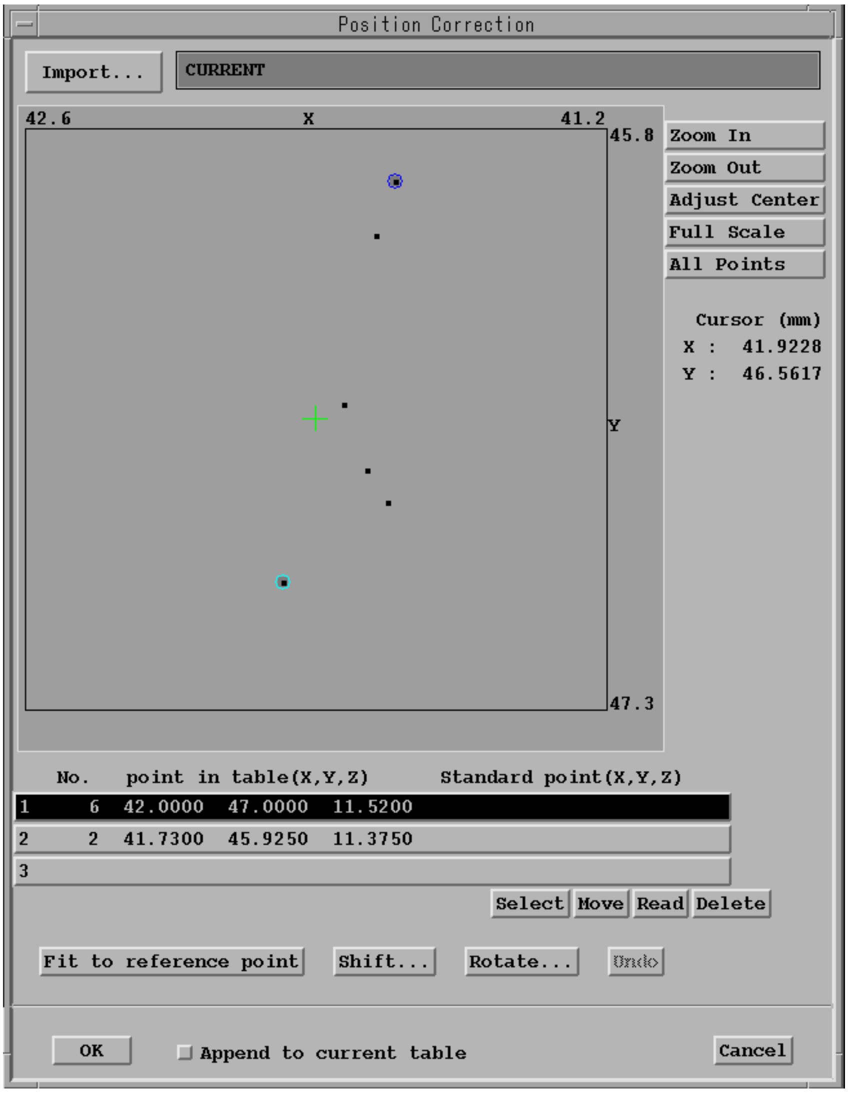
Object	Function
First No.	Enter the first number of the coordinate table.
Accum.	Specify the number of times to accumulate. If you specify one time or more, accumulation is executed at the same point (Fix mode).
Auto Focus (optional)	When an optional auto-focus device is provided, select **Auto Focus** if you want to execute automatic focusing of the stage prior to measurement.
Comment	Enter comments of up to 40 characters. When the setting of coordinates is in the linear form, “Line n” is added to the beginning of the comment. When in the form of a grid, “Grid n-m” is added.

✍ In both **Line**/**Grid** and **Width**, the start point is fixed and the end point is corrected to fit the calculation value.

7.3 Position Correction

When you want to perform measurement by using one of the coordinate patterns or the coordinates that were previously used for measurement, load the coordinate table, and correct the coordinate points by aligning up to 3 reference points using the joystick.
It executes parallel movement of the coordinates when one point is specified as a reference point. When two points are specified, it performs movement and rotation correction. When three points are specified, it performs Z-axis correction in addition to two-point correction.

1. Select **Measurement–Stage Condition** from the Quantitative Analysis function window.
   ☞ The Stage Condition window opens as shown in Fig. 20.
2. Click on the **Position Correction** button in the Stage Condition window.
   The Position Correction window opens.



CURRENT

Cursor (mm)
X : 41.9228
Y : 46.5617

No.	point in table(X,Y,Z)				Standard point(X,Y,Z)
1	6	42.0000	47.0000	11.5200	
2	2	41.7300	45.9250	11.3750	
3					

Fig. 66 Position Correction window

7.3.1 Calling the existing coordinate table

1. Click on the **Import** button in the Position Correction window.

   The Import window menu opens.

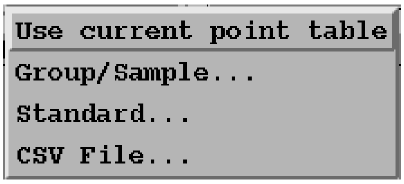


Fig. 67 Import window menu

2. If you want to open the coordinate table that was already set, select **Group/Sample** from the Import window menu.

   If you want to use the presently displayed coordinate table, select **Use current point table**.

   When the coordinate table is selected, each coordinate position is displayed graphically in the stage position display area of the Position Correction window.

7.3.2 Displaying coordinate positions

Since coordinate positions are shown within the entire scale of the stage (0 to 100 mm), in some cases, it is difficult to distinguish coordinate positions due to the fact that they are concentrated on one place on the screen. In these cases, change the display magnification by using **Zoom in**, **Zoom out**, **Full scale** and the other buttons. Clicking the mouse in the stage position display area displays the corresponding stage coordinates.

Button	Function
Zoom In	Use when you want to enlarge the display magnification. Clicking on this button highlights the button and changes the cursor shape. When you specify the area you want to enlarge by using the mouse, the specified area is enlarged. When you finish enlarging, click on this button again and cancel the highlight.
Zoom Out	When the display is enlarged, selecting **Zoom Out** reduces the magnification by one-half centered on the center of the screen.
Adjust Center	Clicking on this button displays the image centered on the present cursor position.
Full Scale	When you click on this button, the entire image is displayed regardless of the present magnification.
All Points	When you on click this button, all coordinate points are displayed again.

7.3.3 Setting reference point

◆ By clicking on the coordinate points displayed on the screen or specifying the coordinate number from the coordinate table, you can set the reference point.

✍ This operation cannot be done while the image is enlarged. Cancel the **Zoom In** button to perform this operation.

The reference table is displayed at the bottom of the Position Correction window.

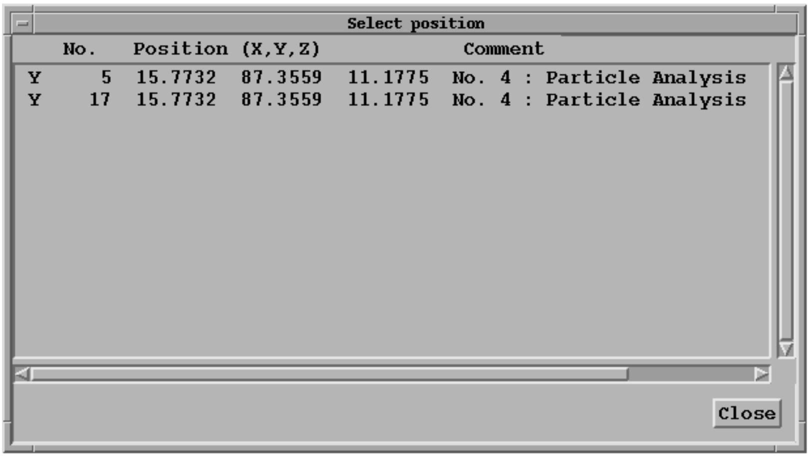
1. Select a reference point from 1 to 3 points that you want to record, and highlight it.
2. Click the mouse on the desired point on the screen.

   The Reference position candidate table window opens as shown in Fig. 68.

   When the multiple coordinate points are positioned in the vicinity of the clicked point, the list of these points is displayed in the Reference position candidate table window.
3. Select the list that contains the point you want to use as a reference point.

   The point is recorded as the reference point.

   If you specify a reference point that has already been recorded, the confirmation window, “**Change point**?” is displayed.



```
    No.  Position (X,Y,Z)           Comment
Y     5  15.7732  87.3559  11.1775  No. 4 : Particle Analysis
Y    17  15.7732  87.3559  11.1775  No. 4 : Particle Analysis
```

Fig. 68 Reference position candidate table window

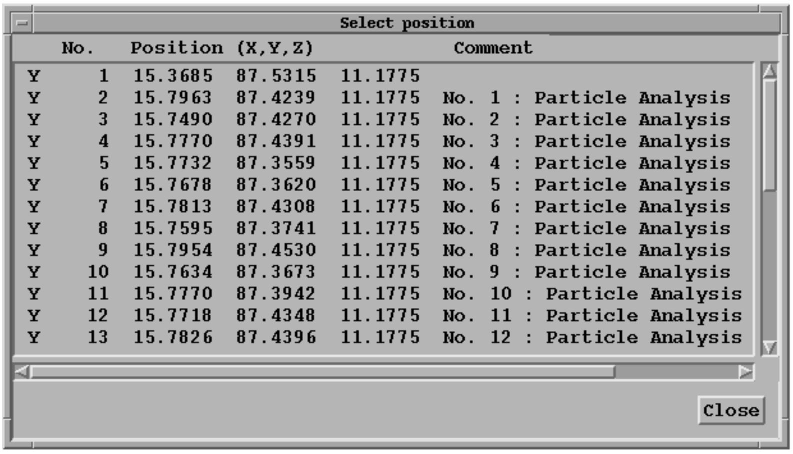
✍ It is also possible to specify the coordinate number from the coordinate table directly to set the reference point.

a. Click on the **Select** button in the Position Correction window.

   The Reference position selection window displaying the list of all the coordinate points that were recorded opens as shown in Fig. 69.

b. To set the reference point, select the desired coordinates from the list.

You can specify one to three reference points of the sample. It is recommended that you select a feature that is easy to find as a reference point.



```
                         Select position
  No.   Position (X,Y,Z)                Comment
Y    1  15.3685  87.5315  11.1775
Y    2  15.7963  87.4239  11.1775  No. 1 : Particle Analysis
Y    3  15.7490  87.4270  11.1775  No. 2 : Particle Analysis
Y    4  15.7770  87.4391  11.1775  No. 3 : Particle Analysis
Y    5  15.7732  87.3559  11.1775  No. 4 : Particle Analysis
Y    6  15.7678  87.3620  11.1775  No. 5 : Particle Analysis
Y    7  15.7813  87.4308  11.1775  No. 6 : Particle Analysis
Y    8  15.7595  87.3741  11.1775  No. 7 : Particle Analysis
Y    9  15.7954  87.4530  11.1775  No. 8 : Particle Analysis
Y   10  15.7634  87.3673  11.1775  No. 9 : Particle Analysis
Y   11  15.7770  87.3942  11.1775  No. 10 : Particle Analysis
Y   12  15.7718  87.4348  11.1775  No. 11 : Particle Analysis
Y   13  15.7826  87.4396  11.1775  No. 12 : Particle Analysis
```

Fig. 69 Reference position selection window

7.3.4 Erasing reference point

The procedure for erasing a set reference point is the following.

1. Select the desired reference point.

   It becomes highlighted.

2. Click on the **Delete** button.

   A confirmation window appears.

3. Click on the **OK** button.

   The selected reference point will be erased.

7.3.5 Setting correction amount for reference point

It is necessary to specify where the set reference points mentioned above are located on the equipment. If the deviation of the coordinates is small, this setting can be performed.

1. Highlight the desired reference point using the mouse and click on the **Move** button.

   The stage moves to the coordinate position.

2. By using the joystick, move the reference point to the center of the view, and click on the **Read** button.

   The amount of movement is calculated and the position of the moved reference point is displayed graphically on the screen.

3. Correct all remaining reference points in this way.

7.3.6 Executing correction

◆ After correcting all reference points, click on the **Fit to reference point** button.

All coordinate points are corrected and the coordinate points after movement are displayed graphically on the screen.

✍ If the position of a coordinate point is out of the range of the stage after correction is executed, the measurement mode is set to OFF.

7.3.7 Saving into the stage table

- When the results of the coordinate conversion mentioned above are reasonable, click on the **OK** button in the Position Correction window.

  The stage table is updated to reflect the results of conversion.
  When you click on the **Cancel** button, the conversion result is ignored and the coordinate table is not updated.

7.3.8 Other conversions

Parallel movement

When you know the distance to move in advance, the procedure for performing the parallel movement is the following.

1. Click on the **Shift**… button in the Position Correction window.
2. Enter the correction amounts for the X, Y, and Z axes in this window and click on the **OK** button.

   Each coordinate point moves in parallel according to the specified values.

Rotation

When you want to rotate coordinate points, the procedure for performing the rotation is the following.

1. Click on the **Rotate**… button in the Position Correction window.
2. Enter the X and Y coordinates of the rotation center, and a rotation angle in this window, and click on the OK button.

   Each coordinate point rotates according to the specified values. At this time, positive rotation angles are clockwise rotation.

8 MEASURING UNKNOWN SAMPLES

Measurement of an unknown sample is performed according to the following procedure. Set the electron optical conditions such as accelerating voltage and move the sample to the analysis position. Then, stop the beam irradiation, measure the beam current, restart the beam irradiation, and perform peak search.

8.1 Peak Search of Unknown Samples

When measuring an unknown sample, check the peak intensity of the sample before executing peak search. If the peak intensity is lower than that listed in the table below, peak search is not executed. This is because when the peak intensity is low, the counting error in X-rays is large, and the program judges that performing peak search is meaningless.

Accelerating voltage (kV)	Count rate (cps)
10	100
15	100
20	200
25	300
30	400

Peak search is performed based on the peak search number. For an ordinary unknown sample, it is enough that this number be 1. By default, the peak search conditions are defined as follows.

Number	Multiple of FWHM	Number of points	Sampling time	Mode
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	No

When you want to change parameters such as number of points and sampling time, do so using **Check Pksk parameter** in the peak search monitor window. However, if you change these parameters, peak search might fail, so it is recommended that only a skilled person do this.
The FWHM used in the above table is calculated using the following equation.

$$\text{FWHM} = (A \times L\ (\text{mm}) + B) \times \text{Pksk}$$

You can find the values of the parameters A and B in **Crystal defaults** in the sub-menu window of **Check Config** in the JEOL menu. (In this window, the fourth and fifth parameters are A and B, and the seventh parameter is Pksk.) If you want to change FWHM, modify it by changing Pksk. Do not modify it by changing A and B because these parameters are also used in qualitative analysis, and therefore, they might influence the results of qualitative identification of elements.

8.2 Asynchronous Measurement and Synchronous Measurement

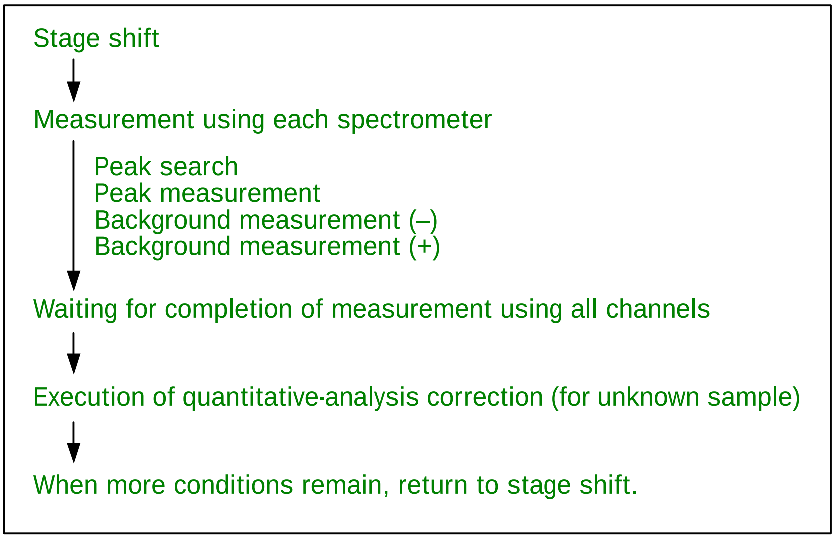
8.2.1 Asynchronous measurement

Ordinary measurement is asynchronous measurement, in which each spectrometer operates independently.

After peak search ends, the intensities of X-rays are measured in the following order: peak position, low-angle background and high-angle background. When a numerical value is entered in **Accumulation**, measurement is repeated on each point in the order mentioned above, and the average value is obtained. However, peak search is performed only on the first point. When measurements other than peak search and background measurement are specified in **Additional Function**, they take priority over peak search and background measurement.

After all elements are measured, measurement conditions, standard sample conditions and the intensity of X-rays in an unknown sample are output, and the result of correction is output.

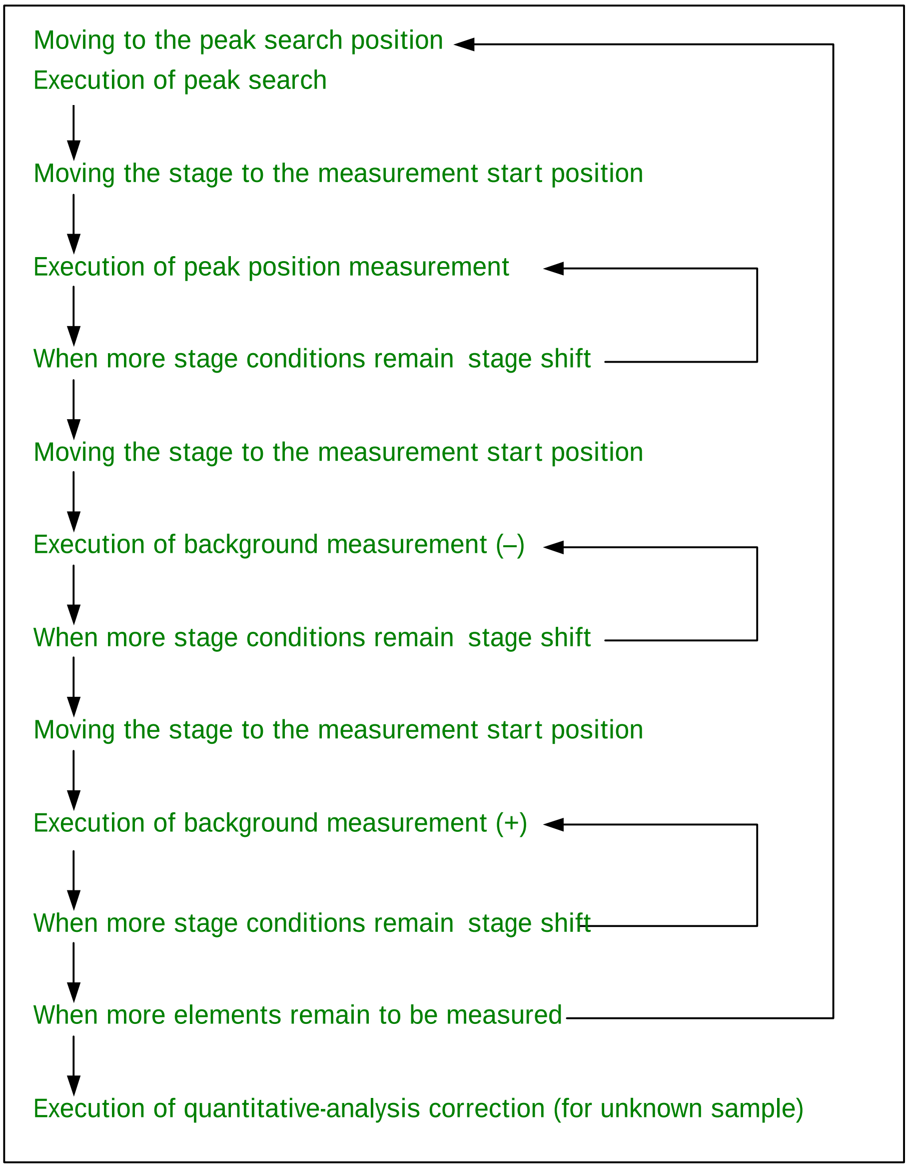
Asynchronous operation flowchart



8.2.2 Synchronous measurement

When **Synchronous** measurement is selected in the Additional Function window, measurement is executed while the stage position is moved with the spectrometer being fixed. The elements are measured in each round in the order you specified using the sequence in the WDS Element Condition window. When you specify this measurement mode, peak-search measurement is performed on only the first sample.

Synchronous operation flowchart



8.3 Method for Calculating Parameters

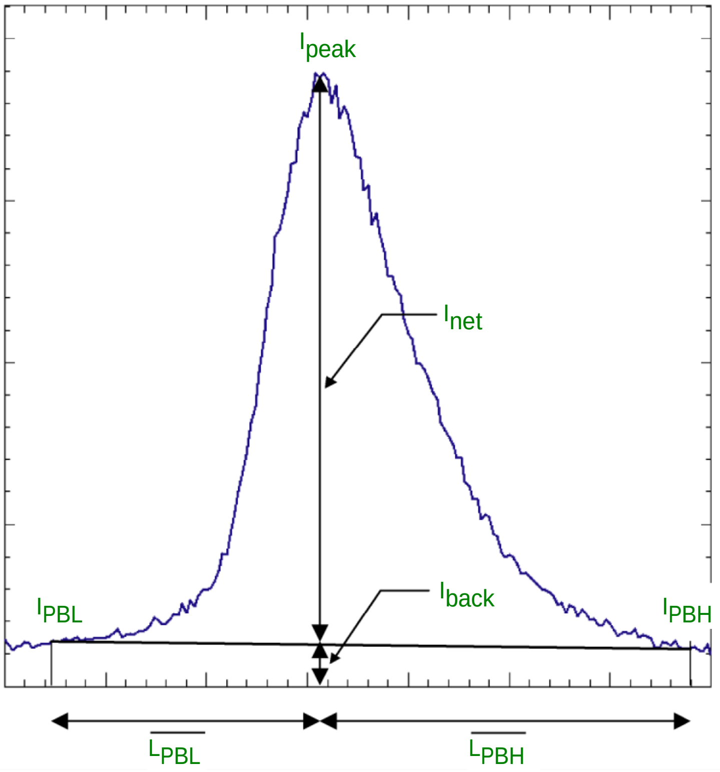


Fig. 70 Relation between peak and background

The parameters are calculated using the following equations.
The parameters shown below are ordinarily obtained with 1σ. If you want to change the coefficient of σ from 1 to 3, open the file /opt/epma/phys/zaf.cnd using the editor, and change the setting of Sigma_factor from 1 to 3.

- 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.

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

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

- Detection limit

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

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

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

8.4 Area Intensity Measurement/FIT Mode Measurement

By performing the following operation, you can select either the following area intensity measurement or FIT mode measurement.

Area intensity measurement:	This is a method of handling X-ray intensity in terms of spectrum area.
FIT mode measurement:	This is a method of handling the maximum value or average value of X-ray intensity calculated from the values measured at five points while the part of the spectrum in the vicinity of the peak position is shifted, in terms of X-ray peak intensity. It is used instead of the traditional single point peak measurement.

8.4.1 Area intensity measurement

■ Measuring area intensity

1. Measure the X-ray intensity while moving the spectrometer with a given step size; find the peak position.

2. Add the X-ray intensity (in cps) between the specified low angle background (bg (–)) and the specified high angle background (bg (+)).

3. Multiply the step width by the added X-ray intensity.

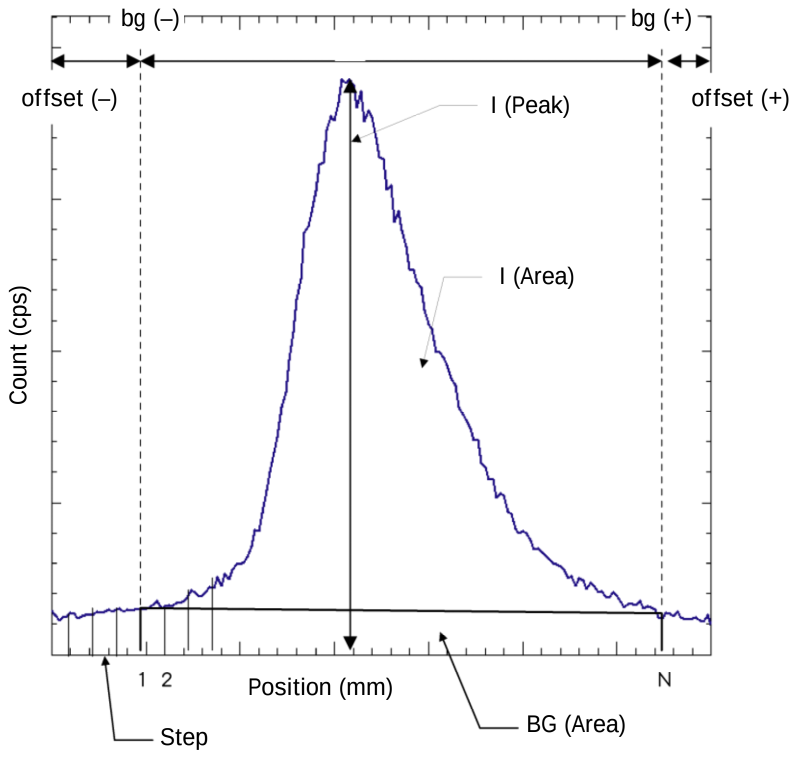


Fig. 71 Measuring area intensity

Calculating area intensity

Carry out calculation using the following procedure.

1. Calculate the average X-ray intensity of ten points between bg (–) and bg (+).

$$Xavg(-) = \sum_{n=-5}^{4} X(n)/10 \qquad \text{[cps]}$$

$$Xavg(+) = \sum_{n=N-5}^{N+4} X(n)/10 \qquad \text{[cps]}$$

2. Calculate the slope and intercept of the BG straight line between bg (–) and bg (+).

$$BG\_A = \frac{Xavg(+) - Xavg(-)}{N-1} \qquad \text{Slope}$$

$$BG\_B = Xavg(+) - BG\_A \times N \qquad \text{Intercept}$$

3. Calculate the background area.

$$BG(Area) = step\left[\sum_{n=1}^{N} (BG\_A \times N + BG\_B)\right] \qquad \text{[cps}\cdot\text{mm]}$$

4. Calculate the peak area.

$$I(Area) = \left(\sum_{n=1}^{N} X(n)\right) \times step - BG(Area) \qquad \text{[cps}\cdot\text{mm]}$$

X (n):	X-ray count at the nth point (cps)
X (1):	X-ray count at the background (–) position (cps)
X (N):	X-ray count at the background (+) position (cps)
bg (–):	Low angle background position (mm)
bg (+):	High angle background position (mm)
I (Peak):	X-ray count at the peak position (cps)
BG (Peak):	Background count at the peak position (cps)
I (Area):	X-ray area between bg (–) and bg (+) (cps·mm)
BG (Area):	Background area between bg (–) and bg (+) (cps·mm)
step:	Measurement step size (mm)
Xavg (–):	Average X-ray count of low angle side background (cps)
Xavg (+):	Average X-ray count of high angle side background (cps)

8.4.2 FIT mode

The FIT mode is a method in which 5-point X-ray intensity measurement is performed while the part of the spectrum in the vicinity of the peak position is shifted by the step size (by default, 100 μm); then the average value I (avg) or the maximum value I (max) is calculated and taken as the peak intensity.

The background is measured using the same method as that described.

■ Measurement using FIT mode

1. Perform a peak search, and move to the peak position.
2. While shifting near the peak position, measure the X-ray intensities, and calculate the average or maximum X-ray intensity.
3. Move to the low-angle side of the background, and measure the X-ray intensity.
4. Move to the high-angle side of the background, and measure the X-ray intensity.

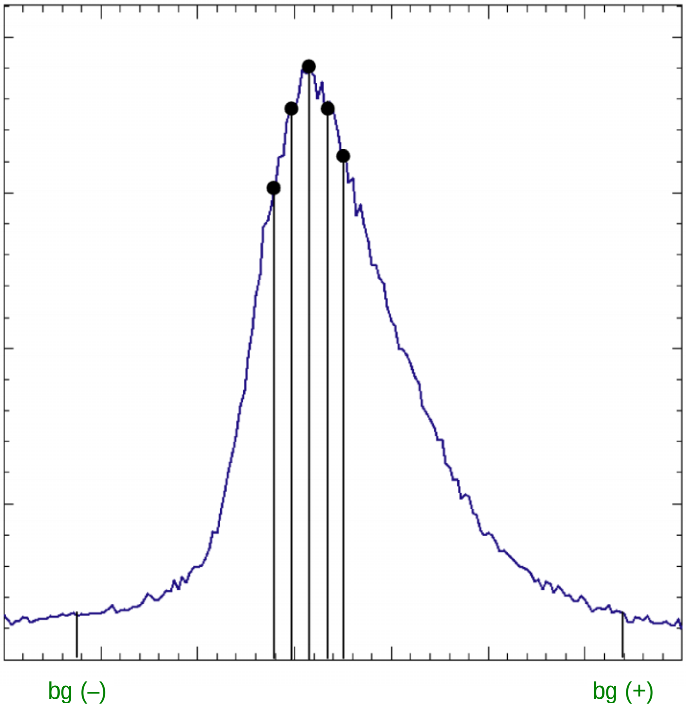


Fig. 72 Measurement using FIT mode

■ Calculating FIT intensity

If you define the 5-point intensity data measured in the vicinity of the peak as I [1] to I [5],

- The average intensity in the Fit mode is as follows:

$$I(avg) = \frac{(I[1] + I[2] + ... + I[5])}{5}$$

- The maximum intensity in the Fit mode are as follows:

$$I(\max) = Max(I[1], I[2]..., I[5])$$

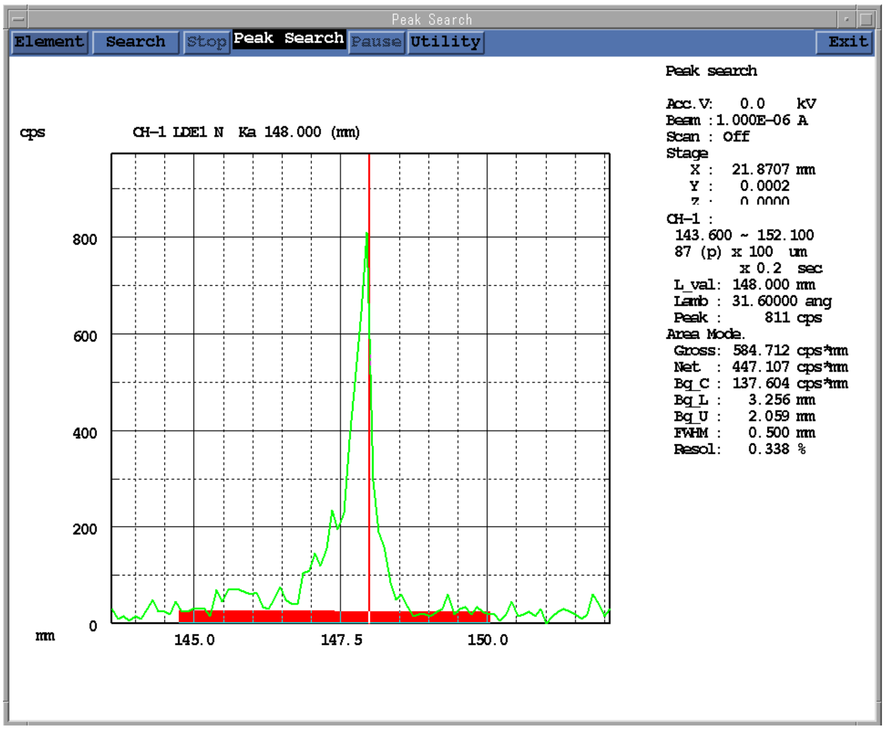
8.4.3 Obtaining area intensity

■ Determining the area intensity scanning conditions

Carry out the following procedure.

1. Click on **Peak Search** in the Monitor menu.

   The Peak Search window opens.



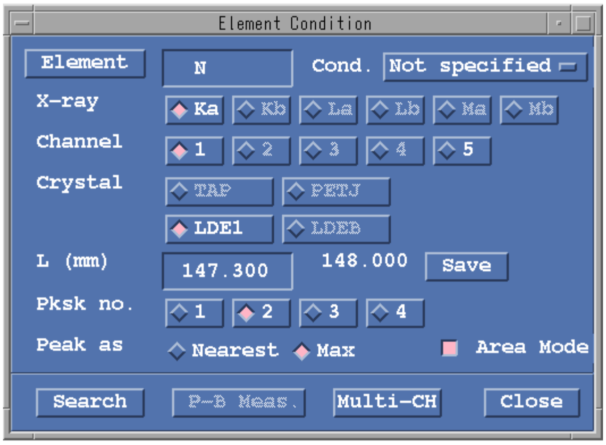
```
Peak search

Acc.V:    0.0    kV
Beam :1.000E-06 A
Scan :  Off
Stage
   X :   21.8707 mm
   Y :    0.0002
   Z :    0.0000
CH-1 :
 143.600 ~ 152.100
 87 (p) x 100  um
          x 0.2  sec
 L_val:  148.000 mm
 Lamb :  31.60000 ang
 Peak :       811 cps
Area Mode.
 Gross:  584.712 cps*mm
 Net  :  447.107 cps*mm
 Bg_C :  137.604 cps*mm
 Bg_L :    3.256 mm
 Bg_U :    2.059 mm
 FWHM :    0.500 mm
 Resol:    0.338 %
```

Fig. 73 Peak Search window

2. Click on the **Element** button.

   The Element Condition window opens.

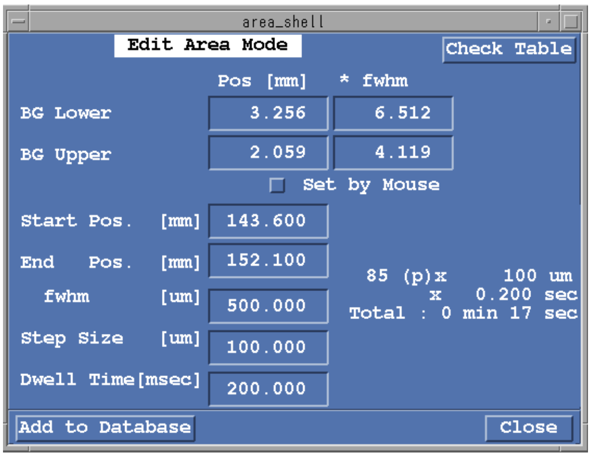


Element: N
Cond.: Not specified
X-ray: ◆ Ka
Channel: ◆ 1
Crystal: ◆ LDE1
L (mm): 147.300 148.000
Pksk no.: ◆ 2
Peak as: ◆ Max
☑ Area Mode

Fig. 74 Element Condition window

3. Enter the desired element name, and then click on the desired **X-ray**, **Channel** and **Crystal** buttons.
4. Click on the **Area Mode** button to activate the mode.
5. Click on the **Search** button and carry out area intensity measurement.
6. Select **Control Area Pksk** from the **Utility** menu in the Peak Search window (☞ Fig. 73).

   The Edit Area Mode window opens.



Pos [mm] * fwhm

BG Lower 3.256 6.512

BG Upper 2.059 4.119

☐ Set by Mouse

Start Pos. [mm] 143.600

End Pos. [mm] 152.100

fwhm [um] 500.000

Step Size [um] 100.000

Dwell Time[msec] 200.000

85 (p)x 100 um
x 0.200 sec
Total : 0 min 17 sec

Fig. 75 Edit Area Mode window

- Determining the background position (integration start position and end position)
  - Specifying the background position by entering values

    Position the mouse pointer in the **BG Lower** and **BG Upper** input boxes, and type the position of the background in mm.
  - Specifying the background position by using the mouse

    Click on the **Set by Mouse** button, and then click on the low-angle side and on the high-angle side in the window in which the spectrum is displayed.
- Determining the start and end positions for scanning

  Enter the start position in **Start Pos. [mm]** and the end position in **End Pos. [mm]**.
- Determining FWHM (Full Width at Half Maximum)

  The default is set to 500 μm. This value has no influence on measurement.
- Determining Step Size [um]

  Enter the step size to be used for measurement.
- Determining Dwell Time [msec]

  Enter the measurement time per point.

7. Click on the **Add to Database** button to store the present conditions.

   If you do not store these conditions, the previous conditions will be used.

   ✍ When you want to refer to the database file, click on the **Check Table** button. Then, you can check the contents of the file.

■ Specifying area intensity by entering quantitative analysis conditions

1. Select **Analysis** from the EPMA Main Menu to open the **Analysis** menu, and then click on **Quantitative Analysis** or **Standard Analysis** to open the window for unknown samples or standard samples in the quantitative analysis.
2. Click on the **Time/Count/Area** button in the WDS Element Data Table window (☞ Sect. 6.1.3, Fig. 13).

   The Time/Count/Area window opens.

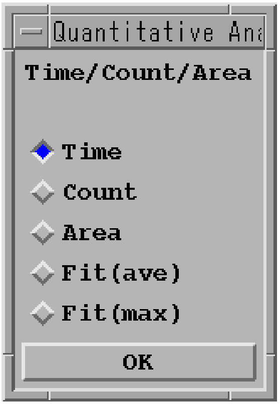


Fig. 76 Time/Count/Area window

3. Click on the **Area** button.

   The conditions of the area intensity created using the Peak Search window are loaded and changed automatically.

   Mes. Time (sec) can be changed for each point. Also, the measurement range for area intensity can be changed using **Back(+)** and **Back(–)**.

8.4.4 Database file for area quantitative analysis

The area intensity table created by using the peak search window (☞ Fig. 75) is stored in the file **/users.epma/phys/fwhm.asc**.

This file consists of the element name, X-ray name, crystal name, and X-ray order, to be used for searching for conditions, and also the full width at half maximum, magnification of BG (–), magnification of BG (+), step size, measurement time, magnification of offset (–), and magnification of offset (+), to be used for determining the scanning conditions.

This file can be changed using **Control Area Pksk**.

#1:	Element name	Cu, C, B
#2:	X-ray name	∗, KA, KB, LA
#3:	Crystal name	∗, TAP, PET, STE
#4:	X-ray order	∗, 1, 2, 3 ...
#5:	Full width at half maximum [μm]	fwhm
#6:	BG (–) magnification [–]	bg_mag (–)
#7:	BG (+) magnification [+]	bg_mag (+)
#8:	Step size [μm]	step
#9:	Measurement time [ms]	dtime
#10:	Offset (–) magnification [–]	of_mag (–)
#11:	Offset (+) magnification [–]	of_mag (+)

#1	#2	#3	#4	#5	#6	#7	#8	#9	#10	#11
Na	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
Mg	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
Al	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
Si	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
P	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
S	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
Cl	∗	∗	∗	500. 0	2. 000	2. 000	100. 0	200	0. 000	0. 000
Zn	KA	LIF	∗	200. 0	4. 513	5. 039	30. 0	200	3. 436	7. 112
Cu	KA	LIF	∗	200. 0	2. 874	4. 166	30. 0	200	12. 87	15. 889
C	KA	LDE1	∗	354. 0	2. 664	2. 212	50. 0	200	2. 635	3. 689

✍ To search for X-ray name, crystal name, or X-ray order, the asterisk (∗) is used. If an item is marked with an asterisk, it means that the item matches all of the search conditions. Consequently, if all items (#2, #3 and #4) bear the asterisk, it means that the items are the default values.

Magnification is defined as the background or offset value indicated as a multiple of the full width at half maximum.

Offset indicates the magnification from the background, as indicated by offset (–) in Fig. 71.

The width of the scan (L in mm) is calculated from the following equation:

$$L = \frac{\{of\_mag(-) + bg\_mag(-) + bg\_mag(+) + of\_mag(+)\} \times fwhm}{1000.0}$$

8.4.5 Changing FIT mode conditions (step width and number of measurement points)

The step width and the number of measurement points for FIT mode measurement are in the file **/opt/epma/phys/fitmode.asc**.

This file consists of the element name, X-ray name, crystal name, and X-ray order, to be used for searching for conditions, and also the full width at half maximum, number of measurement points and step size to be used for determining the scanning conditions.

To change the conditions, execute the following commands.

```
> dtpad         /opt/epma/phys/fitmode.asc
#1    #2        #3        #4        #5        #6        #7
Si    *         *         *         500.0     5         100.0
P     *         *         *         500.0     5         100.0
S     *         *         *         500.0     5         100.0
Cl    *         *         *         500.0     5         100.0
Ar    *         *         *         500.0     5         100.0
K     *         *         *         500.0     5         100.0
Ca    *         *         *         500.0     5         100.0
Sc    *         *         *         500.0     5         100.0
Ti    *         *         *         500.0     5         100.0
V     *         *         *         500.0     5         100.0
Ca    KA        PET       *         500.0     6         200.0
```

#1:	Element name	Cu, Zn, Mg, etc.
#2:	X-ray name	KA, MA, LA, KB, etc.
#3:	Crystal name	TAP, LIF, etc.
#4:	X-ray order	Integers 1, 2, 3, etc.
#5:	fwhm [μm]	Full width at half maximum. Not used.
#6:	Number of measurement points	
#7:	Step size [μm]	