C

CONTENTS

# XM-27330

# WD/ED BASIC SOFTWARE, EDS CALIBRATION PROGRAM

IXM-27330-1CL (608501)
DEC2001-03210211
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.<br>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 (–).<br>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

C

# 1 GENERAL

This program is intended for operating the EDS analyzer, calibrating the EDS energy, measuring the EDS resolution, and calibrating the EDS apertures.

# 2 SPECIFICATIONS

## ■ EDS analyzer settings

Window type, bias voltage, detector geometry
Linear amplifier time constant, linear amplifier gain/zero, lower level discriminator (has an automatic function), fast discriminator (has an automatic function)
Alarm function, pile-up rejecter function
Analyzer present values monitoring function

## ■ EDS energy calibration

Set the coarse gain, fine gain and zero value under each of the four measurement conditions of the EDS.
You can do this by selecting either “complete calibration specification” or “partial calibration specification”.

## ■ EDS resolution measurement

Obtain the FWHM of the characteristic X-ray peak for the specified element.
You can also specify subtraction of the background.

## ■ EDS aperture calibration

Obtain the X-ray intensity ratio by switching the EDS aperture.

# 3 EDS ANALYZER

The EDS Analyzer Main window enables you to monitor the present set values of the EDS subsystem or set the detector.

## 3.1 Operation

**1.** Select **EDS** from the EPMA Main Menu.

The EDS menu opens as shown in Fig.1.

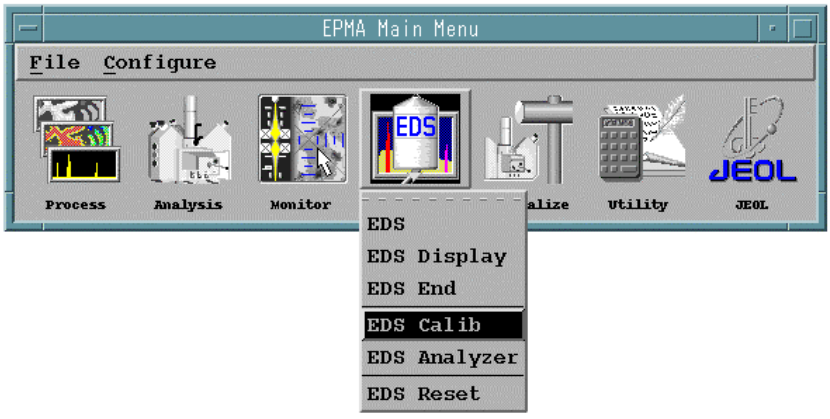


**Fig. 1 EDS menu**

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

The EDS Analyzer Main window opens as shown in Fig. 2.

The upper half of the screen shows the present set values of the subsystem. The lower half is the subsystem operation screen.

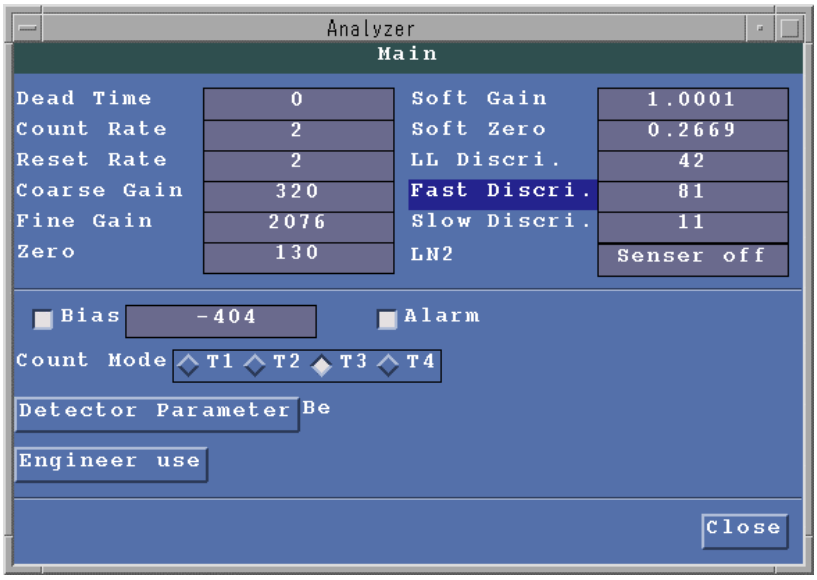


**Fig. 2 EDS Analyzer Main window**

## 3.2 Description of Displayed Items

| Object | Function |
|---|---|
| **Dead Time** | Indicates, as a percentage of the total pulses, the fraction of the X-ray pulses striking the EDS detector that could not be detected as effective pulses at the EDS detector due to overlapping of X-rays with each other. Normally, a value between 20% and 40% is used. |
| **Count Rate** | Indicates the total quantity (in cps) of X-rays detected by the EDS detector each second. |
| **Reset Rate** | The preamplifier used for the EDS detector is a pulse optical feedback type charge amplifier. In order to maintain it in an appropriate condition, it is repeatedly reset by light according to the intensity of the detected X-rays. The value displayed in this box indicates the number of times per second (in cps) that the preamplifier is reset.<br>✍ Normally, when X-rays do not strike the EDS detector, the preamplifier is reset at the rate of 1 cps or less. When X-rays are detected, the rate at which the preamplifier is reset increases in proportion to the intensity of the X-rays. |
| **Coarse Gain** | Indicates the present coarse gain of the linear amplifier.<br>✍ The values from **Coarse Gain** to **Slow Discri.** are determined by EDS calibration. You can set them using the EDS Energy Calibration window. |
| **Fine Gain** | Indicates the present fine gain of the linear amplifier. |
| **Zero** | Indicates the present zero level of the linear amplifier. |
| **Soft Gain** | Used to perform, in software, fine gain adjustment that cannot be done by **Coarse Gain** or **Fine Gain**. |
| **Soft Zero** | Used to perform, in software, fine zero level adjustment that cannot be done by **Zero**. |
| **LL Discri.** | Indicates the value of Lower Level Discriminator. |
| **Fast Discri.** | Indicates the value of Fast Discriminator. |
| **Slow Discri.** | Indicates the value of Slow Discriminator.<br>✍ **Fast Discri.** or **Slow Discri.** is displayed in a dark color. This indicates which output is being displayed as **Count Rate**. Usually, **Fast Discri.** is displayed in a dark color indicating that Count Rate shows **Fast Discri.** |
| **LN2** | Indicates the status of the liquid nitrogen sensor. |

## 3.3 Description of Control Buttons

| Button | Function |
|---|---|
| **Bias** | Indicates the voltage (V) presently applied to the EDS detector.<br>To turn off the high voltage, click on the button on the left side of Bias.<br>To re-apply the high voltage, click on the button once again . |
| **Alarm** | If the liquid nitrogen runs out or the EDS subsystem detects an abnormality, an alarm sounds. You can stop the alarm by clicking on the **Alarm** button it .<br>To re-activate the alarm, click on the button once again. |
| **Count Mode** | The count mode has four stages, **T1**, **T2**, **T3** and **T4**. They are used to specify whether to acquire X-rays at a high resolution or at a high counting rate.<br>To acquire X-rays at a high energy resolution as for quantitative analysis, select **T3** or **T4**.<br>To perform measurement at a high counting rate as for area analysis, select **T1** or **T2**.<br>✍ Count modes **T1**, **T2**, **T3** and **T4** correspond to time constants of the Slow system linear amplifier of 14 μs, 60 μs, 120 μs and 400 μs. |
| **Detector Parameter** | Sets the parameters of the detector. When you click on this button, the Detector Parameter window of Fig. 3 opens.<br>✍ Once you set all the parameters of the Detector Parameter window when the equipment is installed, there is normally no need to subsequently change them.<br>✍ The values of the detector parameters are copied to the spectrum data file that is created at the start of EDS measurement.<br>✍ The items 3.3.1 to 3.3.3 below are used in Detector Efficiency (the X-ray absorption efficiency of the detector) in spectrum calculation. In quantitative analysis, the same detector is used for both standard and unknown samples, so these values are canceled.<br>✍ The X-ray intake solid angle is obtained from items 3.3.5 to 3.3.9 below. This value is used as the K-ratio (the ratio of the X-ray intensity of a standard sample to that of an unknown sample) in quantitative analysis. |

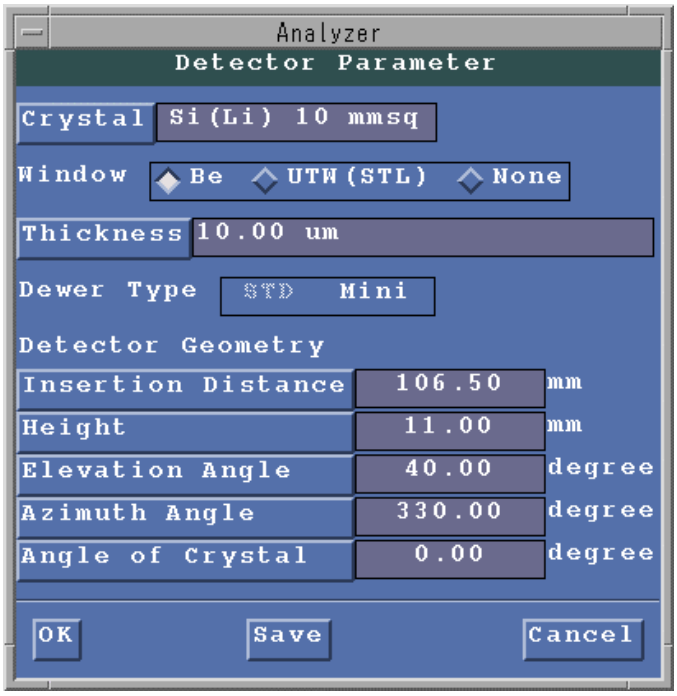


**Fig. 3 Detector Parameter window**

### 3.3.1 Crystal

The crystal is permanently set to Si (silicon).

☞ Refer to Fig. 4.

- Enter the thickness of the dead layer, the thickness of the detector, and the active area.

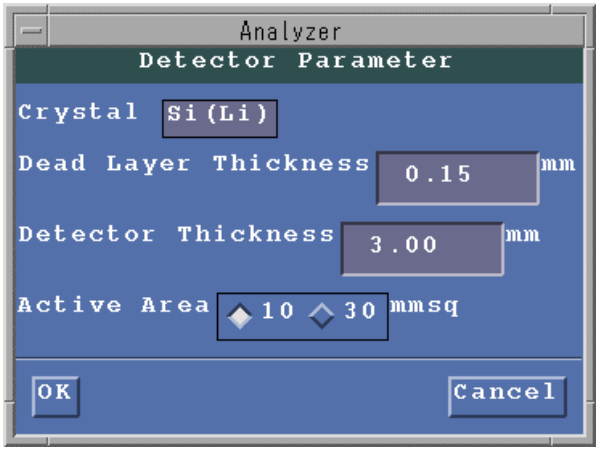


**Fig. 4 Crystal window**

### 3.3.2 Window

- Select the type of window from **Be**, **UTW** (Ultra Thin Window) and **None** (windowless).

  ✍ If you performed **EDS Reset**, be sure to confirm that this parameter is correctly set.

### 3.3.3 Thickness

- Enter the thickness of the window in the **Thickness** input box. If the window type is **UTW**, enter the film density and composition as well.

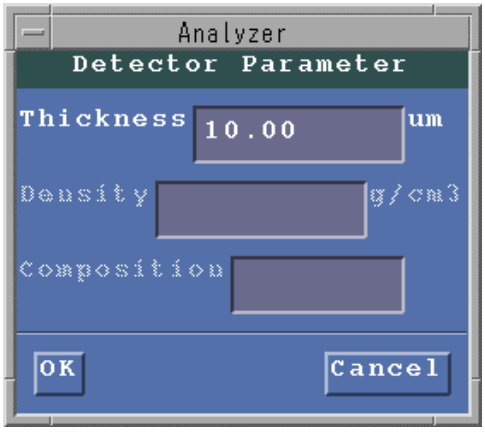


**Fig. 5 Thickness window**

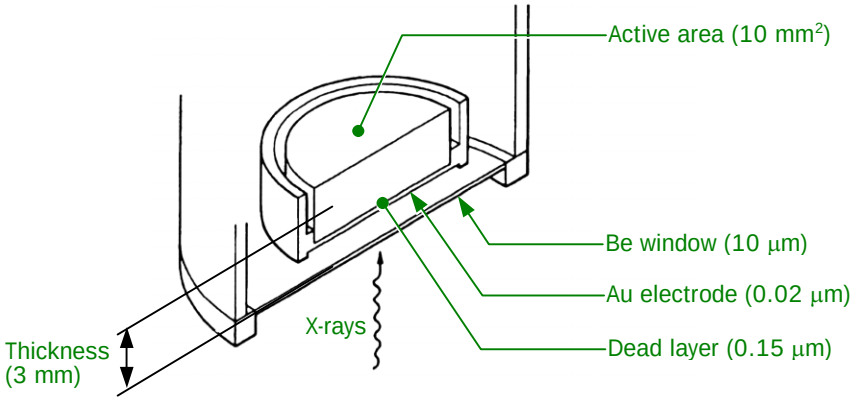


**Fig. 6 Specified values of detector parameters**

### 3.3.4 Dewar type

There are two types of Dewar vessels, a standard type (**STD**) and a mini-cup type (**Mini**).

The type of Dewar vessel that is installed in the EDS is read and displayed from the EDS subsystem when the EDS is started.

### 3.3.5 Detector Geometry

- Enter the parameters of the detector for **Insertion Distance**, **Height**, **Elevation Angle**, **Azimuth Angle** and **Angle of Crystal** in the Detector Parameter window.

  Refer to Fig. 7 and Fig. 8.

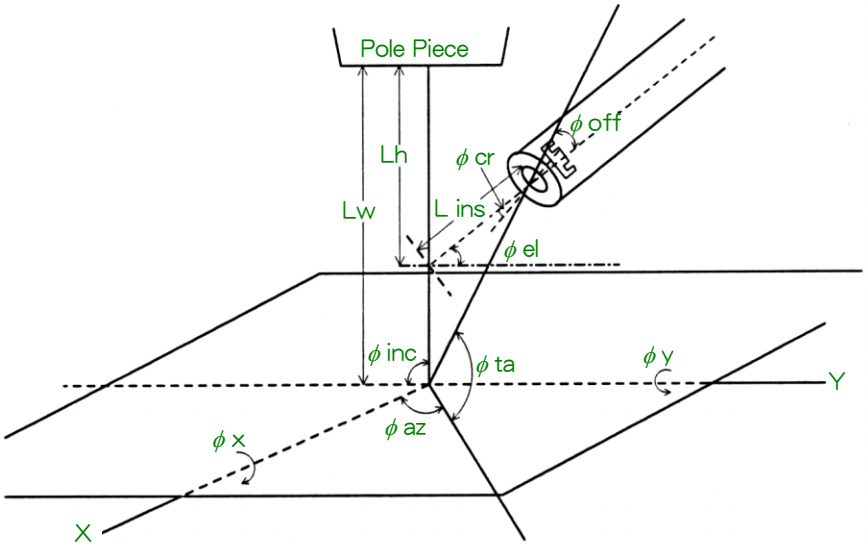


**Fig. 7 Detector geometry**

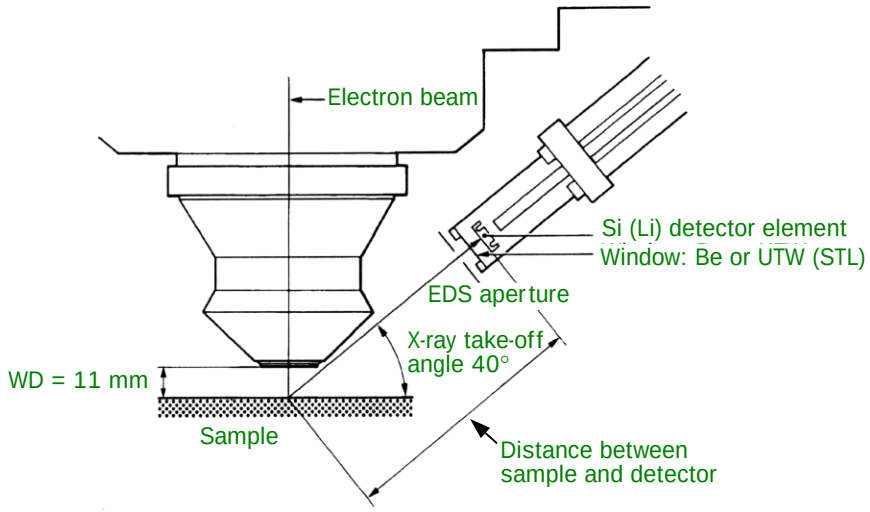


**Fig. 8 X-ray take-off angle and working distance**

### 3.3.6 Insertion Distance

- Enter the value of L ins as shown in Fig. 7.

  In the EX-14810, EX-14830 and EX-14850: 87
  In the EX-14820, EX-14840 and EX-14870: 97


**Fig. 9 Insertion Distance window**

### 3.3.7 Height

- Enter the value of Lh as shown in Fig. 7.

  The value of Lh is equivalent to WD of Fig. 8. Set it to 11 when using a JEOL detector.


**Fig. 10 Height window**

### 3.3.8 Elevation Angle

- Enter the value of ϕel as shown in Fig. 7.
  - This value is equivalent to the X-ray take-off angle of Fig. 8. Set it to 40° when using a JEOL detector.

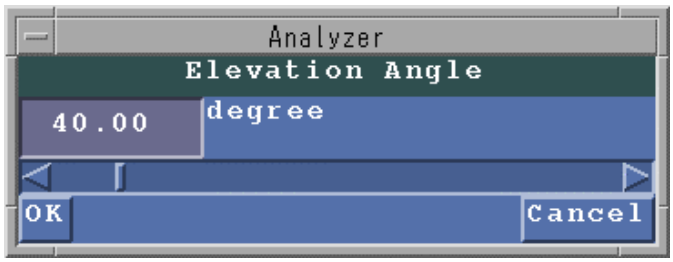


**Fig. 11 Elevation Angle window**

### 3.3.9 Azimuth Angle

- Specify the value of ϕaz as shown in Fig. 7 according to the port on which the spectrometer is installed as follows.
  - You cannot specify a negative value.

    For 1st spectrometer port: 287°
    For 2nd spectrometer port: 78.5°
    For 4th spectrometer port: 140°
    For EDS port: 330°

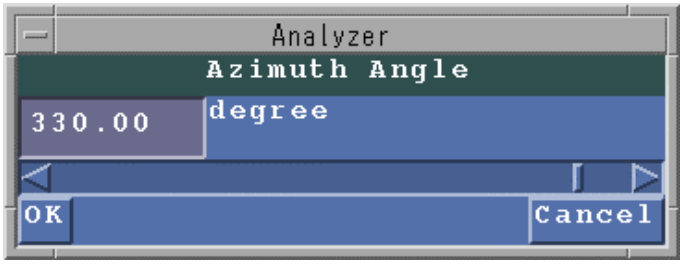


**Fig. 12 Azimuth Angle window**

### 3.3.10 Angle of Crystal

- Enter the value of ϕcr as shown in Fig. 7.
  - Set the value as 0° when using a JEOL detector.


**Fig. 13 Angle of Crystal window**

If the angle α of Fig. 14 is defined as the angle of incidence of the X-rays, the intake solid angle Ω can be expressed by the following equation.

$$\Omega = \frac{A \cos \alpha}{D^2}$$

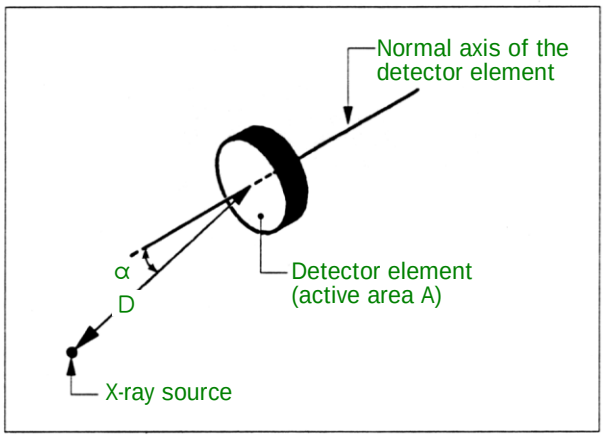


**Fig. 14 Intake solid angle**

## 3.4 Engineer Use

◆ Click on the **Engineer use** button in the EDS Analyzer Main window.

The Engineer use window opens as shown in Fig. 15.

Adjust the EDS subsystem using this window. Normally, however, there is no need to use this window.

✍ After you change any of the values in this window, be sure to open the Detector Parameter window, then click on the **Save** button to save the changes.

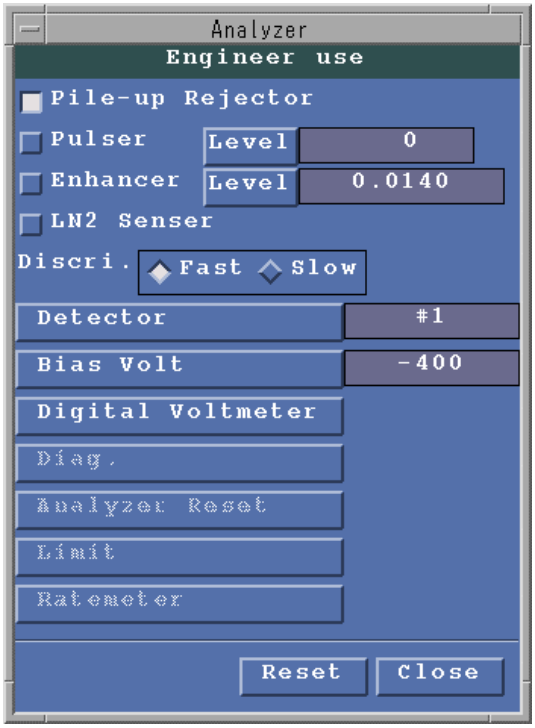


**Fig. 15 Engineer use window**

### 3.4.1 Pile-up Rejector

The Pile-up Rejector function allows you to correct the overlapping of the X-ray pulses during measurement. Usually, leave it on.

◆ Click on the **Pile-up Rejector** button to activate or deactivate this function.

✍ When you turn this function off, live-time correction does not take place. Particularly, when performing quantitative analysis, be sure to turn this function on.

### 3.4.2 Pulser

Pulser is a function that manually creates a waveform. Normally, leave it off.

Turn it on if you cannot collect data or if the energy resolution is poor, and you want to judge whether or not the cause lies in the detector.

◆ If you want to use the **Pulser** function, click on the **Pulser** button to turn it on; then click on the **Level** button.

The scroll bar will be displayed as shown in Fig. 16.. When you change the level, the position at which the pulse appears changes.

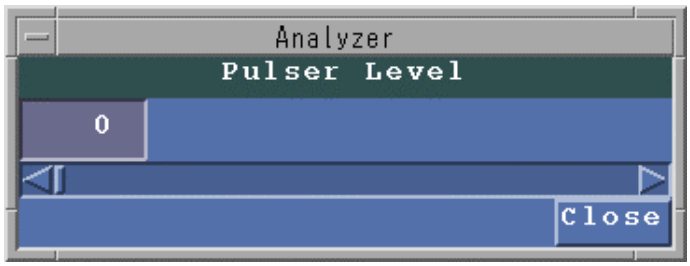


**Fig. 16 Pulser window**

### 3.4.3 Enhancer

The Enhancer function is used to improve the S/N ratio of an EDS X-ray image when you want to display the X-ray image on the Viewing Display. You can activate or deactivate the Enhancer function by clicking on the **Enhancer** button.

- If you want to use the **Enhancer** function, click on the **Enhancer** button to turn it on; then click on the **Level** button.

  The scroll bar will be displayed as shown in Fig. 17. You can adjust the degree of improvement of the S/N ratio by changing the value of **Time Constant** using the scroll bar.

If you make the value in the **Time Constant** box too large, the necessary signal components may sometimes be removed, so to optimize the image, set the value while observing the displayed X-ray image.

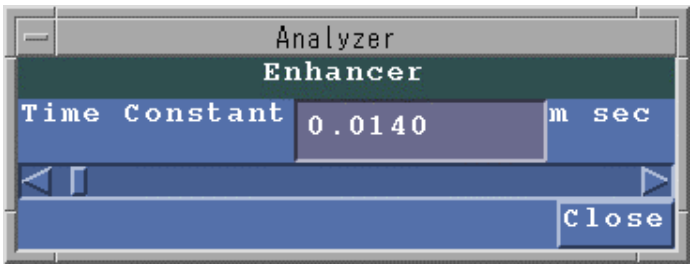


**Fig. 17 Enhancer window**

### 3.4.4 LN2 Sensor

This switch is used when a liquid-nitrogen sensor is installed, to set whether or not to use the sensor.

### 3.4.5 Discri.

These switches are used to set the display of the count rate to **Fast** or **Slow**. Usually, set it to **Fast**.

### 3.4.6 Detector

This switch is used when there are multiple EDS detectors, in order to select the detector whose signal is to be used. Usually, set it to #1.

Use this function to switch to the EDS detector on another EPMA. If you click on the **#2** button using the EPMA of a JXA-8200 series, you cannot acquire a spectrum.

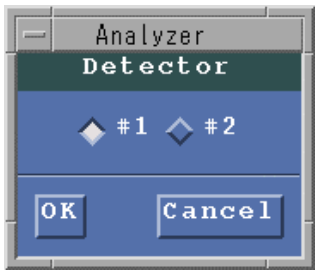


**Fig. 18 Detector window**

### 3.4.7 Bias Volt

- Click on the **Bias Volt** button in the Engineer use window.
  The Bias window opens as shown in Fig. 19.


**Fig. 19 Bias window**

By operating the scroll bar of the Bias window, you can change the bias voltage between –1000 V and 0 V in 100 V steps. The JEOL engineer sets it to the rated bias voltage indicated on the detector at delivery. Usually, do not use the Bias window. You can turn the bias on and off from the EDS Analyzer Main window.

When you set the bias voltage of the detector to a value other than the rated bias voltage, it is sometimes not possible to function according to the specifications, which can lead to a breakdown. For this reason, normally set the bias voltage to the rated value.

### 3.4.8 Digital Voltmeter

◆ Click on the **Digital Voltmeter** button in the Engineer use window.
The Digital Volt Meter window opens as shown in Fig. 20.

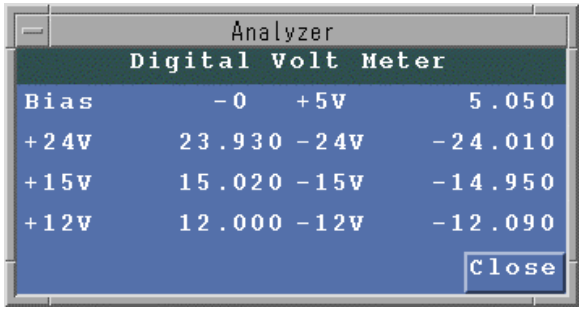


**Fig. 20 Digital Volt Meter window**

When you open the Digital Volt Meter window, the voltage of the DC power supply used in the EDS subsystem is displayed in real time. This function is used when performing maintenance on the subsystem.

# 4 EDS ENERGY CALIBRATION

Calibrate the EDS, and set the various conditions of the linear amplifier such as **Coarse Gain**, **Fine Gain**, **Zero**, **LL Discri.**, **Fast Discri.** and **Slow Discri.** The EDS measurement conditions are 20 keV full-scale and 2,000 channels.

## 4.1 Operation

1. Click on the **EDS** icon of the EPMA Main Menu.
   The EDS menu appears.
2. Select **EDS Calib** from the EDS menu.
   The EDS Calibration window opens as shown in Fig. 21.

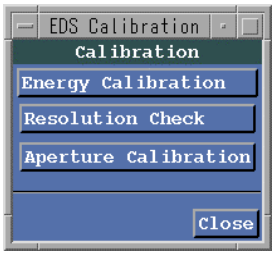


**Fig. 21 EDS Calibration window**

3. Click on the **Energy Calibration** button.
   The EDS Energy Calibration window opens as shown in Fig. 22.

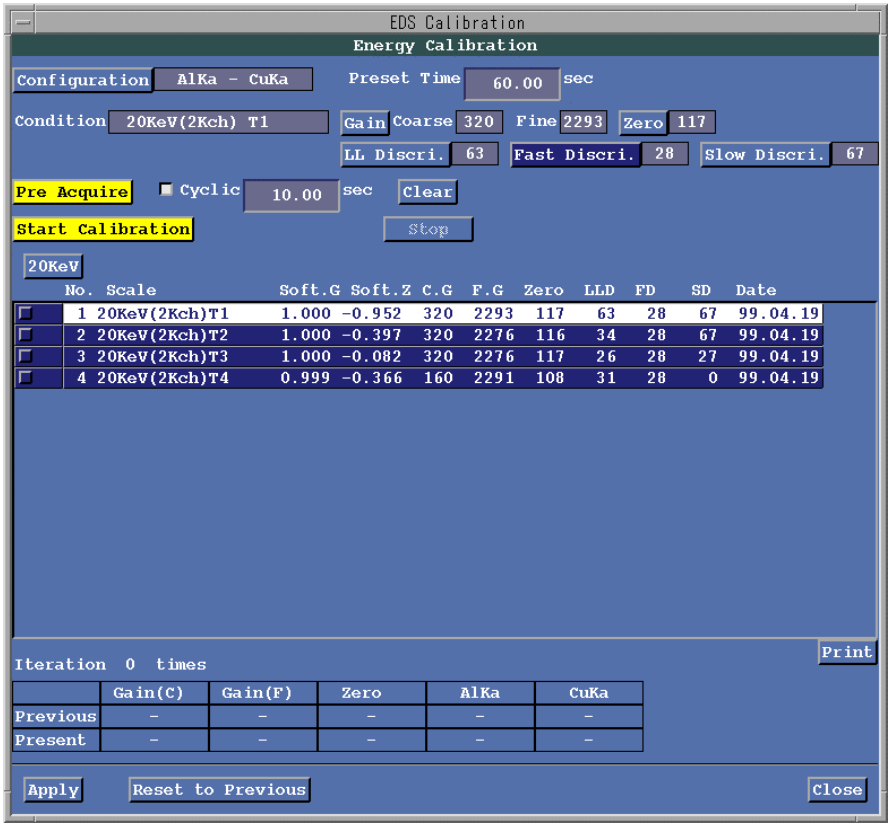


**Fig. 22 EDS Energy Calibration window**

### 4.1.1 Configuration

◆ Click on the **Configuration** button in the EDS Energy Calibration window.
The Configuration window opens.

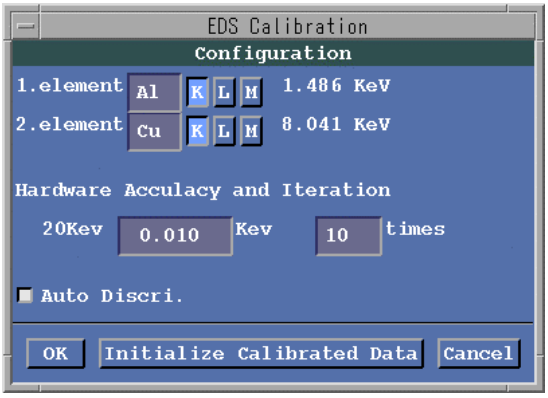


**Fig. 23 Configuration window**

Use this window to select the standard sample to be used for calibration or to set the convergence conditions.
When performing calibration at the very outset, it is necessary to set each item.
Also, by clicking on the **Initialize Calibrated Data** button, you can clear the previous calibration results.

## ■ Initialize Calibrated Data

◆ Click on the **Initialize Calibrated Data** button at the bottom of the window of Fig. 23.
The Initialize window of Fig. 24 appears. If you select **OK**, all previous calibration data will be erased, and the system will revert to its initial conditions.

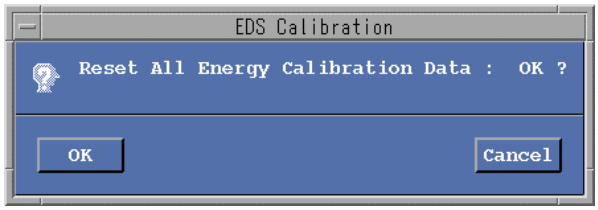


**Fig. 24 Initialize window**

Each calibration item combination is displayed at the center of the EDS Energy Calibration window with a number assigned to it. (From here on, the calibration item combination display is called the Energy Full-Scale/Count Mode button.) Items that have not yet been calibrated are displayed in the ordinary color, while items that have been calibrated are displayed in a dark color (☞ Fig. 22).
When you perform initialization, the following values are initially set.

- Coarse gain: 160
- Fine gain: 2700
- Zero: 128
- Soft gain: 1.0
- Soft zero: 0.0

### Element

Enter the element combination to be used for calibration.

- **Window–Be**: Cu K (8.041 keV), Al K (1.486 keV)
- **Window–UTW (STL)**: Cu K (8.041 keV), Cu L (0.936 keV)

### Accuracy

Key in the convergence energy to be used for calibration. Set a value of about 0.010 keV.

### Iteration

Enter the maximum number of tries for calibration. Normally, set it to about 10 tries.

### Auto Discri.

Select whether to automatically execute **LL Discri.**, **Fast Discri.**, or **Slow Discri.** after each calibration. Normally, select one of them.

## 4.1.2 Preset Time

Set the measurement time for performing calibration. A value of about 30 seconds is appropriate.

## 4.1.3 Condition

The presently selected counter mode item is displayed. Also, the button of the selected counter mode is highlighted in white.

## 4.1.4 Pre Acquire

If you attempt to perform calibration from the outset, the gain and other parameters may be incorrect. Also, the spectrum energy value may differ significantly from the correct value, and in some cases a spectrum may not be displayed at all. In such a case, use **Pre Acquire** to tentatively acquire a spectrum, and then adjust the gain and other parameters.

☞ For details, refer to Sect. 4.2, “Linear Amplifier Adjustment”.

## 4.1.5 Cyclic

This function is used during **Pre Acquire**. When you select **Cyclic** and set the time on the right-hand side, the spectrum will be automatically cleared after the specified time during acquisition, and re-acquisition will take place. As a result, the change in the spectrum when the gain and other parameters are being adjusted can be seen more clearly.

## 4.1.6 Clear

This button is used to clear an EDS spectrum during acquisition.

### 4.1.7 Start Calibration

This button is used to start calibration of the EDS. Calibration takes place in ascending numerical order of the clicked Energy Full-Scale/Count Mode buttons. (The red lamp on the left end of the button clicked is lit during calibration.)

### 4.1.8 Stop

This button is used to stop EDS spectrum acquisition. The lamp in this button is lit red during spectrum acquisition.

### 4.1.9 Energy Full-Scale/Count Mode buttons

A total of four calibration buttons are displayed in the EDS Energy Calibration window. The following eleven calibration item buttons are displayed.

| | |
|---|---|
| **No.**: | From **1** to **4** |
| **Scale**: | The full-scale energy is 20 kV. The count modes are **T1** to **T4**. |
| **Soft. G**: | Displays the soft gain |
| **Soft. Z**: | Displays the soft zero. |
| **C.G.**: | Displays the coarse gain. |
| **F.G.**: | Displays the fine gain. |
| **Zero**: | Displays zero. |
| **LLD**: | Displays the value of Lower Level Discriminator. |
| **FD**: | Displays the value of Fast Discriminator. |
| **SD**: | Displays the value of Slow Discriminator. |
| **Date**: | Displays the date on which calibration was performed. |

Calibration is performed to adjust the energy by using **Coarse Gain**, **Fine Gain** and **Zero** for the linear amplifier. If the calibrated values are inaccurate (but within tolerance of **Configuration**), calculate the deviations from the correct values as the soft gain and soft zero. These calculated values are used when accurate energy values are required for quantitative analysis, and related purposes.

### 4.1.10 Iteration, Previous, Present

These items are displayed during calibration.

| | |
|---|---|
| **Iteration**: | Indicates the number of times that a spectrum has been acquired. |
| **Previous**: | Indicates the values of the gain and other parameters during the previous acquisition. |
| **Present**: | Indicates the values of the gain and other parameters during the present acquisition. |

### 4.1.11 Print

Print the calibration results. The calibration history is printed on the leftmost part of the results.

*****: Shows that calibration has taken place.
**+**: Shows that initialization has taken place.
**–**: Shows that processing has not taken place.


### 4.1.12 Apply

Use to save calibration data.

### 4.1.13 Reset to Previous

Use to stop calibration and restore the previous values.

## 4.2 Linear Amplifier Adjustment

**Gain**, **Zero**, **LL Discri.**, **Fast Discri.** and **Slow Discri.** are used to adjust the linear amplifier of the EDS subsystem. When each of these is adjusted, the results are immediately indicated on the presently pressed Energy Full-Scale/Count Mode button. If you want to use the new values in the future as well, click on the **Apply** button to save the results. If you only intend to temporarily modify the energy, click on the **Close** button to cancel the results of adjustment. You can also cancel the results of adjustment by clicking on the **Reset to Previous** button.

Note that when you operate these buttons, the EDS waveform changes. Normally, after performing calibration, there is no need to change the settings. If a considerable period of time has elapsed since the previous calibration, however, we recommend that you calibrate again. In this case, the values will generally not differ greatly from the previous values, so there is almost no need to change them.

Sometimes when you operate the scroll bar of a window described below, the data being acquired changes significantly. In such a case, if you forget the initial values and have difficultly in restoring them, use the **Reset to Previous** button. The values that were displayed when the window was opened will return.

### 4.2.1 Gain

◆ Click on the **Gain** button in the EDS Energy Calibration window.

The Gain window opens.

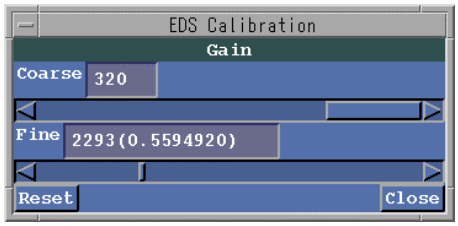


**Fig. 25 Gain window**

This button is used to set the gain of the linear amplifier. When you operate the scroll bar, the energy of the acquired waveform changes. The optimum value of **Gain** differs, depending upon the count mode.

To set gain approximately, manipulate the scroll bar in the **Coarse** input box of the Gain window.

To set gain finely, manipulate the scroll bar in the **Fine** input box of the Gain window.

You can also enter a value using the keyboard instead of using the scroll bars.

### 4.2.2 Zero

◆ Click on the **Zero** button in the EDS Energy Calibration window.
The Zero window opens.

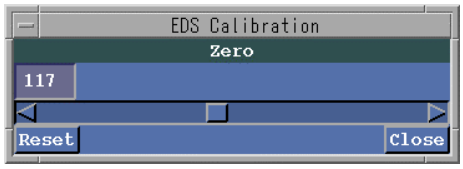


**Fig. 26 Zero window**

This window allows you to set the zero level of the linear amplifier. When you operate the scroll bar, the zero point of the data being acquired changes and moves. The optimum value of **Zero** changes according to the count mode.

### 4.2.3 LL Discri. (Lower Level Discriminator)

◆ Click on the **LL Discri.** button in the EDS Energy Calibration window.
The LL Discri. window opens.

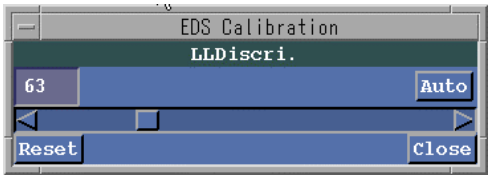


**Fig. 27 LL Discri. window**

This window is used to adjust the lower limit of the energy input to the AD converter. Adjust it so as to eliminate unwanted noise components at low energies. When you operate the scroll bar, the noise level changes. Use the following procedure to set it automatically.

1. Press the **PCD** button on the Control Panel of the EPMA so that the electron beam does not irradiate the sample (**PCD–IN**).
2. Click on the **Auto** button in the LL Discri. window.
   The optimum value is automatically calculated (this takes between several seconds and several tens of seconds), and the value of **LL Discri.** is set.
3. If an error message appears, check the settings and repeat the previous step.
4. Carry out automatic setting using the **Auto** button several times until roughly the same value is displayed.
   To acquire an accurate spectrum, it is necessary to execute **Auto** at least once using the energy full-scale value, the number of channels and the count mode that will be used for actual measurement.

✍ During actual spectrum measurement, if noise is not cut sufficiently with respect to a peak, particularly that of a light element, operate the scroll bar to obtain the optimum value manually, then save it by clicking on the **Apply** button in the EDS Energy Calibration window.

If the EDS detector is a super mini-cup EDS detector (EDMS1, EDMS2) or an equivalent detector for detecting light elements, turn off the illumination lamp of the optical microscope and also the illumination lamp of the spectrometer before adjusting the value of **LL Discri.** If you adjust the value of **LL Discri.** while the illumination lamps remain lit, the illumination may prevent you from setting the optimum value of **LL Discri.**

### 4.2.4 Fast Discri. (Fast Discriminator)

◆ Click on the **Fast Discri.** button in the EDS Energy Calibration window.
The Fast Discri. window opens.

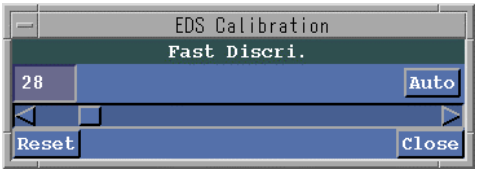


**Fig. 28 Fast Discri. window**

This window allows you to set the noise level that is to be detected on the fast signal of the linear amplifier to judge whether or not the X-ray beam is striking the sample. Use the following procedure to set it automatically.

1. Press the **PCD** button on the Control Panel of the EPMA so that the electron beam does not irradiate the sample (**PCD–IN**).
2. Click on the **Auto** button in the window.
   The optimum value is automatically calculated (this takes between several seconds and several tens of seconds), and the value of **Fast Discri.** is set.
3. If an error message appears, check the settings and repeat the previous step.
4. Carry out automatic setting using the **Auto** button several times until roughly the same value is obtained.
   To acquire an accurate spectrum, it is necessary to execute **Auto** at least once using the energy full-scale value, the number of channels and the count mode that will be used for actual measurement.

If the EDS detector is a super mini-cup EDS detector (EDMS1, EDMS2) or an equivalent detector for detecting light elements, turn off the illumination lamp of the optical microscope and also the illumination lamp of the spectrometer before adjusting the value of **Fast Discri.** If you adjust the value of **Fast Discri.** while the illumination lamps remain lit, the illumination may prevent you from setting the optimum value of **Fast Discri.**

### 4.2.5 Slow Discri. (Slow Discriminator)

◆ Click on the **Slow Discri.** button in the EDS Energy Calibration window.
The Slow Discri. window opens.

This window allows you to set the noise level that is to be detected on the slow signal of the linear amplifier, which is output during X-ray irradiation. It is useful when the subsystem is XM-Z6013T or a later model. It is not displayed for any other subsystem. This function is set for each count mode. To set it automatically, use the following procedure.

1. Press the PCD button on the Control Panel of the EPMA so that the electron beam does not irradiate the sample (**PCD**–**IN**).
2. Click on the **Auto** button in the window.
   The optimum value is automatically calculated (this takes several seconds), and the value of **Slow Discri.** is set.
3. If an error message appears, check the settings, and repeat the previous step.
4. Carry out automatic setting using the **Auto** button several times until roughly the same value is obtained.
   To acquire an accurate spectrum, it is necessary to execute **Auto** at least once using the count mode that will be used for actual measurement.

If the EDS detector is a super mini-cup EDS detector (EDMS1, EDMS2) or an equivalent detector for detecting light elements, turn off the illumination lamp of the optical microscope and also the illumination lamp of the spectrometer before adjusting the value of **Slow Discri.**. If you adjust the value of **Slow Discri.** while the illumination lamps remain lit, the illumination may prevent you from setting the optimum value of **Slow Discri.**

## 4.3 Measurement

### 4.3.1 Measurement preparations

1. Open the Configuration window (☞ Sect. 4.1.1), and confirm that **Element**, **Accuracy** and **Iteration** are set. The element combination changes according to the selected window.
   **Window–Be**: Cu K (8.041 keV), Al K (1.486 keV)
   **Window–UTW (STL)**: Cu K (8.041 keV), Cu L (0.936 keV)
   Set the convergence energy to about 0.010 keV. in the **Accuracy** input box.
   Set the number of tries for calibration to about 10 times in the **Iteration** input box.
   When you want to execute **LL Discri.**, **Fast Discri.**, or Slow **Discri.** after calibration, select **Auto Discri.**
2. Click on the **Preset** button in the EDS Home Window.
   The Preset window opens.
   ☞ For details, refer to the separate instruction manual of the EDS Operation Program.
3. Select **Mode–Live Time** and enter a value of about 30 seconds as Preset Time in the **Value** input box in the Preset window.
4. Click on the **Detector Parameter** button in the EDS Main Analyzer window.
   The Detector Parameter window opens.
5. Confirm that the kind of detector window (**Be** or **UTW (STL)**) is set correctly.
6. Click on the Aperture # button in the EDS Home Window.
   The Aperture window opens.
7. Select aperture No.**1**, No.**2** or No.**3**.
8. Click on the desired Energy Full-Scale/Count Mode button in the EDS Energy Calibration window to perform calibration.
   The Energy Full-Scale/Count Mode button will be highlighted in white, and the values of **Coarse Gain**, **Fine Gain**, **Zero**, **LL Discri.**, **Fast Discri.**, and **Slow Discri.** will be set in the EDS subsystem.
9. Prepare a standard sample according to the kind of detection window.
   **Window–Be**: Sample containing Cu and Al only (Cu/Al boundary of a sample consisting of a piece of aluminum foil placed on a Cu block, or the boundary part of a standard sample made of Al and Cu)
   **Window–UTW (STL)**: Cu standard sample

### 4.3.2 Preparatory measurement

1. Select **Monitor–EOS Monitor** from the EPMA Main Menu.
   The EOS monitor window opens.
2. Open **PCD**, apply the electron beam to the sample (**PCD–OUT**), and turn ON **Scan**.
3. Click on the **Pre Acquire** button in the EDS Energy Calibration window.
   Acquisition of an EDS spectrum starts.
4. Use the **CL** button in the EOS monitor window, or operate the Control Panel to adjust the current so that the dead time (**DT**) displayed on the EDS Home Window is between 20% and 30%.

5. When using Cu and Al, adjust the stage position so that the heights of the Cu Kα and Al Kα peaks are roughly equal.
6. Select an accelerating voltage of 20 kV or 25 kV so that the Cu Kα peak becomes higher than the Cu Lα peak.

   This calibration program detects the highest peak and also the next highest peak, and defines them in sequence from the low energy side as peak 1 and peak 2.
7. Adjust the energy position.

   Of the selected Energy Full-Scale/Count Mode buttons, click on the button with the lowest number so that it is highlighted in white; then the EDS subsystem will be set to the condition of the linear amplifier for the very first measurement.

When you are acquiring an EDS spectrum, white cursors appear at peak 1 and peak 2 to display the peak positions to be set. If the positions of the actual peaks differ greatly from the positions of the cursors, calibration may sometimes fail to take place. It is therefore necessary to roughly align the peak positions manually. To this end, operate the **Gain** button and the **Zero** button to adjust the peak positions. Also, in order to eliminate noise on the low-energy side, insert PCD to prevent the probe current from irradiating the sample, and execute **Auto** of **LL Discri.**

### 4.3.3 Performing calibration

◆ Confirm that the peak positions are roughly correct; click on the **Stop** button to stop **Pre Acquire** in the EDS Energy Calibration window; then click on the **Start Calibration** button to start calibration.

Calibration takes place in sequence from the lowest numbered Energy Full-Scale/Count Mode button.

- If you want to stop calibration, click on the **Stop** button.
  Items that have already been calibrated are valid and are stored in the memory.
- If you have performed calibration at least once already, data acquisition will start after the linear amplifier is set to the previous condition. Immediately after the system is initialized, the above initial values are used.
- After data has been acquired as far as **Preset Time** of calibration, evaluation of the peaks of the spectrum takes place. If a peak is not inside the convergence energy range, **Coarse Gain**, **Fine Gain** and **Zero** that are to be corrected are calculated, the linear amplifier is reset, then data acquisition starts once again. This operation is repeated until the peak falls inside the convergence energy range.
- During calibration, the number of iterations, and also the respective **Gain**, **Zero** and energy values of the previous and current data acquisitions are displayed.
- If **Auto Discri.** is selected, PCD is automatically inserted after the end of calibration. Then calibration takes place automatically in the sequence **LL Discri.**, **Fast Discri.**, and **Slow Discri.**
- If the calibrated results do not converge, the peak search error window appears as shown below.

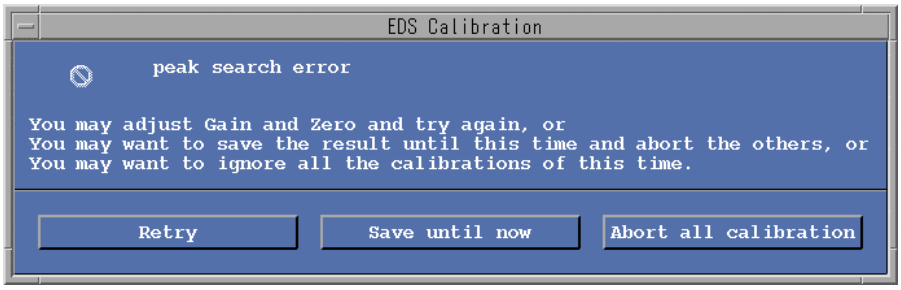


**Fig. 29 Peak search error window**

- Adjust **Gain**, **Zero** and **LL Discri.** so that the peak roughly aligns with the cursor. Adjust the beam current so that the dead time is between 20% and 30%. If the convergence energy range is too narrow, re-specify it to a slightly wider range. Subsequently, when you carry out **Retry**, calibration will resume.
- If you select **Save until now**, the results of calibration up to the present are stored in the memory and measurement is ended.
- If you select **Abort all calibration**, the results of calibration up to that point in time are all rendered invalid, and data acquisition stops.

If you attempt to perform calibration using combinations of all count modes, the dead time may become extremely long under certain conditions. In such a case, correction may not take place successfully, so reduce the probe current so that the dead time is between 20% and 30%.

### 4.3.4 Ending calibration

◆ After the end of calibration, click on the **Close** button.
The Close window opens as shown below.

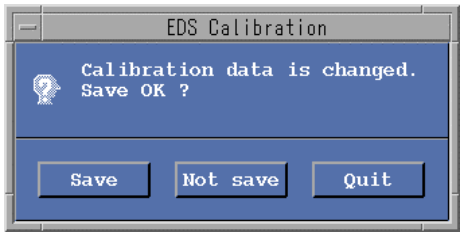


**Fig. 30 Close window**

- To save the calibration results, click on the **Save** button.
- To cancel the calibration results, click on the **Not save** button.
- If you select **Quit**, closing of the window is canceled.

### 4.3.5 Calibration file

Calibration files are stored in the directory**/opt/epma/cali/eds**. The filename of a calibration file is automatically assigned according to the kind of window in the detector and the effective area of the detector.
Shown below is a list of filenames of stored files.

**Table 1 Calibration Filename**

| Filename | Meaning |
|---|---|
| Be10.anl | The detector window is Be, and the effective area is 10 $mm^2$. |
| Be30.anl | The detector window is Be, and the effective area is 30 $mm^2$. |
| UTW10.anl | The detector window is UTW, and the effective area is 10 $mm^2$. |
| UTW30.anl | The detector window is UTW, and the effective area is 30 $mm^2$. |

# 5 EDS RESOLUTION MEASUREMENT

## How to open the Resolution Check window

1. Click on the **EDS** icon of the EPMA Main Menu.
   The EDS menu appears.
2. Select **EDS Calib** from the EDS menu.
   The Calibration window opens (☞ Fig. 21).
3. Click on the **Resolution Check** button of the Calibration window .
   The Resolution Check window opens as shown in Fig. 31.
   This window enables you to measure the resolution of the EDS.

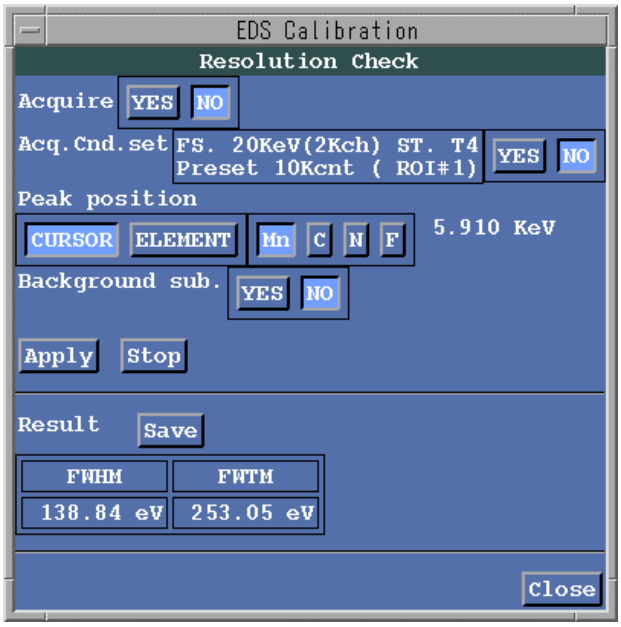


**Fig. 31 Resolution Check window**

## How to measure the resolution

1. Click on one of the **Acquire**–**YES** and **NO** buttons in the Resolution Check window.
   **YES** button: The resolution is measured after data acquisition.
   **NO** button: The resolution is measured for the spectrum of the acquired data (front data memory).
2. If you clicked on the **YES** button in step 1 above, click on the **Acq. Cnd. set**–**YES** or **NO** button in the Resolution Check window.
   **YES** button: Measurement takes place under the data acquisition conditions displayed in the **Acq. Cnd. set** condition display area of the Resolution Check window.
   **NO** button: Measurement takes place under the present set EDS measurement conditions.

   ✍ If you click on the **YES** button in step 2 above, the specified energy full scale (2 kch at 20 keV), count mode (T4) and preset (10 kcnt and ROI No.1 in the ROI accumulated value mode) will be set automatically.

✍ Set a ROI corresponding to one channel at the characteristic X-ray peak of the element concerned under the name ROI No. 1. Set this ROI by selecting **Spectra ROI** from the **Operation** menu in the EDS Home Window (☞ the separate manual "EDS Operation Program").

**3.** Click on either the **CURSOR** or **ELEMENT** button of **Peak position**.

**CURSOR** button: Designates the peak position as the cursor position.
**ELEMENT** button: Designates the peak position as an element.

When you click on one of these buttons, select an element by clicking on the **Mn**, **C**, **N** or **F** button.

**4.** Click on the **YES** or **NO** button of **Background sub.** and specify whether (click on the **YES** button) or not (click on the **NO** button) to subtract the background.

**5.** Click on the **Apply** button.

If you clicked on the **Acquire**–**YES** button in step 1, EDS measurement starts, and after the end of measurement the resolution is measured and the results displayed.

If you clicked on the **Acquire**–**NO** button, the resolution with respect to the spectrum in the front data memory is calculated and the results displayed.

✍ The displayed results are FWHM (Full Width at Half Maximum) and FWTM (Full Width at Tenth Maximum).

**6.** If you want to store the results, click on the **Save** button.

The results of EDS resolution measurement will be stored in a file.

✍ You can only store the results from when the peak position is **Mn**.

# 6 EDS APERTURE CALIBRATION

## How to open the Aperture Calibration window

1. Click on the **EDS** icon of the EPMA Main Menu.
   The EDS menu appears.
2. Select **EDS Calib** from the EDS menu.
   The Calibration window opens (☞ Fig. 21).
3. Click on the **Aperture Calibration** button of the Calibration window.
   The Aperture Calibration window opens as shown in Fig. 32.
   This window enables you to perform EDS aperture calibration.

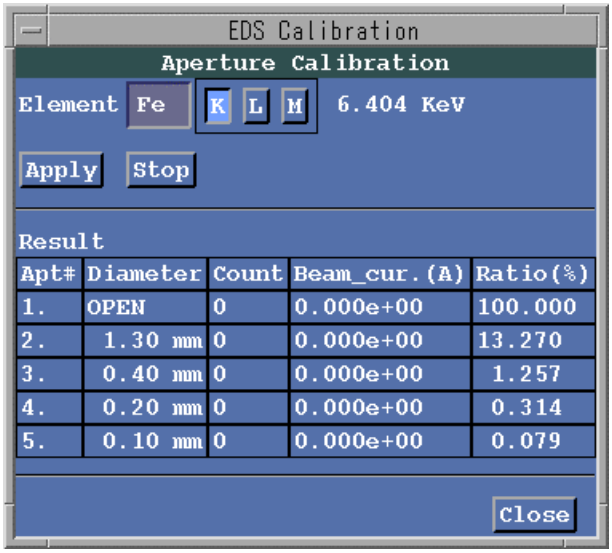


**Fig. 32 Aperture Calibration window**

## How to measure the X-ray intensity ratio for each EDS aperture

1. Click on the **Preset** button in the EDS Home Window.
   The Preset window opens.
2. Select **Mode–Live Time** and enter 100 seconds in the **Value** input box of the Preset window.
3. Key in the name of the element in the sample to be used for aperture calibration in the **Element** input box of the Aperture Calibration window.
   Normally, enter **Fe**.
4. Click on the **Apply** button.
   Measurement starts. The X-ray intensity at the presently specified aperture number is obtained, and then a message appears.
5. After the message appears, click on the **Aperture #** button in the EDS Home Window.
   The Aperture window will be displayed.
6. Select the aperture number set in the Aperture window, and click on the **OK** button.
7. Then set the next aperture number to obtain the X-ray intensity ratio for each EDS aperture, using EDS aperture #1 (fully open) as a reference.

✍ When using an EDS detector that has no EDS aperture control function (aperture selector function), change the EDS aperture manually, and set it as the next aperture.

**8.** Set the probe current so that the dead time is 15% to 35%.

✍ If you want to interrupt aperture calibration, click on the **Stop** button.
In this case, the calibration results up to this time point are stored in a file, and the processed calibration results are valid.

## ■ The displayed default value of the aperture ratio for aperture calibration is appropriate to detectors manufactured by JEOL.

If you wish to change the setting for another manufacturer's detector, carry out the following procedure.

**1.** Click on the **Console** icon on the monitor, enter the following to start the vi editor, then change the contents of the calibration file (Directory name: **/opts/epma/cali/eds**, Filename: **UTW10.anl**).

☞ Regarding the name of the calibration file, refer to Sect. 4.3.5.

Example: jxl>vi/opts/epma/cali/eds/UTW10.anl

**Table 2 Example of changing the setting**

| Current values (detector manufactured by JEOL) Apt_rat: | | Values for other manufacturer's detector (example)* |
|---|---|---|
| apt#1 | 1.000000 | 1.000000 |
| apt#2 | 0.132700 | 0.100000 |
| apt#3 | 0.015000 | 0.020000 |
| apt#4 | 0.004000 | 0.000000 |
| apt#5 | 0.000910 | 0.000000 |

* For another manufacturer's detector, enter the area ratio based on apt#1.

**2.** Operate the EDS Analyzer Main window to display the Detector Parameter window; then change the **Window** of the detector (for example, change it from **UTW** to **Be**), and return it once again.

The calibration file is read as a result of this.

**3.** Display the Aperture Calibration window, and confirm that the new default values are displayed.

✍ You can also change the default values of Diameter using the above procedure. The values of Diameter will be stored in the same calibration file.