

# XRF

## JEOL, JSX-3100RII

### Operating Procedure

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# 1. Overview

When an element is irradiated with X-rays, characteristic X-rays (fluorescent X-rays) with the energy inherent to that element are emitted. This apparatus detects the fluorescent X-rays obtained by irradiating the sample with X-rays emitted from the X-ray generator by energy discrimination by a semiconductor detector, Measure the type and concentration of the element.

For the quantification of elements by XRF, the FP (fundamental parameter) method, which is checked against an internal database, is commonly used.

## 2. Samples

### Measured elements

Elements with atomic numbers lower than Na cannot be measured by the detector of this instrument

Na, Mg ..... Can be measured by vacuuming during measurement

Al, Si, P, S, Cl, K, Ca, Sc, Ti ..... Vacuuming is recommended as you go to the left.

Elements with atomic numbers higher than Ti Can be measured under atmospheric conditions

- ✓ This instrument has a high background of Rh (rhodium) and Cl (chlorine). Therefore, to measure these elements, it is necessary to compare the background with a sample with the same reflectance of X-rays, or to quantify them by the calibration curve method.
- ✓ For N a, Mg, Al, and Si, the energy of X-rays fluorescence is low, and the amount of attenuation of X-rays before they reach the detector varies greatly depending on the conditions in the database. The accuracy is lower.

### Characters

If the bulk surface is clean and the diameter is 1.8cm or more, it is placed in the measuring window of the device as it is. If there is a slight stain, place the Prolene film between the device and the small Prolen film and place it in the sample cup for measurement.

Measured in small pieces, particulates, and liquid in a sample cup.

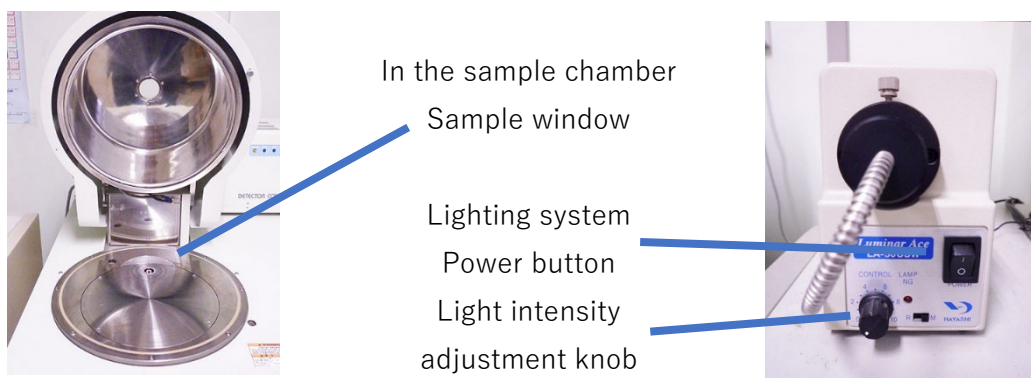
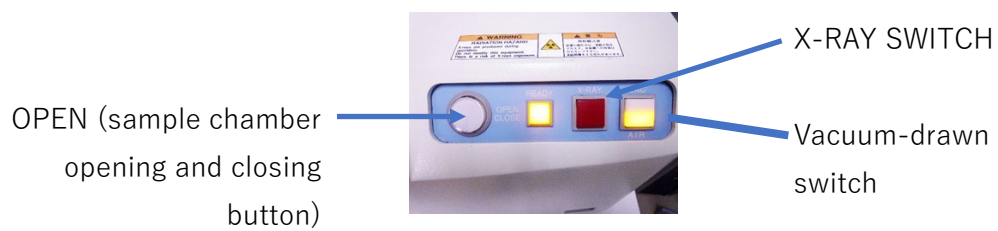
- △ Liquid samples cannot be vacuumed.

# Measurement size

Using a collimator, the diameter of the measuring range is selected.

Collimator size	7mm	3mm	1mm
Measuring range	14mm	6mm	2mm
Sensitivity	1	1/5	1/50

### 3. appearance



## 4. Equipment and application preparation

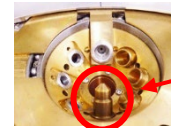
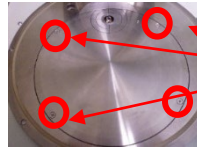
1. PRESS THE X-RAY BUTTON AND WATCH THE BUTTON LIGHT UP RED.
2. Turn on the lighting device.
3. Power on P C and start JaxStartar from desktop
4. Since the daily check item selection dialog appears, check the following items.
  - Look at the usage record book, select aging if there is no user who measured the day before, and do not select if there is no (if aging is not performed, the life of the equipment will be shortened).
  - Select Energy Check.
  - Don't select either ROSE check.
5. When the start button is pressed, a dialog is displayed prompting the setting of standard samples.
6. When the OPEN button on the main unit is pressed, the sample chamber opens, so set the standard sample for energy calibration in the measurement window, and press OPEN again to close the sample chamber.
7. Press the OK button in the dialog to start the daily check.
  - It takes about 30 minutes when aging is performed, and about 3 minutes when not performed, but during this time, the sample is prepared.
8. When completed, the message "Daily check was successful" will appear, so close all dialogs, take out the standard sample, and close the sample chamber. (If you do not change the collimator, you can leave it open.)
9. On the desktop of the PC, click "Analyzer manager" (displays X-ray tube voltage and current) Start the three "Stage eye" (display CCD image of the sample) and "Element Station" (application launcher), and when the Element Station starts, press the folder mark button in the upper left corner of the application to start the "Element analyzer" (measurement application).



## 5. Collimator settings

Check the collimator currently set in the device from the rotation display in front of the device body, and follow the procedure when replacing the collimator.

1. Right-click the radiation icon at the bottom right of the desktop and select Filter ☸  
– Open. (The collimator moves to the foreground.)
2. Open the sample chamber, Smaller Metal Disc 4 screws outside and then remove the disc.
3. Remove the collimator in the foreground like a screw and reinsert the collimator to be used.
4. Put the disc back and stop the screw.
5. Modify the rotation display of the collimator setting on the device body.

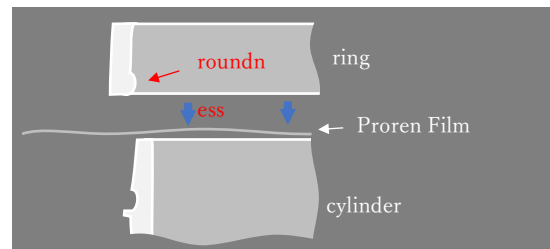


## 6. Sample preparation

### Creating a Sample Cup

If you want to use a sample cup, follow the steps below to create it.

1. Place the prolene film on the upper surface of the cylinder (without top and bottom).
2. Make sure that the ring has a rounded side and a pointed side with an inner corner, so that the side of the rounded corner is on the cylinder side. Fit the prolene film between the cylinders.



3. Place the sample in the sample cup. The amount of sample does not change its efficiency if the approximate diameter when placed is 15 mm or more. However, in the case of powder, it may spread to the entire area during the operation process, so it is desirable to cover the entire surface. In addition, the efficiency is low even with a small amount of sample, but there is no limit.

### Sample cup lid (for vacuuming powder)

When vacuuming is performed to measure powder samples, it is necessary to stretch a porous film on the top surface of the sample cup (otherwise the instrument will fail), so it is

necessary to use the prolene film in the preparation of the sample cup. In the same way as sandwiching, a porous film is inserted on the top surface after inserting the sample.

## 7. Set of samples

1. Open the sample chamber and place the sample at the position of the sample window.
2. While looking at the screen of the StageEye, turn the light intensity adjustment knob of the lighting device to the appropriate brightness up to the 12 o'clock position.
3. While looking at the StageEye screen, make sure that the point you want to measure is in the red circle.



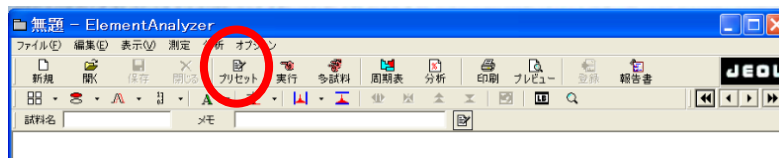
There are three red circles, corresponding to 2 mm, 6 mm, and 14 mm (collimators are 1 mm, 3 mm, and 7 mm).

4. When positioning is completed, the sample chamber is closed.
5. When vacuuming, turn on the vacuum switch. This switch flashes on the VAC side while the vacuum is being drawn, and lights up when it is completed. If the vacuum is not drawn, AIR is on.

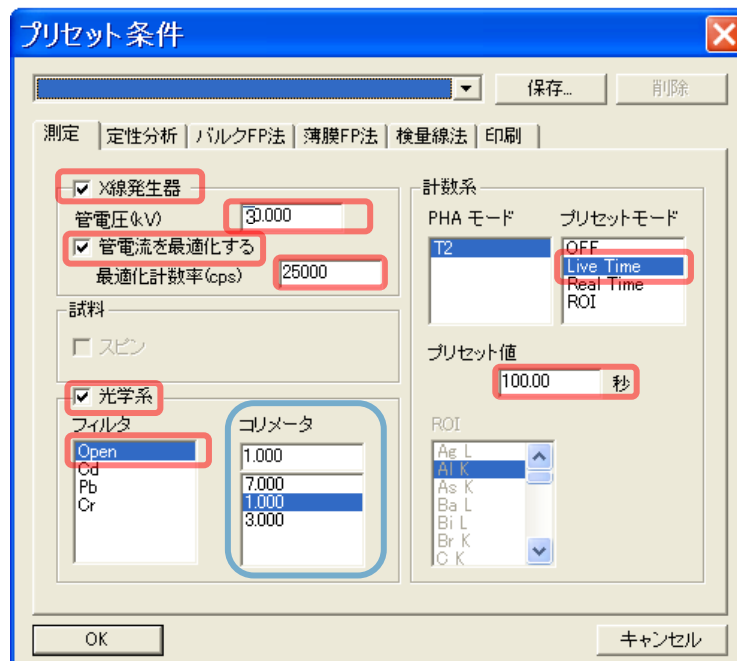


## 8. measurement

Press the "Preset" button of Element Analyzer to open the preset condition setting screen.



Check for matches in the red frame in the screenshot below.



Set the collimator item to the device settings and press OK.

Press the "Run" button on the Element Analyzer to start the measurement.



When the measurement is completed, the spectrum of X-ray energy, qualitative results of elements in periodic form, and quantitative results appear, respectively.

If you select [Analysis] – [Identify] on the menu bar and select the energy on the spectrum, the elements that emit characteristic X-rays at the corresponding position are listed, which can be useful for analysis if necessary.

When you click an element in the periodic table, you can check the characteristic X-ray

energy of the target element by displaying a line on the spectrum.

The quantitative result is redisplayed under the changed condition when the condition is changed.

If you think that the measurement time is insufficient, such as there is a lot of noise in the measurement result, click [Measurement] – [Integrating measurement] Select and add the measurement time.

Enter the measurement time to be added and click the [OK] button to continue the measurement.

When vacuuming the next sample is also vacuumed after vacuuming, press the O PEN button as it is, the sample chamber will open after returning to the atmosphere, and when it is closed, vacuuming will start automatically.

## 9. After using the device

When vacuuming measurement is performed, press the vacuum switch to AIR, open the sample chamber, and take out the sample.

Since measurement data cannot be saved all at once, activate each one before saving.

The collimator does not need to be returned.

PRESS THE X-RAY BUTTON AND WATCH THE BUTTON TURN OFF.

Set the light intensity adjustment knob of the lighting device to the lowest position, make sure that the ceiling of the lighting device is not hot, and then turn off the power switch of the lighting device. Do.

Exit "Stage eye", "Element analyzer", and "Element Station" respectively.

Copy data to U SB memory, etc.

Shut down Windows.

**Do not turn off the power switch on the main unit.**

Make an entry in the usage record book.