# User Manual

## WDX200 Compact Multichannel X-ray Fluorescence Spectrometer

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# Chapter One: General

WDX200 Compact Multichannel X-ray Fluorescence Spectrometer is a high precision analytical instrument, being able to analyze contents of several elements in a substance precisely, quantitatively and simultaneously. It is widely used in industrial analysis fields, also indispensable to quality, commodity inspection, environment protection, scientific research institutes and labs of colleges. This chapter gives an overview of its working principle, main specifications and system structure.

## Working Principle:

WDX Series X-ray Fluorescence Spectrometers works on the principle of wavelength dispersion technique, where CHARACTERISTIC X-rays of the interest element are separated and measured quantitatively according to its wavelength.

### Characteristic X-rays of elements

It is well known that matter is composed of atoms. And atom is composed of nucleus and extranuclear electrons. According to Classic Atomic Theory, i.e. Bohr Atomic Model, extranuclear electrons are distributed in electron shells having different energies (Figure 1-1). The innermost shell is K shell, which has the lowest energy and holds 2 electrons only. Other shells, from inner to outer, are L shell, M shell …. Their energy escalates along with them. Atom of different elements has different electron energy, but to atom of the same element, no matter what chemical forms it takes (compounds or simple), it has the same energy in the same shell.

When atom of certain element is excited by the primary X-ray with proper energy emitted from an X-ray tube, the extranuclear electron of this atom, usually an electron on the inner K shell or L shell, absorbs the energy and jumps out of the shell, which results in an electron vacancy hereafter. At this time, an electron on higher energy shells leaps and fills the vacancy automatically. This process is called electron leap. When this happens, the leaping electron sends out energy by giving off X-rays (X-ray photons). The energy of the X-ray photons equals to the energy difference between the two shells. As stated above, extranuclear energies of different elements are different. So X-ray photons released by different elements after excitation have different energies, i.e. each element emits X-rays with an energy

specially symbolizing this element. These X-rays represent the characteristics of this element, so it is called characteristic X-rays. For example, Al Kαline is characteristic X-rays of Al atom，where an electron of K shell is ejected and vacancy is filled by an electron of L shell. The energy of the X-rays is 1.487 keV (keV is kilo electron voltage, a unit for calculating the energy of microscopic particles), SiKα-1.74 keV, CaKα- 3.69 keV, FeKα-6.4 keV ……

According to modern theories in physics, microscopic particles such as X-ray photons have wave-particle duality， that is, they have properties of waves -– wavelength and properties of particles—energy. Wavelength and energy are convertible in the following formula.

$$E(keV) = \frac{12.3964}{\lambda(A^{\circ})} \quad \cdots\cdots\cdots\cdots\cdots\cdots (1)$$

As stated above, characteristic X-rays of each element have its own specific wavelength. As long as we detect X-rays of this wavelength, we can identify the existence of the element in a sample.

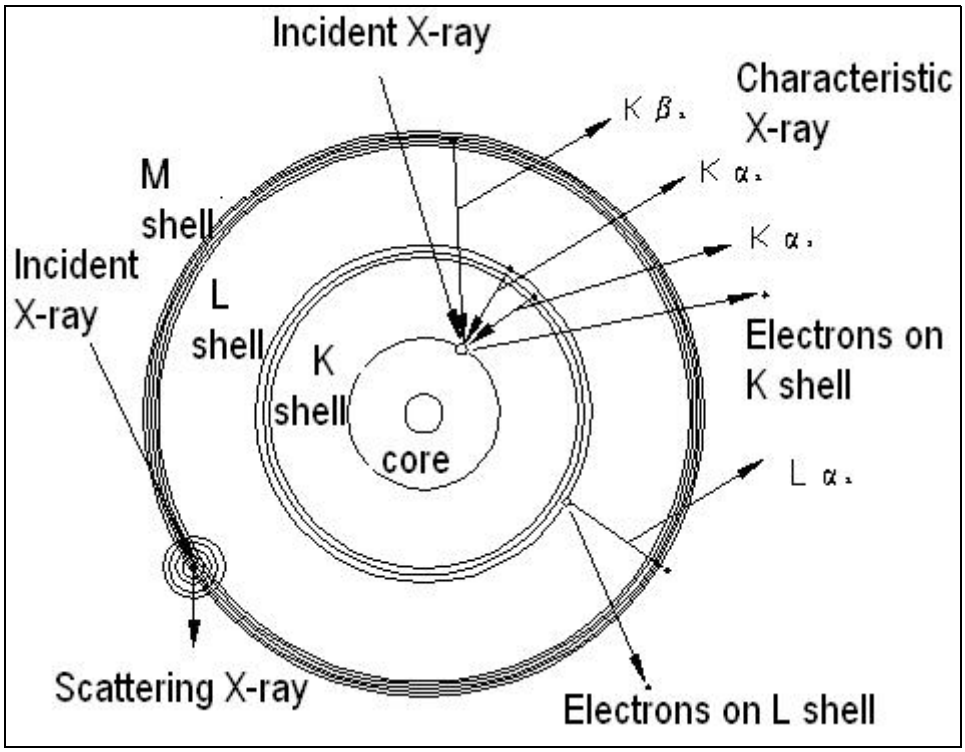



Figure 1-1: Bohr Atomic Model and Principle of Characteristic X-rays Generation

Principle of Wavelength Dispersive Spectroscopy

In condition that many elements co-exist in a sample and irradiated by primary X-ray from X-ray tube, each will emit its own characteristic X-rays, which in total are called X-ray fluorescence. The

technique to separate and measure the characteristic X-rays is called X-ray spectroscopy.

As each element produces X-rays of specific wavelength，X-ray separation can be done by using crystal diffraction technique on basis of Bragg Law. This X-ray separation method is called Wavelength Dispersive X-ray Fluorescence Spectroscopy.

Bragg's Law:

$$2d \sin\theta = n\lambda \quad \text{……（2）}$$

上 In the above equation, **d** is the distance between atomic layers in a crystal; **θ** is certain angle of incidence; **λ** is the wavelength of the incident X-ray; **n** is an integer of diffraction. In common situation, we measures only primary diffraction X-rays, which means n is 1.

Based on this law, if the analyzing crystal is determined (i.e. d-spacing of the crystal is determined), then only Characteristic X-rays with wavelength λ of the interest element can be measured at θ angle. And other elements, as their wavelength λ does not comply with the above formula, can not be measured. The analyzing crystal is either flat, where X-ray fluorescence is cast on the crystal after paralleled by the parallel collimator, named parallel spectroscopy, or curved, in which X-rays are separated by curved crystal with focusing ability, named focusing spectroscopy. Figure 1-2 shows the principle of the two systems.

Based on Wavelength Dispersive Principle, we make instruments on crystal diffraction technique. This kind of instrument is called Wavelength Dispersive X-ray Fluorescence Spectrometers, or abbreviated as WDS. As X-ray separation can be done on basis of the following two approaches, the X-ray Fluorescence WDS is also divided into two categories.

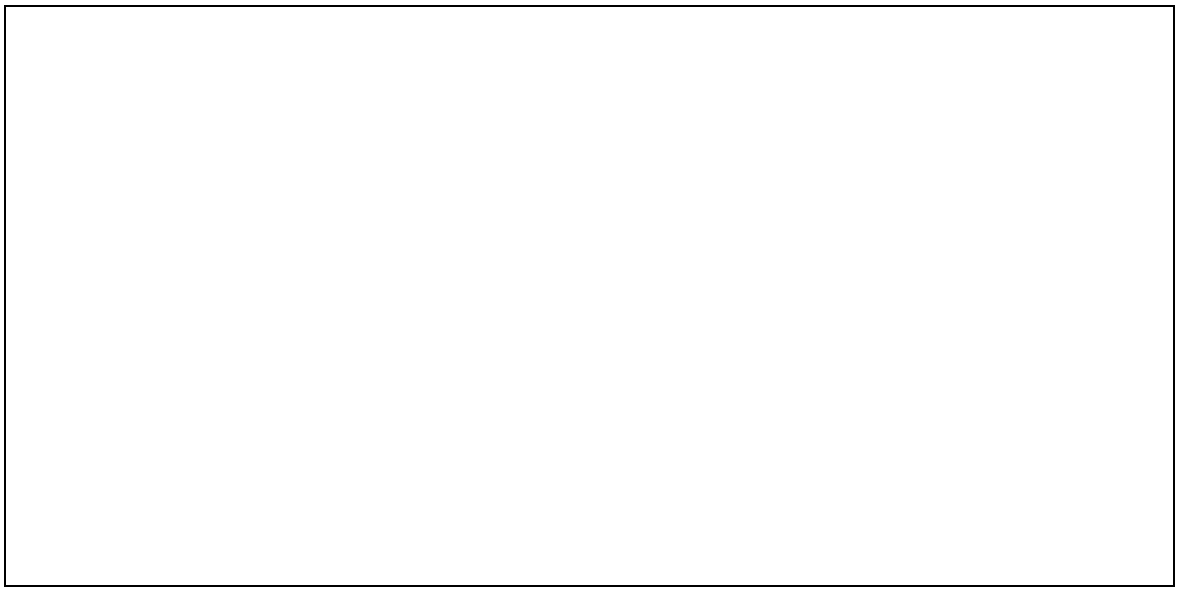


Figure 1-2 Schematic Diagram of X-ray Fluorescence Spectroscopy

### 1.3 Categories of X-ray Fluorescence Spectrometers

①单道扫描型 X 荧光光谱仪，简称扫描型 X 荧光光谱仪，也称作顺序测定型 X 荧光光谱仪（图 1-3）。

Single-channel Scanning X-ray Fluorescence Spectrometer is abbreviated as SCXRF, also called Sequential X-ray Fluorescence Spectrometer.

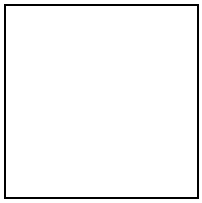


Figure 1-3 Schematic Diagram of Scanning X-ray Fluorescence Spectrometer

According to Bragg's law, we measure at θ angle only wavelength of Characteristic X-rays of a specific element. When the analyzing crystal rotates, i.e. comprising different θ angles, Characteristic X-rays of different elements can be measured. This is how X-rays are separated. As a single crystal can not separate Characteristic X-rays of many, crystal drum for auto crystals switch is adopted. During the measurement, the crystal changes automatically at θ angle and measures different CHARATERISTIC X-rays of different elements.

On the other hand, the measurement of this type may take a long time. This is because the instrument scans one element after another during the process. So the more elements we test, the longer measurement time it takes. In order to solve this problem, we adopted X-ray tube with power above 3000W. At present, X-ray tubes with power of 4,000W and 5,000W have appeared in commercial market. The structure of this kind of instrument is composed of the single channel (the main part) and fixed light elements channels (auxiliary). The reason behind this is the difficulty of measuring light elements Na, Mg, etc.

② Fixed Multi-channel (also Multi-channel Synchronic) X-ray Fluorescence Spectrometer is known as multi-channel XRF. WDX series belong to this type.

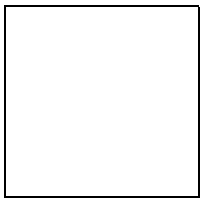


Figure 1-4 Principle of Multi-channel X-ray Fluorescence Spectrometer

Different from single-channel XRF, multi-channel XRF has an X-ray separation system composed of several fixed channels (X-ray separators). Each separator measures one element only. The more elements we n test, the more separators we need. Up to now, multi-channel XRF can hold approximately 30 separators at the most.

Due to simultaneous measurement, multi-channel XRF takes less time to analyze the sample at the same X-ray tube power when compared with single-channel XRF. If same accuracy is demanded, then multi-channel XRF consumes less power than that of the single-channel XRF. At present, X-ray tube for compact multi-channel spectrometer is 200w or 400w, and some 100w or 50w.

The structure of multi-channel XRF is fairly complex as fixed channels are many. The development tendency of these instruments is to take many fixed channels (light elements and main elements) as the main frame and add single-crystal heavy element scanning channel or semi-conductor detector as the supplementary, which is to enlarge its application scope and does not add cost of production in sharp amplitude.

### 1.4 Principle of X-ray Fluorescence Qualitative and Quantitative Analysis

As stated above, wavelength of characteristic X-ray corresponds with the elements. So to separate the characteristic x-ray with different wavelengths in the x-ray fluorescence of the sample relying on wavelength dispersive method is to identify the elements and realize the qualitative analysis of the elements.

On the other hand, the intensity of each element's characteristic X-rays is proportional to the element's mass percentage in the sample, that is, if the content of one element in a sample runs high, the characteristic x-ray photons/s emitted is high too and they satisfy the following equitation.

$$C_i=A_iI_i+B_i \quad \text{……（3）}$$

Among them, Ci is the content percentage of element i(or its compound), Ii is the intensity of the element's characteristic X-rays, constant number Ai or Bi is obtained by establishing working curve through standard sample measurement. Ai is the rate of curve of the working curve and Bi is intercept of the slope. The process to use standard sample to establish working curve is the calibration of the instrument. During the calibration, we measure a sample with known content. For an element i, if the content Ci is already know, the intensity Ii of the characteristic x-rays can be measured through X-ray separation. After measuring several sample, and through linear fitting, rate of curve $A_i$ and intercept $B_i$ of the working curve for each element can be calculated. After calibration, we work out the content of the element to be tested base on the equation 3 through measuring the intensity of each element's characteristic x-rays in the unknown sample.

$$C_i=A_{ij}I+B_{ij} \quad \text{……（4）}$$

$$A_{ij}=A_i（k'_{ij}C_j+1）+B_i(k''_{ij}C_j+1) \quad \text{……（5）}$$

Actually, intensity of characteristic x-rays of an element is influenced by other element's atomic absorption and enhancement. The inter-atomic absorption and enhancement is called matrix effect. Due to the existence of matrix effect, characteristic x-rays of an element is not only related to the content of the element but also influenced by other element, that is to say, Ai and Bi in equation 3 is not a constant number, 3 should be modified into 4.

$$Ci=AijI+Bij \quad \text{……（4）}$$

Where Aij=Ai（k′ijCj+1）+Bi(k″ ijCj+1) ……（5）

The matrix can have a considerable effect on the way the analysis is conducted and the quality of the results obtained. It can have an effect on the activity coefficients of the analysis; such effects are called matrix effects. The most common approach for accounting for matrix effects is to build a calibration curve using standard samples with known analysis concentration and which try to approximate the matrix of the sample as much as possible. This is especially important for solid samples where there is a strong matrix influence. In cases with complex or unknown matrices, the standard addition method can be used. In this technique, the response of the sample is measured and recorded, for example, using an electrode selective for the analysis. Then, a small volume of standard solution is added and the response is measured again. Ideally, the standard addition should increase the analysis

concentration by a factor of 1.5 to 3, and several additions should be averaged. The volume of standard solution should be small enough to disturb the matrix as little as possible

Equation 5 is a group of multi variables, among them $k'_{ij}$ and $k''_{ij}$ refer to the matrix effect factor element j has to the element i of interest. In order to find answer to 4 and 5, several generations of international x-ray fluorescence analysis has been working hard. There are roughly several typical ways as follows

**1.** Empirical coefficient: by measuring a group of standard samples (there is no rule in the inter-elemental content of the same group of the standard samples),and using non-linear minimum double fold approach, neuron network arithmetic mode and arithmetical way of many kinds of multi time overlapping, then build the working curve.

The measurement result of empirical coefficient method is accurate and reliable and this method is most widely used then. However, in order to improve the accuracy of calibration, we need many standard samples. And if the kinds of elements (compounds) are many, the standard samples should be many.

**2.** Fundamental parameters; according to the parameters and the matrix effect between the elemental atoms, and based on the theoretical calculation and measurement to the pure element (or pure compound), we calculate the influence factor of (5) and realize the quantitative analysis. Due to the instrument structural transformation and others, quantitative precision of this method is not good enough. So this method is usually applied for semi-quantitative analysis.

**3.** Theoretical **α**-coefficient： This method is between those two methods, and it is growing mature and applied in practice. In x-ray fluorescence spectrometers and other high-end X-ray fluorescence spectrometer, it has developed into quantitative analysis software for common use. The most significant advantage of adopting this quantitative analysis is to minimize the number of the standard samples for calibration. And usually 3 to 5 standard samples are used for correct multi elements quantitative analysis. This is to minimize the making of standard sample and work load and cost of analysis, this is especially true in fields that the sample making is difficult, e.g. bio sample analysis, it is impossible to adopt the empirical coefficient method requiring many standard samples. The theoretical α coefficient method has

a broad prospect.

**§1.2 Main technical specifications of WDX200 compact multi-channel X-ray spectrometer**

**1.** Measurable elements: 10 fixed channels, arbitrary 10 elements from Na to U can be measured.

**2.** X-ray tube: nominal power 400w, service power 200w, Rh anode (Pb anode)

stability of tube voltage and tube current (12 hours): less than 0.1%

**3. Quantitative analysis**

Analyzing method: empirical co-efficient approach, theoretical coefficient approach, linear regression approach

$SiO_2$、CaO ≤0.03%、 NaO、MgO other oxide ≤0.01%

Analysis accuracy (typical value): $\sigma_{n-1}$(12 hours stability, cement raw material, percentage content)

**4. Others:**

①Single sample measurement time: ≤5～7min

②Temperature of thermostat room: setting value±0.2℃

③Maximum vacuum degree of measurement room: ≤10Pa; vacuum pump: 2 L/min, ～220V

④ Oil pump: 16 L/min; oil temperature control: setting value±0.5℃

⑤Gas flow: self-researched gas flow control device. Voltage of secondary gas flow pressure regulating valve: 0.04～0.05MPa; Fluctuation scope of gas flow pressure: target value(approximate 110kPa) ±0.1kPa;flow rate 0～80 ml/min manually adjustable.

**5.** Industrial control microcomputer:

**6.** Master PC: Pentium 4; CRT: 17inch flat colorful monitor; printer: 80 columns ink-jet colorful printer.

**7.** Power supply: X-ray high voltage tube: 1000W~220V purified stabilized voltage power supply

Main frame: 1000W~220V purified stabilized voltage power supply

Vacuum pump: AC~220V, 1A

**8.** Dimension: 700(width) ×760(depth) ×800(height)

**9.**Weight: approximately 350kg

### §1.3 Structure of the instrument

Figure 1-5 is the diagram of systematic structure. The instrument is composed of tow parts: instrument and PC. The accessories include vacuum pump station, PR-10 gas bottle and 220v purified stabilized power supply.

The instrument is composed of the upper and lower parts. The upper part is thermostat chamber, which is equipped with auto-control heating zone, controlled by the electric circuit. The fluctuation of the temperature of the thermostat room is restricted in the scope of ± 0.2℃. Main parts like vacuum measurement room, X-ray separators of all elements and electric and mechanic parts requiring working in thermostat condition are installed in this chamber, which ensures the stability and repeatability of the measurement results. And as temperature of the thermostat room (30℃～38℃) is higher than that of the lab, it can effectively prevent the water vapor from condensing on the surface of the analyzing crystals so to protect them.

The lower part is electric room. X-ray high voltage power supply and its cooling system, industrial computer and auto-control electric circuit, spectrometer circuit and other electric devices are installed here. It is equipped with two 1000w AC purified stabilized power supplies, one for X-ray tube and the other for the control system and other electric appliances. The next chapters explain the structure, functions and technical requirements of the devices

X-ray Generation and Cooling System

Figure 2-1 is x-ray Generation and Cooling System, consisting of X-ray, high voltage power supply and cooling system. The cooling system includes X-ray cooling jacket, oil pump, heat sink, oil/gas separator, electric contact pressure gauge, and oil temperature sensor and so on.

X-ray tube is wrapped in the cooling jacket and fixed on the lower part of the central line of the vacuum measurement room. High voltage power +50kV、4mA（+50kV、6mA）is provided to X-ray tube by the power supply installed under the mainframe. Meanwhile, the supply provides heating current +2.5V、8A to the filament of X-ray tube through the cable. The high voltage output socket and filament connection terminal is fixed on the rear bracket of the high voltage power supply. In addition, ten-core airline socket on the rear plate connects with the controlling case through the cable. See the details on Chapter 6.

Tube voltage and tube current adjuster on the front plate of the high voltage power supply is designed for manual adjustment by the professionals. At this time, the inner/outer switch is placed in the inner control position. This switch is in outer control position when leaving factory. And it is automatically controlled by industrial control single chip on PC upper machine.

The work flow of X-ray cooling system: the oil flows out of the oil pump and passes through the oil gauge to enter X-ray cooling jacket. This cools the X-ray tube adequately. Then oil flows out of the cooling jacket and enter the heat radiator through the oil sensor, and it flows back to the oil pump through the oil-gas separator.

The technical requirements of the cooling system are as follows:

1. The high voltage transformer uses oil with enduring pressure ≥ 80kV/cm$^2$ and the oil path is exquisitely designed which not only strictly prevents high voltage sparks and arcs or surface discharge, but also cool X-ray tube adequately.

2. Point-to-point oil gauge indicating value＜0.03 MPa or ＞0.07 MPa, or temperature of

output oil of the cooling jacket surpasses 45℃, the chain control circuit will cut the power supply of X-ray high voltage power supply. This is to protect the X-ray tube. And it will also send off the alarm, requesting for inspection and expulsion of oil path failures.

3. There is an oil temperature adjusting fan fixed on the side of the heat sink. When the oil temperature sensor measures the oil temperature overpasses the set value of the starting temperature, the electric fan runs immediately to make the temperature of the oil drop. When the oil temperature drops to the set value of the stopping temperature, the fan stops running, which ensures the temperature to fluctuate in the set scope and prevent the cooling oil in X-ray tube from losing control.

4. Oil-gas separator (oil cup) can separate oil and gas rapidly and effectively and erase the bubble in the oil rapidly. At the same time, it functions as the cushion container in the oil path system, which ensures a reliable supply of the oil by oil pump, and also prevents the failures such as the light part damage caused by the heat expansion.

Chapter 3 Vacuum measurement room and vacuum system

In the wavelength dispersive X-ray separation system, the light path is comparably long. In order to avoid the absorption of X-ray radiation by the air in the light path, vacuum light path is commonly used. Figure 3-1 indicates the schematic diagram of the vacuum measurement and vacuum system.

**1. Vacuum Measurement room**

Vacuum measurement chamber is divided into upper and lower rooms. The upper room is sample room and the lower room X-ray separation room. Between the two rooms are middle plate and optical shutter. Both of them have exhaust nips, which connects with vacuum pump by vacuum duct. On the central part of the lower room is fixed with X-ray cooling jacket, and the round wall X-ray separation splitter if every element. The X-ray separation room will be vacuumed not only in working time but also after the machine stops. Regular vacuum pump must be done every 12 hours. This is to maintain the vacuum of the X-ray separation room and to protect the analyzing crystal from getting eroded by water vapor. (Many analyzing crystals tend to absorb water and resolve, which results in fungus on the surface.)

The sample room is used for putting samples. Inside the sample room and on top of the cover installed optical shutter driving mechanism, sample self-turning driving mechanism, protection cover driving mechanism and their respective driving motors.

**2. components of the vacuum system**

The vacuum system is composed of vacuum pump, switch valves(two), charge valve, chare globe valve, manual charge valve, four-way connector, three-way connector(two), vacuum gauge, and driving circuit for each valve, start-stop circuit for motor of vacuum pump. The vacuum gauge reads out the vacuum degree of the X-ray separation room to the industrial control computer. The vacuum pump is separated with the main frame, and other parts are all within the thermostat room.

3. **the function of every valve is as below:**

① Switching valve: there are two switching valve. One is installed in the vacuum path of the X-ray separation room, and called X-ray separation splitting room switching valve. When the pump station pumps vacuum for the X-ray separation room, this valve is opened. The other is installed in the vacuum path of the sample room. It is called the sample room switching valve.

When the pump station pumps vacuum for the sample room, the valve is opened.

② Charge valve: when pumping vacuum for the measurement room (including X-ray separation room and sample room), this valve is closed. When there is a need to charge gas into the sample room, this valve is opened.

③ Manual charge valve: this valve is installed on the four-way connector. When the instrument is maintained, there is a need to charge gas into the measurement room. At that time, open the valve with the screwdriver.

④ Chare globe valve: it is installed between the vacuum pump and the three-way connector. During vacuum-pumping, this valve is opened. And at the emergencies, e.g., sudden power cut or failure, this valve is closed. And during this time, air is charged into it, which prevents the vacuum pump from retuning oil.

**4. logic relation in the motion of every part when measuring the sample**

① Before sample measurement, the pump station vacuumizes the X-ray separation room; meanwhile, the optical shutter plate is closed, the switch valve of the X-ray separation room is open and the switch valve of the sample room is closed. Charge valve is opened; the sample room is in air charge state. The protection cover of the sample room is controlled by the controlling circuit for free opening and closing.

② After the sample is put into the sample room, close the protection cover. At this moment, the charge valve and the switch valve of the X-ray separation room are closed while the switch valve of the sample room is opened. The pump station vacuumizes the sample room only. The X-ray separation room is closed, but maintains a very high vacuum degree. When the vacuum degree of the sample room almost levels with that of the X-ray separation room, the switch valve of the X-ray separation room opens. At this moment, the X-ray separation room and sample room connect with each other. The pump station vacuumizes both rooms at the same time. When the vacuum degree reaches the set valve, the light shutter plate opens. The actions are completed and the measurement of the sample starts.

After the sample measurement is completed and sample change is demanded, first shut the

switch valve of the sample room, shut the light shutter plate, and open the charge valve to charge the sample room. At this time, the pump station vacuumizes the sample room only. The state returns to the sample changing state.

**5. Auto control**

The running of the vacuum pump station, the driving motor of the light shutter plate, sample room self turning driving motor, protection cover driving motor are all under control of the industrial computer through the control circuit for their sequential control and logic protection control. See Chapter 6 for the related instructions.

# X-ray separation system

The function of the X-ray separation system is to separate the characteristic x-ray of every measurable element in x-ray fluorescence and send them to their respective detector for measurement. The X-ray separation-splitting system is composed of X-ray separation splitters for different elements. WDX200 compact multi-channel x-ray fluorescence spectrometer can install 10 fixed elemental channel X-ray separation splitter at most, and has the ability to measure arbitrary 10 elements from F to U. The category and number of the measurable elements are specified by the users who order the products. If there is a special need by the users, e.g. they need a scanning channel light splitter or semiconductor detector channel; one fixed elemental channel must be reduced.

Two types of light splitter, i.e. flat crystal and curved crystal, are adopted in fixed elemental channel light splitter of WDX200.

① The composition of the flat crystal light splitter: figure 4-1A is its structural diagram. It is composed of the following parts: head of the light splitter and primary collimator: used for connecting the light splitter to the side wall of the X-ray separation room, whose inner barrel is used for installing the primary collimator. Primary collimator is a group of paralleled molybdenum plates. One of the functions is to

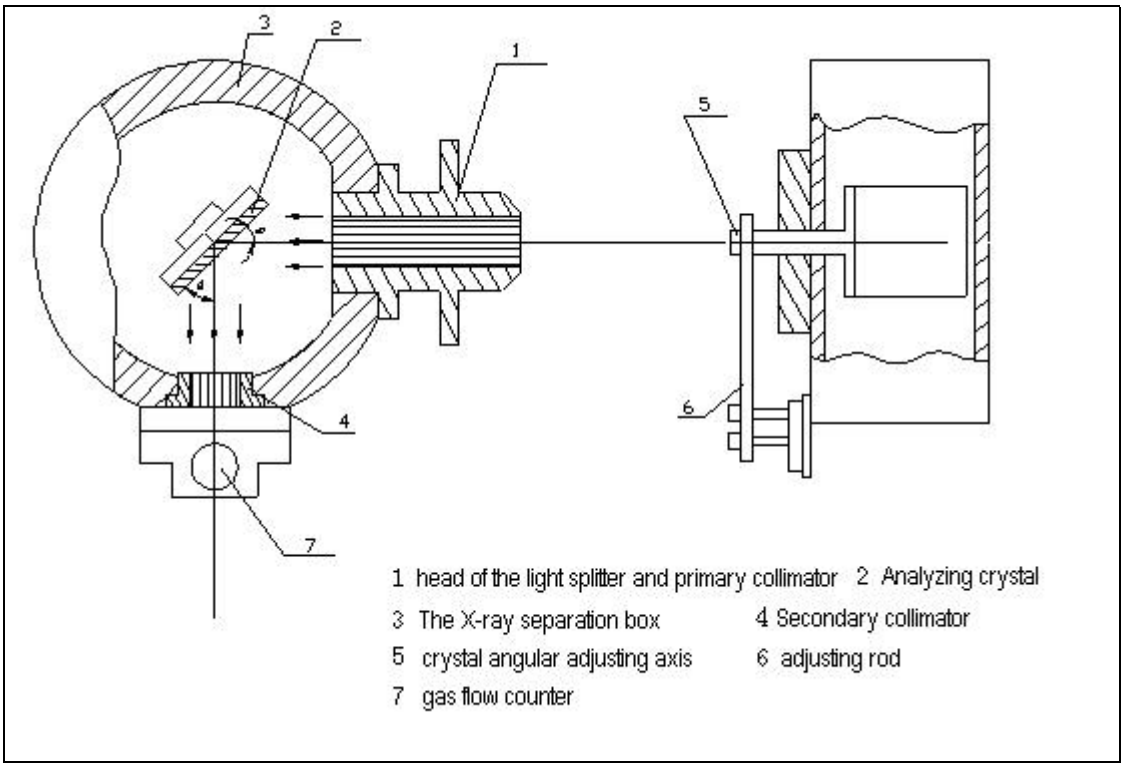


figure 4-1A: flat crystal light splitter structural diagram

② Analyzing crystal: to diffract the characteristic X-ray of the interest element in θ angle while other X-rays of other wavelength either be absorbed or scattered in every direction. Therefore, the category of the analyzing crystal is determined by the wavelength of the characteristic X-ray of the interest elements in accordance with Bragg Law.

③ The X-ray separation box: it contains the analyzing crystal, and the detector is installed on the emergency hole. And the inter-parts are strictly sealed by the sealing devices so as to prevent the vacuum leakage.

④ Secondary collimator: there is a group of paralleled Mo plates, which comes into angle θ with the analyzing crystal and makes the characteristic x-ray of the interest elements to enter the detector with high passage rate while minimizing the x-ray of other wavelength scattered out on the crystal surface. Secondary collimator is fixed on the window of the detector, which uses the polyester film to separate with the detector and the work gas in the gas flow count can be leaked into the vacuum system to influence the vacuum degree. The mo plates of the secondary collimator functions as the support to the membrane, which won't suffer the damage in the work air pressure.

⑤ 、⑥5 and 6 are crystal angular adjusting axis and adjusting rod, which are used for precise adjustment of the crystal angle. They maximize the characteristic x-ray which enters the detector.

2. Figure 4-1B is the composition of the curved crystal. Na path and Mg path in WDX200 must be equipped with this kind of light splitter. The difference between this splitter and the flat one is as below:

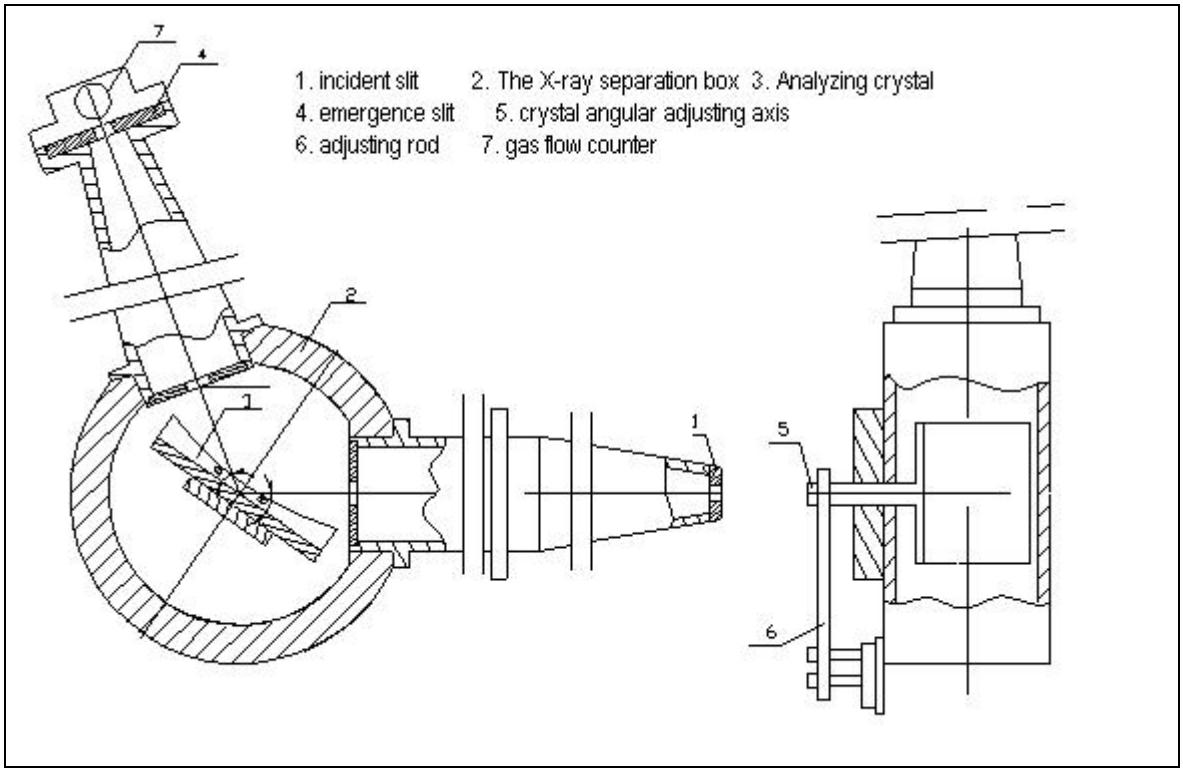



Figure 4-1B: the structure of the curved crystal

① The curved light splitter uses incident slit and emergence slit to replace the primary and secondary collimator in the flat light splitter.

② The surface of the analyzing crystal is curved. Crystal surface of Na path and Mg path in WDX200 is log spiral surface. X-ray

3. The configuration of the analyzing crystal: the analyzing crystal is the main part of the instrument. The configuration of the analyzing crystal is based on the requirements of the users who propose the elements to be tested. Table 1 is the detailed configuration of the instrument used in silicate industries like cement and glass.

Table One

| No. | Element channel | Category of the crystal |
|---|---|---|
| 1 | Na | TAP(001) log spiral curved surface curved crystal or high efficient multi-layer tactoid |
| 2 | Mg | AP(001) log spiral curved surface curved crystal or high efficient multi-layer tactoid |

| | | |
|---|---|---|
| 3 | Al | AP(001) log spiral curved surface curved crystal or PET flat crystal |
| 4 | Si | InSb(111)flat crystal |
| 5 | S | Ge(111)flat crystal |
| 6 | K | LiF(200) flat crystal |
| 7 | Ca | LiF(200) flat crystal |
| 8 | Fe | LiF(200) flat crystal |
| 9 | Cl | Ge(111) flat crystal |

# Chapter 5 X-ray detection and gas flow systems

## §5.1 Structure and working principle of x-ray detection system

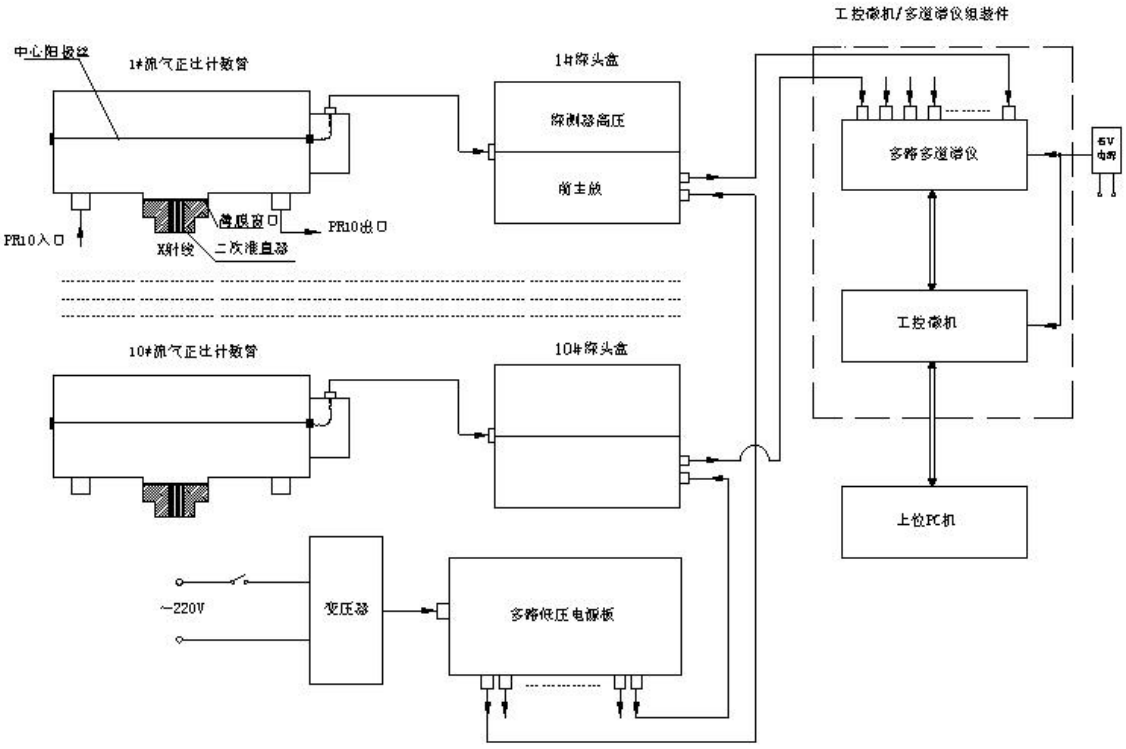


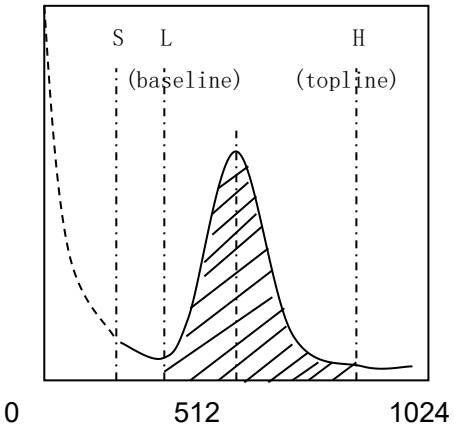

Figure 5-1 Schematic Diagram of X-ray Detection System

Figure 5-1 is the schematic diagram of X-ray detection system. At the emergence end of the spectroscopy of every element is installed with detector of characteristic x-rays. In this instrument the element channel adopts gas flow counter as the detector.

1. Pulse height digit to analog conversion

This is to save the pulse in the saving units with corresponding addresses according to their different heights. The address is called channel address, and the number of the saving units is called channel number, e.g. 512 channels, 1024 channels, the max height (5V for this instrument) corresponds with the highest channel address (1024 channel for this instrument). The industrial computer can read out the number of the pulses of each channel directly, which is displayed on CRT screen as the pulse

height spectrum lines.




图中的谱峰为被测元素的特征谱峰，谱线即为该元素特征 X 射线的脉冲高度分布曲线。L 线和 H 线之间的谱峰面积代表该特征 X 射线的强度，与该元素的含量近似成正比。L 和 H 分别称为基线和顶线。由操作人员在 PC 上位机上设定。

The spectrum peak here is the characteristic spectrum of the element to be measured, and the spectrum line is the pulse of the element's characteristic X-ray.

Height distribution curve: The spectrum area between L line and H line stands for the intensity of the characteristic X-ray. It is proportional to the content of the element. L and H are called base line and top line respectively. They are set by the operator on PC upper machine.

2. Noise elimination

Among the pulse signals output by the amplifier, the massive low amplitude pulse come from the electronic circuit itself, for example, the doted line on the left side of S line in figure above. Noise repression circuit is installed on the input electric path of the spectrometer, which eliminates the signals of this kind to enter the digit/analog conversion circuit, minimizing the influence on the conversion circuit.

One shining point of our wavelength dispersive X-ray fluorescence spectrometer lies in the integrated multi path multi channel spectrometer. It is combined with the industrial computer. This design militates in favor of not only instrument tuning and failure diagnosis but also correction of analysis deviation caused by spectrum peak drift as it measures characteristic X-ray of every element at every

time. This has improved the correctness and stability of the instrument.

The industrial computer/multi path multi channel spectrometer is installed in the control box located in the lower right part of the mainframe. The detector case of every element channel and multi path low voltage power supply plate, which provides ±12V、＋5V for detector case, are installed within the thermostat room. The latter is located in the left side wall of the room. It adjusts the voltage of the detectors through the small holes on the wall. This enables the center of the spectrum to be put in the proper position.

**§5.2 gas flow system**

As stated above, gas proportional counter starts to work only when PR-10 gas is supplied. Primary ion number and electronic multiplication number generated by X-ray photons are closely related with the gas in the counter.

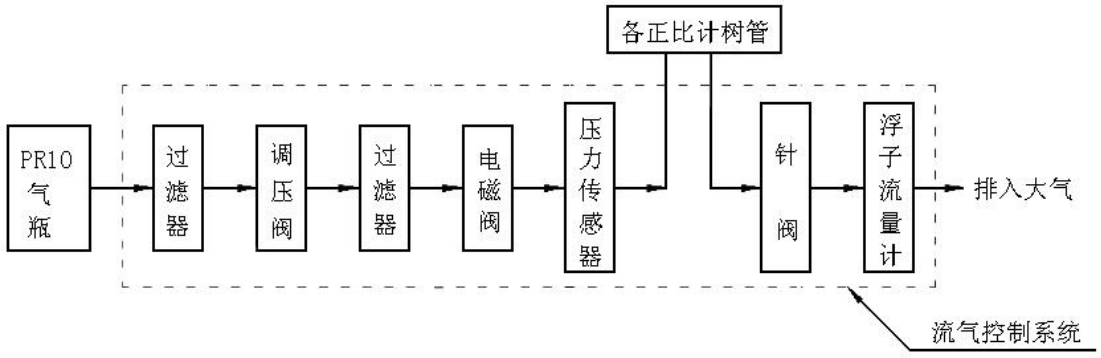



图 5-3

Figure 5-3 Gas flow path

**1. gas bottle and outlet pressure adjustment**

P PR-10 Near the outlet of Gas Bottle, there are installed with primary pressure gauge, pressure reduction valve and secondary pressure gauge. When we open the valve of the gas bottle, the pressure will be displayed on the gauge; turn the adjusting button of the pressure reduction valve clockwise slowly and regulate the gauge indication to 0.14～0.15 MPa. When PR-10 is fully filled, the value shown on the primary pressure gauge is 10MPa. With the consumption of the gas, the pressure of the bottle reduces. In

**2. Gas flow control system and pressure reduction valve setting**

The gas flow control system is composed of secondary pressure reduction valve, filter, electro-magnetic regulating valve, pressure sensor, need valve and flow meter. The adjusting handle of the secondary pressure reduction valve is configured with locking mechanism,

**3. Electro-magnetic regulation valve**

This valve keeps the pressure in the counter stable, and

**4. 压力传感器**

用以测定计数管进口压力，其压力信号接入连锁保护电路，一旦流气压力低于 103 kPa，保护电路立即切断探头盒低压电源板的～17V 供电，使所有流气正比计数管的工作电压降为零以保护计数管的中心阳极丝。

**5. 设定流量**

针阀用于调节气体流量，逆时针调节旋钮流量上升，反之下降。从浮子流量计上可看到流量大小。浮子流量计也可小范围调节流量，旋钮顺时针转动流量减小，反之增大。流量的出厂设置为 10～20ml/min，请用户不要随意改动。

请千万注意：开机前，**务必提前 4 小时打开流气**，流量设定在 15～30ml/ min（一旦设定后不再变动）。以便充分排除流气正比计数管内的空气，以防止空气使中心阳极丝氧化或烧坏。以后即在此流量下工作，不可再调。原则上短期停机时（3 天以内），不必关流气。3 天以上的长期停机或维修需要时，关闭气瓶上的阀门以关断流气。

# Chapter 6 Auto control circuit

Electric and auto control circuit includes two control systems, i.e. intelligent auto control system and electric power supply system. They are illustrated respectively as follows

## §6.1 function and composition of the auto control system

he function of the auto control system: 1. high automation of the operation of the instrument 2. Chain protection to the key parts 3. To monitor the working status and important parameters of the key parts and realized the auto diagnosis of failures and alarming.

The two grades management system is composed of the master PC microcomputer and industrial computer. Figure 6-1 is the schematic diagram of the auto control system. Except the master PC computer, other parts are all installed inside the control case on the below right side of the mainframe and it is powered by the +5V, ±12V switch supply.

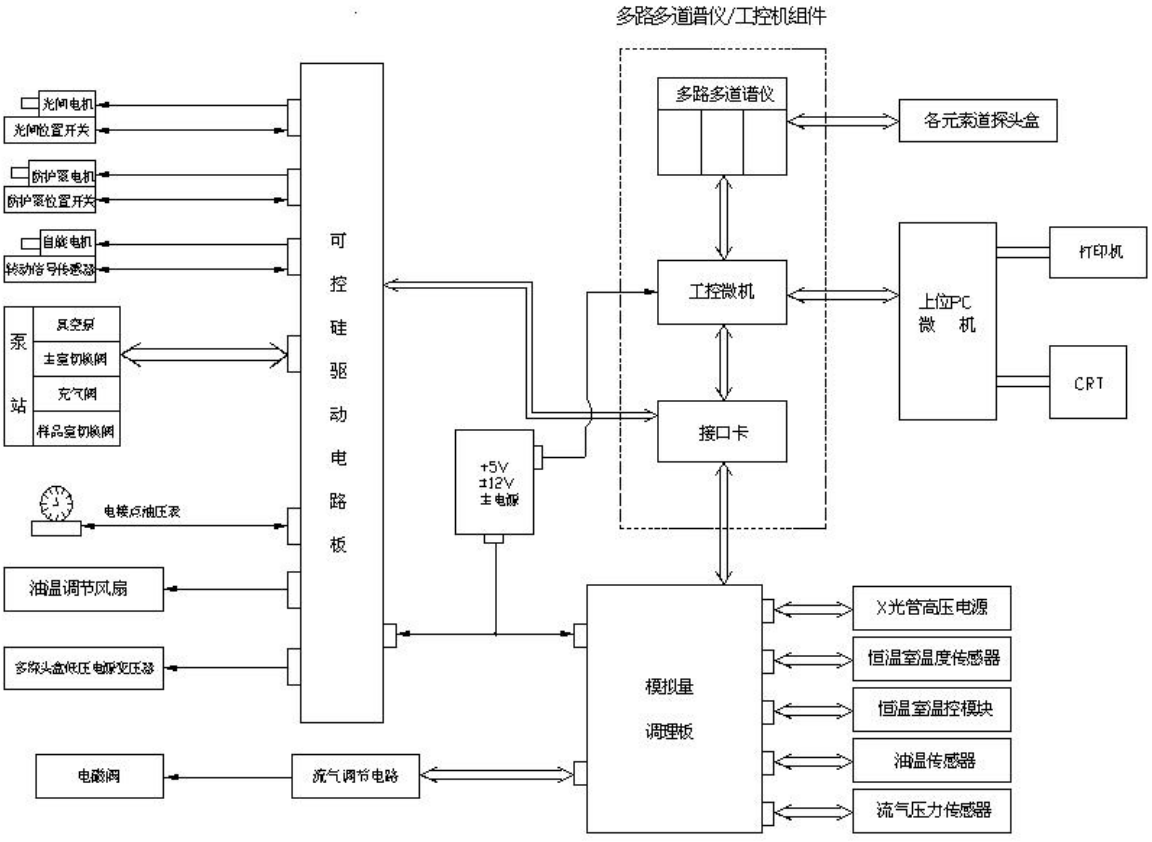



图 6-1 自动控制系统构成原理图

1. **master PC (including CRT and printer)**

to communicate with the industrial computer through RS-232 interface. The functions include:

①To release the operation order, and control the operation through the industrial computer

②to conduct the data management

③ quantitative calculation, printing and saving of the analysis results.

④ monitoring the main working parameters and the working status of the instruments, to give off failure alerts.

⑤to connect with the production control system and provide the analysis results

⑥to communicate remotely on the internet

**2. Industrial computer and interface board**

Figure 6-2 is the layout of the control computer case and its power supply diagram.

The industrial computer integrates with the multi path multi channel spectrometer board through

① The industrial computer collects the working parameter and work status information and provide to the master PC for monitoring.

② To receive the operation order from the master PC and send off work order to auto control each of the work parts.

③ To collect X-ray fluorescence data of the multi path multi channel spectrometer directly through the bus and provide them to the master PC for data treatment and quantitative calculation.

The composition and function of the interface plate is as follows:

① AD port: 16 path 12 digit ADC, AD port: 16 path 12 digit ADC, AD port:: 16 path 12 bit ADC, to collect analog signals like the tube voltage, tube current, oil temperature, constant room temperature, gas flow pressure and vacuum degree through the analog relay plate.

② DA port: 4 path 12 bit DAC, to provide set voltage value for X-ray tube high voltage power supply through analog relay plate; and to provide adjusting voltage for the solid relay of temperature control and pressure regulation and constant current module of gas flow pressure.

③ I/O port： 24 path. The controllable silicon driving board collects the information of the location of the switches and on-off signals of switches provided by electric contact oil pressure gauge

**3.** Analog quantity transmission board: to transmit the analog information which enters and leaves interface boards AD and DA and provide working voltage ±12V or +5V to relevant sensor.

**4.** controllable silicon drive plate

①112path～220V controllable silicon solid electric relay, which is used to drive all the working parts.

②Switching amount input electric circuit, which collects the switching amount signal of every position switch , electric contact oil gauge and provides +5V or ±12V working voltage to theses parts.

**§6.2 basic control loop**

The whole self-control system is composed of 9 auto control loops. Table 2 is the name, composition, workflow and chain relationship of every control loop

Table 2 Table of workflow and chain relationship of auto control loop

| No | name of the loop and its work parts | work flow | chain relationship and alarm |
|---|---|---|---|
| 1# | optical gate control loop<br>① controllable silicon chip 1#A 、 1#B solid relay<br>② optical driving motor<br>③ open position switch<br>④ close position switch | open: the industrial computer issues the “open” order→1#A controllable silicon becomes live→ drive motor for optical gate turns clockwise to open the gate→ the optical gate comes to the “start” position→ open position switch moves and sends out the signal “at place” → the industrial computer receives the signal and sends out the “stop ” signal→1#A controllable silicon cuts off→ the drive motor strops | The open conditions:<br>1. protection cover is closed<br>2. the switch valves of the pump station and x-ray diffraction room open simultaneously, the gas charging valve is closed, and the sample room and x-ray diffraction room start to pump at the |

| | | | |
|---|---|---|---|
| | | close: the industrial computer sends out the “close” signal→1#B controllable silicon gets live→ drive motor for optical gate turns anti-clockwise to shut the gate →the industrial computer sends out the stop signal→1#B controllable silicon is cut off→ the drive motor stops | same time |
| 2# | protection cover switching loop<br>① controllable silicon chip 2#A 、 2#B solid relay<br>② protection cover driving motor<br>③ open position switch<br>④ close position switch | open: the industrial computer sends out the “open” order →2#A controllable silicon becomes live→ drive motor turns clockwise to open the protection cover→ protection cover comes to the open position, the open position switch moves and sends out the signal “at place” →the industrial computer receives the message and sends out the signal “stop” →2#A controllable silicon is cut off→ the drive motor stops | open conditions:<br>1. the optical gate is closed<br>2. the switch valve of the sample room on vacuum pump is closed, the gas charging valve opens and the sample room gets charged |

| | | | |
|---|---|---|---|
| | | close: the industrial computer sends out the “close” order →2#B controllable silicon becomes live→ the drive motor turn anticlockwise to shut down the protection cover → when it reaches the close position, the close position switch works and sends out “at place” message → the industrial computer sends out “stop” order→2#B controllable silicon is closed→ the motor stops | |
| 3# | X-ray tube high voltage power supply control loop<br>① interface plate AD、DA port<br>② electric contact oil pressure gauge<br>③ oil temperature sensor<br>④ alarming light | Open: 220V AC stabilized power supply for X-ray tube switches on, and the alarming light is on→ the industrial computer provides baseline voltage value to the high voltage supply through DA port, and it accelerates till tube voltage and tube current reach the set value→ to monitor the tube voltage and tube current though AD port. | the open conditions for X-ray high voltage power supply<br>1. oil pressure value of electric contact oil pressure gauge is between 0.02 and 0.06 MPa<br>2. the oil temperature detected by the sensor is ＜ 45℃<br>3. Either the |

| | | | |
|---|---|---|---|
| | | Close: the industrial computer receive the order from the upper master PC → the interface board DA control the High Voltage Unit to low down the voltage and current gradually until to zero | optical gate or the protection cover is closed. |
| 4# | oil temperature adjusting loop<br>① 4# controllable silicon<br>② oil temperature sensor<br>③ oil temperature adjusting fan | the industrial computer receives the message that the oil temperature reaches the higher limit→ the industrial computer sends out the order to start the adjusting fan→ 4# controllable silicon becomes live→ the adjusting fan starts→ when message shows the oil temperature is lower than the lower limit→ the industrial computer sends out the message to stop the fan→4# controllable silicon is cut off→ oil temperature adjusting fan stops | If the oil temperature exceeds the allowance value （45℃） , the tube voltage and tube current will come down to zero and send off alarm. |
| 5# | detector case low voltage power supply open/close loop<br>① 5# controllable silicon<br>② Detector case low voltage power supply transformer | open: the industrial computer sends out the order: to open the detector case→5# controllable silicon becomes live→ low voltage power supply transformer input end is live<br>close: the industrial computer sends out the message: to close | When pressure value of Gas flow pressure sensor is ≤103 kPa , the low voltage power supply is cut off and sends off an alarming signal |

| | | | |
|---|---|---|---|
| | ③ gas flow pressure sensor | the detector case→5# controllable silicon is cut off→ to cut off the low voltage power supply transformer input end | |
| 6# | gas flow pressure adjusting loop<br>① interface plate DA port, AD port<br>② electro-magnetic adjusting valve<br>③ gas flow pressure sensor<br>④ valve adjustment circuit | AD port detect the difference between the gas flow pressure and set value → when the difference is greater than the set range, it will keep the pressure of the gas flow fluctuate around the set target value by adjusting the electro-magnetic valve through DA port | |

| | | | |
|---|---|---|---|
| 7# | pump station control circuit<br>① 6#A ～ 6#D controllable silicon<br>② vacuum pump power supply relay<br>③ switch valve drive circuit<br>④ gas charging valve<br>⑤ vacuum gauge | ①vacuum pumping of X-ray diffraction or light-splitting room: the industrial computer issues the message: to open the pump →6#A controllable silicon is live→ the vacuum pump power supply relay is tightly closed → vacuum pump starts → after a delay of 0.5～1s the industrial computer issues the order: to open the switch valve of the X-ray separation room →6#B controllable silicon is live → switch valve for X-ray separation room is opened and the X-ray separation room is get pumped | Prerequisits：<br>1. the optical gate plate is closed to full extent<br>2. the switch valve and gas charging valve are closed |
| | | ②Vacuum pumping of the sample room: the industrial computer issues the order: to shut the switch valve of X-ray separation room →6#Bcontrollable silicon is cut off. The valve is closed and the order “to open the sample room” is issued →6#C controllable silicon is live → switch valve of the sample room is opened, and the sample room gets pumped; when the vacuum degree approximates to that of the X-ray separation room, the switch valve of the X-ray separation room is opened. And the X-ray separation room and sample room are pumped at the same time | Prerequisites:<br>1. the X-ray separation room is being pumped<br>2. the optical gate plate is opened to its full extent<br>3. the protection cover is closed to its full extent |

| | | | |
|---|---|---|---|
| | | Gas charging of the sample room: the industrial computer sends off the order: to charge gas→6#C silicon control is cut off and switch valve of the sample room is closed→6#D silicon control is live. The gas charging valve is opened and the sample room gets charged→ several minutes later, 6#D silicon control is cut off → the gas charging valve is closed. | Prerequisites:<br>1. the optical gate plat is closed to its full extent.<br>2. the switch valve is opened. |
| | | to stop the pump: the industrial pump issues the order: to stop the pump→ the optical gate closes fully →6#C silicon control cuts off → the switch valve of the sample room closes→6#D silicon control is on, the gas charging valve opens and the sample room charges gas→ several minutes later 6#D silicon control cuts off and the gas charging valve closes→6#B silicon control cuts off and the switch valve of the X-ray separation room closes→0.5～1 second later, 6#A silicon control cuts off→ vacuum pump power supply contactor breaks and vacuum pump stops | Prerequisites:<br>Tube voltage and tube current are zero. |
| 8# | sample spin control loop<br>① controllable silicon plate 3# solid relay | Open and close: the industrial computer issues the order: spin→3#controllable silicon | entry condition: to enter the status of sample measurement and spectrum collection |

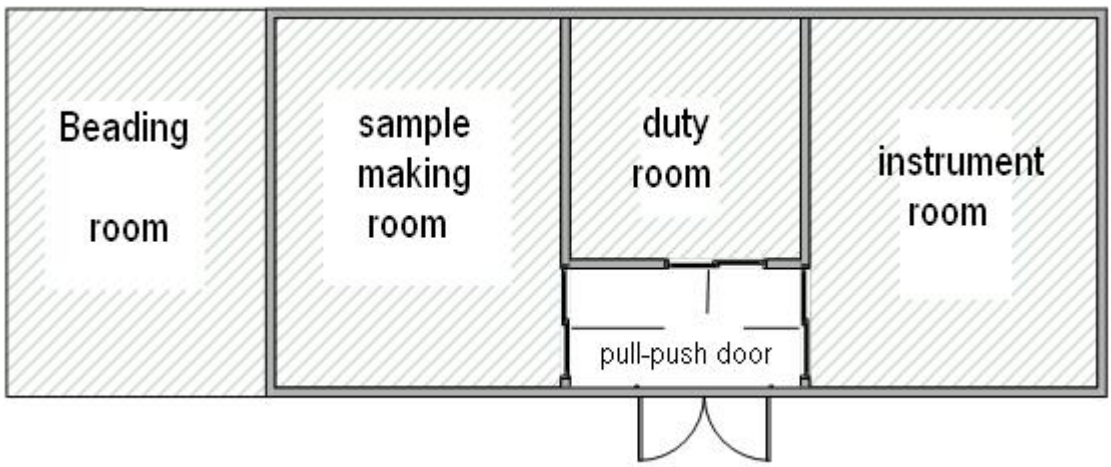
| | | | |
|---|---|---|---|
| | ② spin drive motor<br>③ rotate sensor | becomes live → spin drive motor starts rotating and drives the sample to spin → when the test time arrives, the industrial computer issues the order ”stop” →3#controllable silicon is cut off → the spin drive motor stops | the failure alarm: when the industrial computer issues the “start” order and can not receive the signal from the spin sensor, the master computer gives off alarm and stops the measurement of the sample |
| 9# | thermostat room temperature control loop<br>① temperature control<br>② heater<br>③ temperature sensor<br>④ AD、DA transfer board<br>⑤ switch valve drive circuit<br>⑥ gas charging valve | the mains switch is electrified → the industrial computer receives the temperature signal by way of AD port and compares it with the set value<br>①if it is smaller than the set value, increase output of DA port of the interface plate → increase the pressure of the adjusting silicon control → increase the heat energy of the heater<br>②if it is greater than set value, decrease the output of DA port of the interface plate→ the pressure of the adjusting silicon control drops → the heater reduces the heating energy or stops working | |

# Charpter7 Requirements of the lab and instrument installation

This chapter describes the requirements of the lab and instrument installation

**§7.1 requirements of the lab**

As WDX belongs to large precision instrument, it has certain requirements for the conditions of the lab: ① the lab must be far away from the strong vibration source and noise source. Keep it far away from the strong electro-magnetism source. Keep it away from the dust. ② two lab rooms at lease: one for putting the instrument, the other for sample preparation, where the sample preparation equipment is put. If the users demand to make melting sample, then another room for putting the melting machine is needed. In addition, the office for the operator should also be included. And the layout is usually as Figure 7-1




The lab of X-ray fluorescence analysis

**1.** the instrument room

① the area should not be less than 15m$^2$ and the width of the sliding door not less than 1m.

② The refrigerator setting: the temperature in summer should not exceed 28℃ and the temperature in winter should not be less than 22℃. The temperature should be kept within ±2℃ scope of the set value. The humidity should be maintained within the scope of 35~70%.

③ Power supply and distribution: the switch box is installed on the wall near the instrument. The requirement of the switch box is shown in 7-3.

④ The earth line: special earth line must be laid. （≤4Ω，for earthling of the instrument frame）

⑤ Keep the instrument clean. the operator should change shoes and put on working uniform before entering the room.

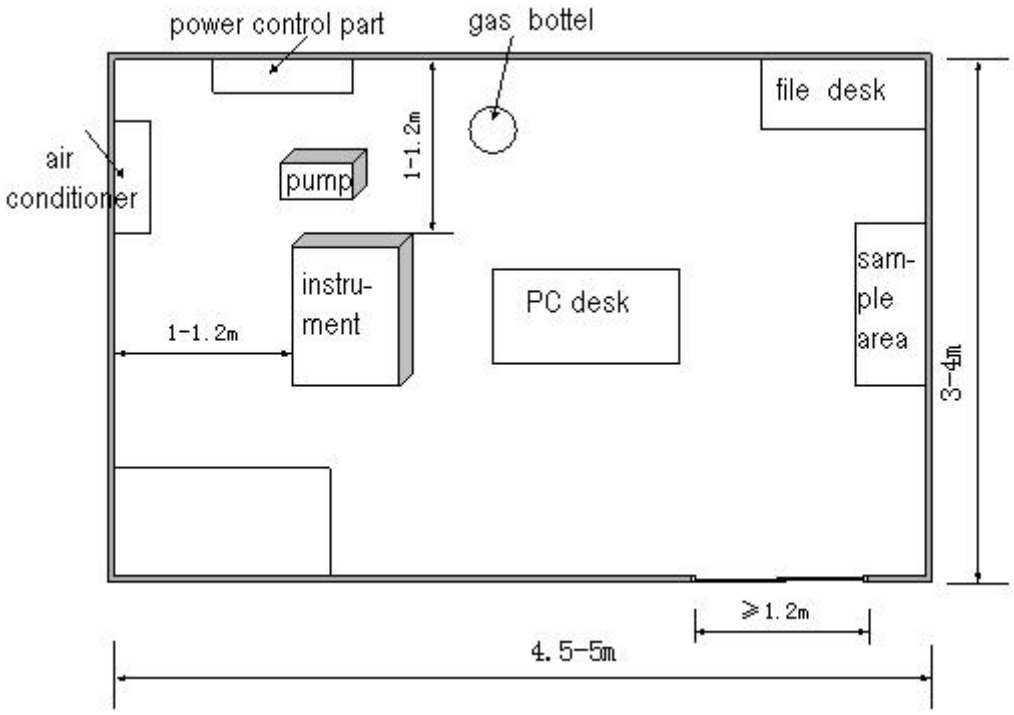



Figure 7-2 room layout of X-ray fluorescence spectrometer

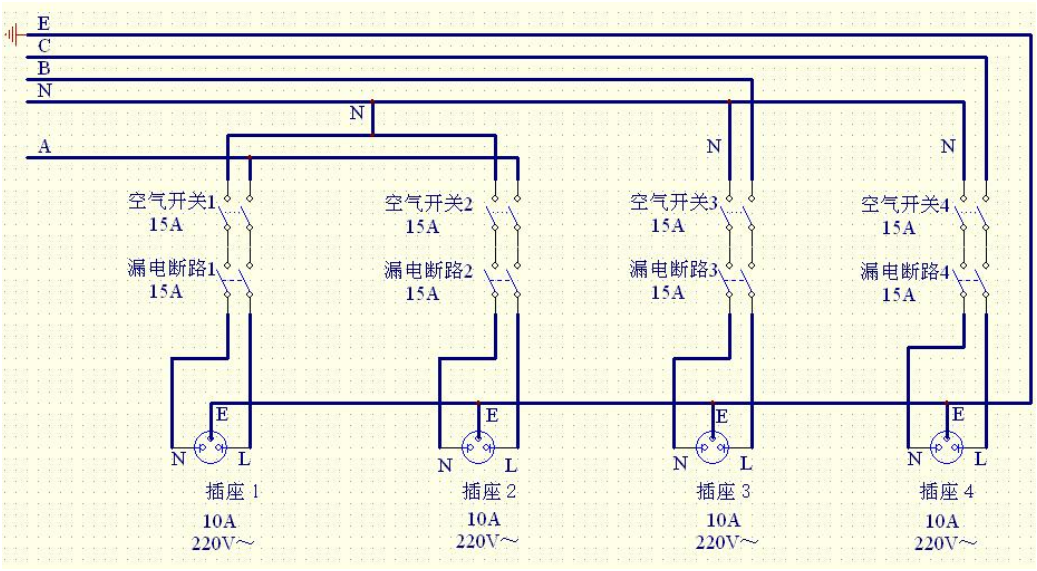



Figure 7-3 the configuration chart of the switch box in the instrument room

2. **sample preparation room**

There are bead machine, small-sized vibrating mill and other preparation equipments in this room. And at the same time, one balance with precision above 0.1%, another with 100g～200g pans and corresponding work bench should be included. Drier and sample rack (or cabinet) must be provided for putting the standard samples according their categories and quantities. And if necessary, include the agate mortar and agate vessel. The area of the sample preparation room should be no less than 15m$^2$. And the configuration of the switch box is shown as Figure 7-4.

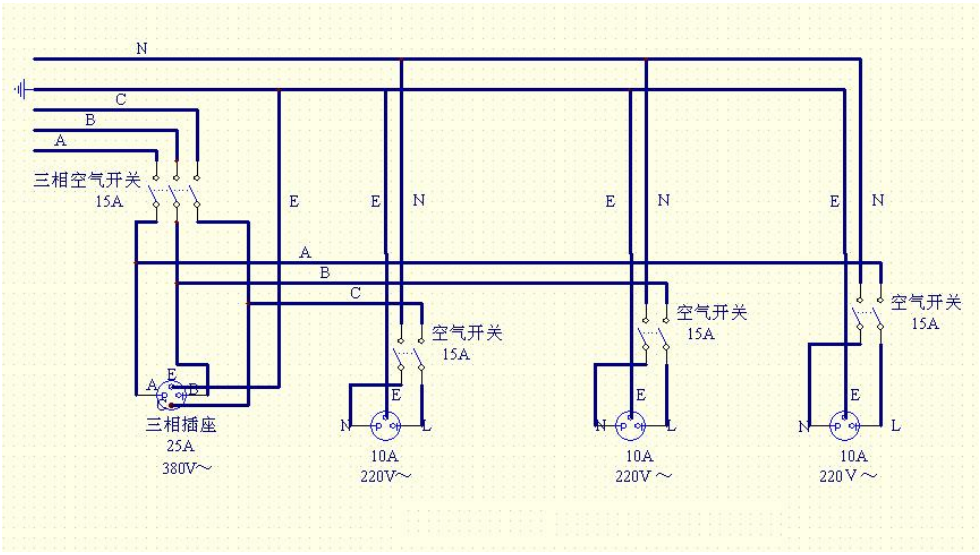



**Figure 7-4 the configuration of the switch box in the sample preparation room**

**§7.2** Check after unpacking and instrument installation

1. **unpacking and checking**

As we pack the spectrometer, vacuum pump, PC host, CRT and printer in individual boxes, check them one by one according to the packing list. And then check the following items:

① See whether there is loosening and leakage of oil pump, heat dissipater and connection pipes or oil pipe breakage or casting off.

② Open the side plates and front door of the spectrometer. Check the assembly parts, circuit board, connectors and wires.

③ Open the side plates and top cover of the thermostat room of the spectrometer. Check the measurement room, X-ray separator for each element channel and other parts fixed on the thermostat room. Check whether the connection parts of vacuum path, oil pipe and gas flow pipe and other cable plugs becomes loose or not; check whether parts of the vacuum system becomes loose or not, whether the connection parts of the vacuum path is loose or not, whether the connection is cast off.

During the examination, if there are any loose parts or come-off parts, fix them for certain. If there is any cable or connection casting, connect them again according to the illustrations in other chapters. If there is any serous leakage occurred in the transport process, notify our maintenance staff please.

Close the plates, front door and top cover after examination.

**2. instrument installation**

① Refer to chapter 7.1 for the layout of the instrument after the instrument enters the lab. The front side of the instrument is the operation space. It should not be less than 1.5 meter, and ample maintenance room should be saved for maintenance. The instrument should be no less than 1 meter to the wall. The distance between the spectrometer and the computer table is 0.5 meter.

② After the spectrometer is at place, drop the foundation bolt, let the wheel hang over (5-10mm to the ground), put the horizontal scale on the table. Adjust the foundation bolt to make it even.

③ After the vacuum pump is at place, fix the vacuum connection pipe between the vacuum pump and the spectrometer. Plug the vacuum pump station with the relay in 2 of Figure 3-1.

④ After the steel gas bottle is at place, connect it with the gas flow connection pipe of the

spectrometer as shown in Figure 5-2.

⑤ After the two stabilized power supplies are at place, put the power switch on the close position. Plug the power supply cable into the socket of the switch box. And connect the cable for supplying the spectrometer: one of the purified stabilized power supply provides electricity to X-ray tube high voltage only; and another to the spectrometer, PC microcomputer, CRT, and printer.

⑥ Connect the power supply cable of PC, CRT, printer and cable of CRT, printer, keyboard, and mouse. Connect the communication cable between PC and spectrometer. at this time, start the machine and run the spectrometer in trial.

# Chapter 8 Operation of main frame

**The operation of instrument, except that of the opening/ closing mains switch power supply, can be carried out by mouse clicking the relevant buttons on the operation interface. If we need to put in data or letters, use the keyboard to do so. After the spectrometer is live, we see the main interface first. Other interfaces are entered by clicking on the main interface. The buttons on the interface are in a chain protection relationship. When one button is pressed, relevant chain protection button are under the self-protection status and can not be operated. The black letters will be turned into vague style. This is for avoiding misconduct. (buttons on other interfaces also have a function like this ). The composition and function of the main frame is listed below.**

## §8.1 the prerequisites of entering the main operation interface

Before entering the main interface, do the following preparations:

① Make sure the vacuum path, the gas flow channel and oil path is connected correctly. And make sure there is no leakage of gas and oil in the oil cooling system and gas flow system.

② Make sure the cable plugs are correctly connected.

③ Put the switch on the front plate of X-ray tube high voltage in the open position; local/remote switch on the remote position; tube voltage, tube current, adjust the knob anticlockwise to come down to zero.

Open PR-10 gas bottle according to the chapter 5.2 and adjust the exit pressure of the gas bottle to 0.14～0.15MPa, set the pressure of the gas flow adjusting valve inside the instrument to 0.04～0.05MPa, set the gas flow rate to 70ml, and 30 minutes later, set the flow rate to 10ml. One hour later, open the machine to get it live and enter the main interface.

## §8.2 进入主操作界面的方法 the approaches to enter the main interface.

① Switch on two stabilized power supplies.

② Press the switch of the mains power supply for the spectrometer, the indicator light of the mains switch is on. The oil pump in the instrument, the heater of the thermostat room and the electric fan open automatically. At this time, the industrial computer begins to initialize, which keeps the x-ray tube power supply, detector power supply, vacuum pump, optical gate, protection cover, spinning structure are in a closed status.

③

Open PC microcomputer, CRT and printer. When the windows interface is shown. Double click the X-ray fluorescence spectrometer icon on the interface and we see the main interface of the spectrometer operation. PC micro-computer and industrial is communicating. The industrial computer will input some parameters to the master PC. In the column of the communication status the hint appears as ????. this means the operation is not available. When OK appears in this column, which suggests the initialization is completed, we can operate in this main interface.

### §8.3 the composition of the main interface

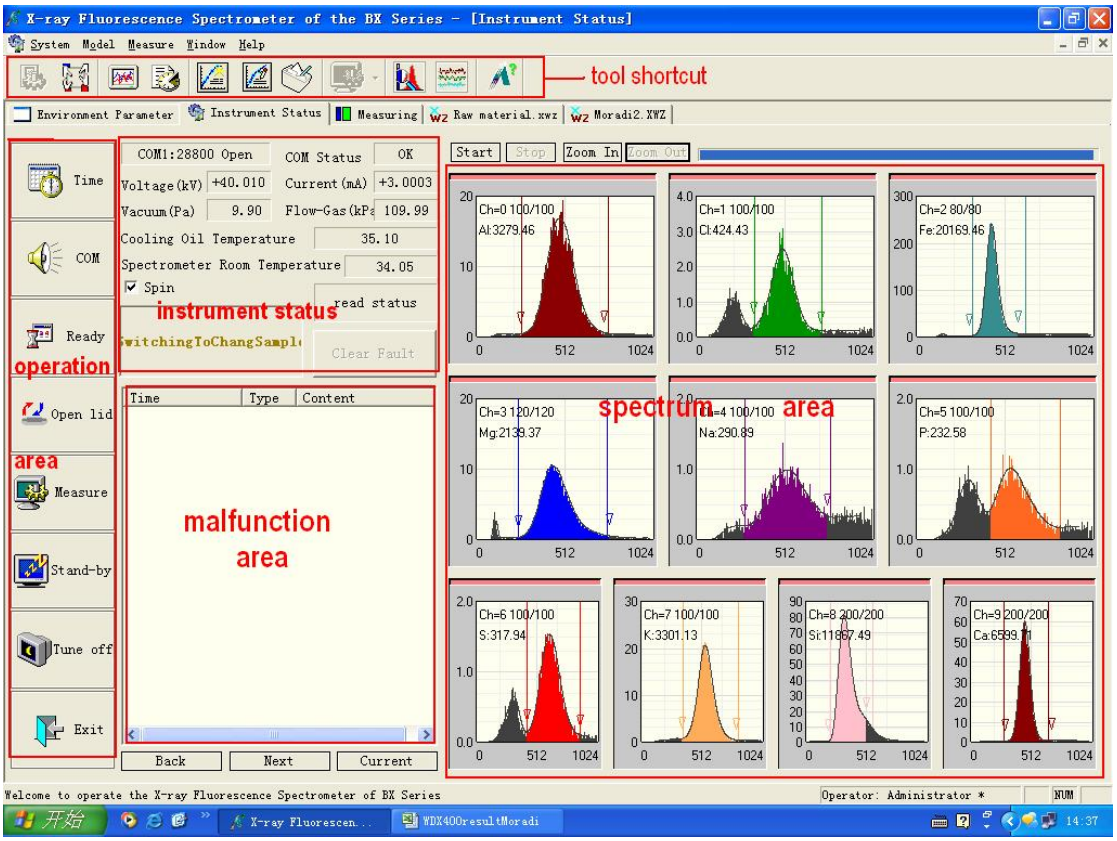



Figure 8-1 main interface

figure 8-1 is the main interface. It is composed of the following parts.

**1.** Operation buttons: they are composed of six buttons ready, change sample, measure, standby, shut, spin. They will be explained later. Time, contact are above the ready button. They are used for tuning the software, which will not be explained in the context.

**2.** Management buttons: they are composed of channel configure, drift correction,

performance test, performance record, create work area, modify work area, Delete work area, sample test, record inquiry, instrument status, environmental parameters. All of them have drop down menus.

3**. The view box of instrument status and work parameters**

Instrument parameters view box: X-ray tube voltage, tube current, vacuum degree, the gas flow pressure, oil temperature and temperature of thermostat room. A total of six parameters are shown. After master computer and slave computer has completed the communications with the slave industrial computer, these parameters box will display the work parameters at the time.

Instrument status view box include:

①Communication status box: only two states

②Work status show box: In the implementation of the operation instructions of the above operation buttons, the instrument will automatically do what are relevant to the content of the software operation. In this process, working status display box will show prepare, ready ,Measurement Preparation, initialization……. after these operations, the view box will show standby, ready, measurement, or free statuses

③Read status box: the top right box above work status view box is the display box of current communication tasks. In the course of transmission of set parameters of master computer to the industrial computer, this box shows set parameters, access parameters, and then returns to read status. In the scanning process, if the industrial computer transmits data to the master computer, this box shows that reading state and reading spectrum alternatively.

④Fault information show box: When the instrument fails in the working process, it will prompt date, time, information type (warning…… etc.) and fault content. After the troubleshooting, click the Clear trouble button, then the equipment returns to normal work

**4.** Peaks display and operating area: the left side of the main interface is the peaks display area of all elements tested. On the top are four operation buttons: begin end, zoom, uniform distribution. Their usage and function will be explained in § 9.1.

**§8.4 Definition and function of operation Buttons**

1. Standby: After the instrument is switched on, click on this button, the status box will show standby. The instrument will automatically carry out the following action

① start vacuum pump, open the switch valve of X-ray separation room and the pump station starts to pump the X-ray separation room. You can see the displayed value (Pa) decrease gradually.

② Open the X-ray tube high-voltage power supplies, the warning light is lit, X-ray tube pressure will gradually rise to 10 kV, the gas flow rises to 0.5 mA, (or 20 kV, 1.0 mA). The oil temperature is gradually increased too; in the relevant parameters Box we can observe the change of the indication value. When the pressure and gas flow achieve the above settings, work status display box will show standby. This suggests that the instrument has been in standby status and the initial preheating has begun.

2. Ready: after the standby status is achieved, click the ready button, the work status box shows ready, the instrument will automatically carry out the following actions:

① X-ray tube voltage and tube current goes up in turn until reaching the set work tube voltage (40 ~ 45 kV) and tube current (3.0 ~ 3.5 mA)

② vacuum of measurement room reaches 14 Pa or below

After achieving the above conditions, work status box will prompt ready. This means the preparation work for the operation has been done.

3. Click on this button, the Working status display box shows ready-for-changing sample, and the instrument automatically do the following:

① Close the optical gate。

② close the switch valves of sample room and the pump stations stops vacuumizing the sample room.

③ Open gas charging valve, filling the sample room with atmosphere

④ open the protective cover

Upon completion of these actions, the status display box gives the hint change sample. At this time, we load or change the sample. After the samples are put into the chamber, the measurement can be started.

4. Measure: after reaching the state of Ready, click on this button, the measurement state box

shows measurement preparations. At this point the instrument automatically does the following

① Automatically checks gas flow pressure, if it is > 103 kPa, then turn the multi-path low pressure powers of detection system, the power indicator light of detection system is lit and the detection system comes into the work state

② Close protective cover; close the switching valve of the x-ray separation room, open the sample room switching valve and the pumping stations begins to vacuumize sample room. When the vacuum of the sample room comes close to that of x-ray separation room, open the vacuum chamber switching valve and then open the optical gate. And it also monitors whether the vacuum has reached the work settings (14 Pa)

③ To monitor the pressure and current has reached the work settings. If it has failed to reach (for example, it enters measure status directly from standby status) the set value, then it will automatically adjust the pressure, current to gradually arrive at work settings.

④ After the operation is completed and the working parameters rise to set value, measurement preparation is done. The work status box shows measure by changing from ready to measure; you can click on the start button at the top of peaks display zone and it starts peaks measurement. Work Status display box shows Scanning Spectrum. However, if any of the parameters does not meet set value, measurement preparation remains unchanged, while the fault information display bar will display fault information and give off alarm.

5. Shutdown: Here Shutdown is to drop X-ray tube pressure and current down to zero, and close the power supplies of high-voltage X-ray tube, detection systems and pumping stations. Prior to this, in principle, the instrument must be in a standby status. Then click the Shutdown button. The status display box shows the initialization and the instrument automatically does the following:

① Decrease pressure, and current of X-ray tube to zero, and then close the X-ray tube high-voltage power supply and warning lights

② Close detection system power; the power indicator light turns off

③ Close the optical gate, close the switch valve of sample room, and open the gas charging valve so that the sample room is charged. Finally close switching valves of the room and the vacuum pump.

Upon completion of these actions, the work state shown in the box is idle, that suggests the

instrument has been shut down.

It must be said when the instrument is in measure or scan spectrum states, for some reason (for example, high voltage power supply ignites or proportional counter window film ruptures suddenly which leads to that the gas flow pressure drops to below 103 kPa), , Click on the button directly to shut the instrument down when this kind of emergency occurs.

6. Spin option open: this box is used to choose whether to spin samples. When the check box is blank, that means not to spin, and if we click on the check box, it means to start the spin option.

### §8.5 Check and adjust set value of instrument work parameters

The main parameters of the instrument include the following parameters. They are explained as follows:

1. The tube pressure (kV) and current (mA) settings: they are X-ray tube working voltage and current when the instrument is in a state of the sample measurement.

2. Preheat control intermittent (seconds): During the instrument preheating process, the pressure and current of X-ray tube does not allow for sudden increase. In the control procedure, the pressure and current upgrade alternately to the settings. This control intermittent is the length of stay after each increased batch. Click on the top to bottom arrow of the settings dialog to adjust the value of the time, but not less than 10 seconds .

**3.** Protection Temperature (℃): this refers to the maximum allowed value of export oil temperature value of X-ray tube cooling gasket. When the oil temperature achieves the setting export value, it will decrease automatically pressure and current of X-ray tube to zero. And it closes X-ray tube high-voltage power supply and warning lights to protect the X-ray tube. It prompts alarm at the same time.

**4.** Control temperature settings of cooling fan: when the temperature is higher than the start temperature(℃), the oil temperature regulating fan starts working, which makes the oil temperature change gradually from rise to decline. The fan stops when the temperature dropped to below (℃). After that, the Oil temperature gradually stops decreasing and changes into increase. So that stability of the oil temperature control temperature stays within ± 0.5 ℃.

**5.** Thermostat room control temperature (℃): that is the target value of thermostat room

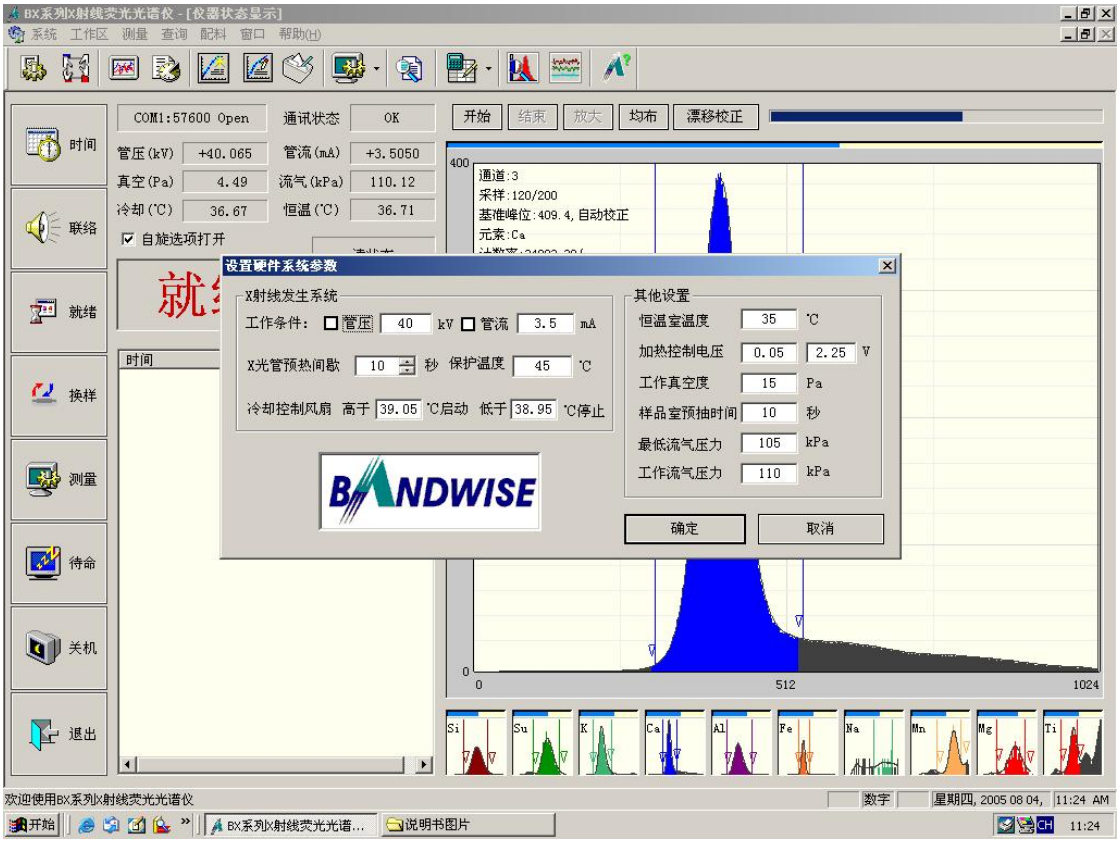
control.

**6.** Heating control voltage A (V) B (V): When the temperature of thermostat room exceeds target value, the instrument insulates itself with A voltage value. When the temperature drops to below target value, the instrument heats with the value of voltage B. A and B values are automatically adjusted by Computer so that the temperature stays within ± 0.2 ℃ over the target value.

**7.** Work Vacuum (Pa): When vacuum of the measuring room achieves the set value, the sample measurement starts.

**8.** Lowest gas flow pressure (kPa): when gas flow pressure is below this value, it would be considered failure to provide gas flow, then the power of detection system will automatically shut down to protect the gas flow proportional counter

As the above parameters have been set before the instrument leaves the factory, the operators can check the above parameters by pressing the Alt and Ctrl keys on the keyboard, and then clicking on X button in the main menu to display the dialog of the operating parameters as shown in Figure 8-2. Among these parameters, only the limits of oil temperature and control temperature settings of room temperature can be adjusted in accordance with the change of lab environment by our company installation personnel or specially trained operators. For adjustment method, see § 9.2. Other parameters have been set up at the factory. The users may not change them.


BX系列X射线荧光光谱仪 - [仪器状态显示]
系统 工作区 测量 查询 配料 窗口 帮助(H)
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就绪
换样
测量
待命
关机
退出
COM1:57600 Open
通讯状态
OK
管压(kV) +40.065
管流(mA) +3.5050
真空(Pa) 4.49
流气(kPa) 110.12
冷却(℃) 36.67
恒温(℃) 36.71
自旋选项打开
开始
结束
放大
均布
漂移校正
通道:3
采样:120/200
基准峰位:409.4,自动校正
元素:Ca
设置硬件系统参数
X射线发生系统
工作条件: 管压 40 kV 管流 3.5 mA
X光管预热间歇 10 秒 保护温度 45 ℃
冷却控制风扇 高于 39.05 ℃启动 低于 38.95 ℃停止
其他设置
恒温室温度 35 ℃
加热控制电压 0.05 2.25 V
工作真空度 15 Pa
样品室预抽时间 10 秒
最低流气压力 105 kPa
工作流气压力 110 kPa
确定
取消
BANDWISE
Si
Su
K
Ca
Al
Fe
Na
Mn
Mg
Ti
欢迎使用BX系列X射线荧光光谱仪
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# Chapter IX Instrument Tuning and Performance Test

## §9.1 Spectrum Scanning and Peak Parameters

### 9.1.1 Spectrum Scanning

In measuring state, the instrument can carry out the spectrum scanning of peaks of every element. The steps are as follows

①Click on the start button at the top of peaks display area. The status display box shows scanning spectrum that is to start the simultaneous spectrum scanning of peak of every element. We can see the peaks rising and the bar chart of scanning spectrum time extending. Spectrum scanning doesn't stop until it arrives to set value. The status display box shows measurement.

② If we want to stop spectrum scanning in the process, click on the end button to suspend spectrum scanning and then return to the status of measurement.

③ Uniform display and zoom display: Click uniform display button, the peaks of elements in the peaks display region are displayed uniformly at the same time. Click zoom display button, the peaks display area is divided into two areas: top and bottom. The bottom area is the smaller, displaying peaks of all elements. Click on the one we need to enlarge, and peak are zoomed to display in the above peak-enlarging areas.

### 9.1.2 Peak parameters

On the top left of the enlarged peaks, 12 peaks parameters are shown there:

1. Channel ×: that is the channel number of the peak in the multi-path multi-channel spectrometer, set before leaving the factory.

2. Sampling ×××/×××(S): The denominator is the set time of the spectrum scanning (in seconds), set before leaving the factory. The numerator is the time spent for actual spectrum scanning.

3. Benchmark peak (ch): this is the peaks position used for the benchmark of automatic peak correction, identified in the process of tuning.

4. Elements ×: the name of the elements with such peaks, established before leaving the factory.

5. Count rate (cps): Real-time count rate of peaks.

6. Baseline (ch): The border-bottom of interest area, identified in the process of tuning.

7. Top Line (ch): the border-top of interest area, identified in the process of tuning.

8. Peak position (ch): real-time position of peaks, identified in the process of tuning.

9. Resolution (%): peak resolution. This is an indicator parameter used for determining the quality and the life of the counter, e.g. when the resolution is bad (percentage being too large), we should replace the counter.

10. Peak correction factor: the general setting is 1.00, which can not be amended arbitrarily by the users.

11. Correction: K = 1.00 when leaving the factory, which, in practical applications, should be changed accordingly based on results of drift correction.

12. Correction: B = 0.00 when leaving the factory, which, in practical applications, should be changed accordingly based on results of drift correction.

In the following “equipment tuning method” section, the above parameters in need of tuning will be given a detailed description.

### §9.2 Equipment Tuning

The purpose of tuning instrument, on the one hand, is to observe whether parts of the instrument work well; on the other hand, to adjust and determine the work parameters and the peak parameters. Instrument tuning must be operated by specially trained staff. Only by entering the password can the operator gain access to the relevant tuning interface or menu.

#### 1. Regulation of oil temperature and control temperature of thermostat room

The premise of the instrument tuning is the measurement state. At this time, the oil temperature and the temperature of the thermostat room reach the settings values and achieve stability in the range near the settings.

The oil control temperature and the control temperature of thermostat room have been set in accordance with the laboratory’s ambient temperature range of 24 ℃ ~ 28 ℃ before leaving the factory. We can see the two temperature settings by pressing on the Ctrl and Alt buttons on the keyboard and press X button at the same time. If environment temperature of users’ laboratory is between 24 ℃ ~ 28 ℃, there is no need to modify the settings.

We can judge whether the instrument runs in a stable working condition by checking the real-time

values of the oil temperature and the thermostat room temperature displayed on the main menu. The requirements are as follows:

① Oil temperature: this fluctuates within ± 0.5 ℃ of the set oil temperature.

② Thermostat room temperature: this fluctuates within ± 0.2 ℃ of the set thermostat room temperature.

In case all the above requirements have been met, do the next step of the instrument tuning.

Due to regional differences or power or installation location of laboratory air-conditioners, the oil temperature or the thermostatic room temperature are out of control, including the following:

①Top and bottom limitations of oil temperature lose control: The top oil temperature limit runs out of control means it is +0.5 ℃ higher than the set oil temperature and remains unchanged. It can not be reduced to the setting value. To solve this problem, first check whether the two cooling fans behind the oil temperature radiator have been opened, and then check whether there is oil flow on surface of the oil cup. If we confirm that both of them are under normal circumstances, however, the oil temperature limit is still out of control. And the temperature stabilizes basically at a certain temperature below 40 ℃. We can increase the set oil temperature by using the keyboard. They must be 0.5 ℃ higher than the highest actual value, but not more than 45 ℃. If it exceeds 45 ℃, adjust the inter distances of the air-conditioner and the instrument or replace the air-conditioner with a more powerful one. Bottom limit of oil temperature runs out of control means the oil temperature always fails to level with the setting value. At this time, first inspect whether the two cooling fans behind the oil temperature radiator is stopped. If it does not stop, it means there is a failure in the oil temperature control loop. Repair it at once. If fans do have stopped, but the oil temperature still can not rise, reduce the set value with the keyboard. Make it be 0.5 ℃ lower than the minimum Real-time oil temperature value.

②Temperature of thermostat room went out of control: this is also divided into two cases, the top limit and the bottom limit. When the top limit goes out of control, improve the setting value of the control temperature. Let it be 0.2 ~ 0.3 ℃higher than the real-time value; When the bottom limit runs out of control, lower the settings and make it be 0.2 ~ 0.3 ℃ lower than to real-time value.

**2. Peak adjustment of various elements**

Click the Start button to stop the scanning of the peaks of various elements. Put it in zoom state to

observe the position of every peak and adjust the high-voltage potentiometer of detectors of every fixed channel installed in the left panel of the thermostat room. Let the peaks of various elements be within the following range.

③ Peaks of Al, Fe, S, K, Cl should be in between 480 ~ 530 ch.

④ Peaks of Si, Ca should be between 420 ~ 510 ch.

⑤ Peaks of Mg, Na should be between 510 ~ 640 ch.

**2. User interface for system settings and method for setting the spectrometer parameters**

In condition that the instrument is fully stable, peaks of various element have been adjusted and collected in the main user interface, click the channel configuration button to show password box and input the passwords from keyboard.

Click OK after inputting the password, and then we enter the multi-channel spectrometer system settings interface (9-1). This interface covers on the main interface. In the middle of this interface is the peaks display area, with longitudinal coordinates being the count rate (cps) and horizontal coordinates the channel sites (maximum 1024) of multi-channel spectrometer. At the top of peaks display area, a row of labels from the channel: 0 → Channel: 9 are channel labels for multi-path multi-channel spectrometer, each corresponding to an element. Click on a label and that will show the corresponding elemental peak. Click on the drop-down button in the bottom of peaks display area the menu for all elements definition is pulled out. On this menu we may amend the definition of the various elements. The elements (or their compounds) corresponding to each channel have been set in accordance with user requirements before the instrument leaves the factory.

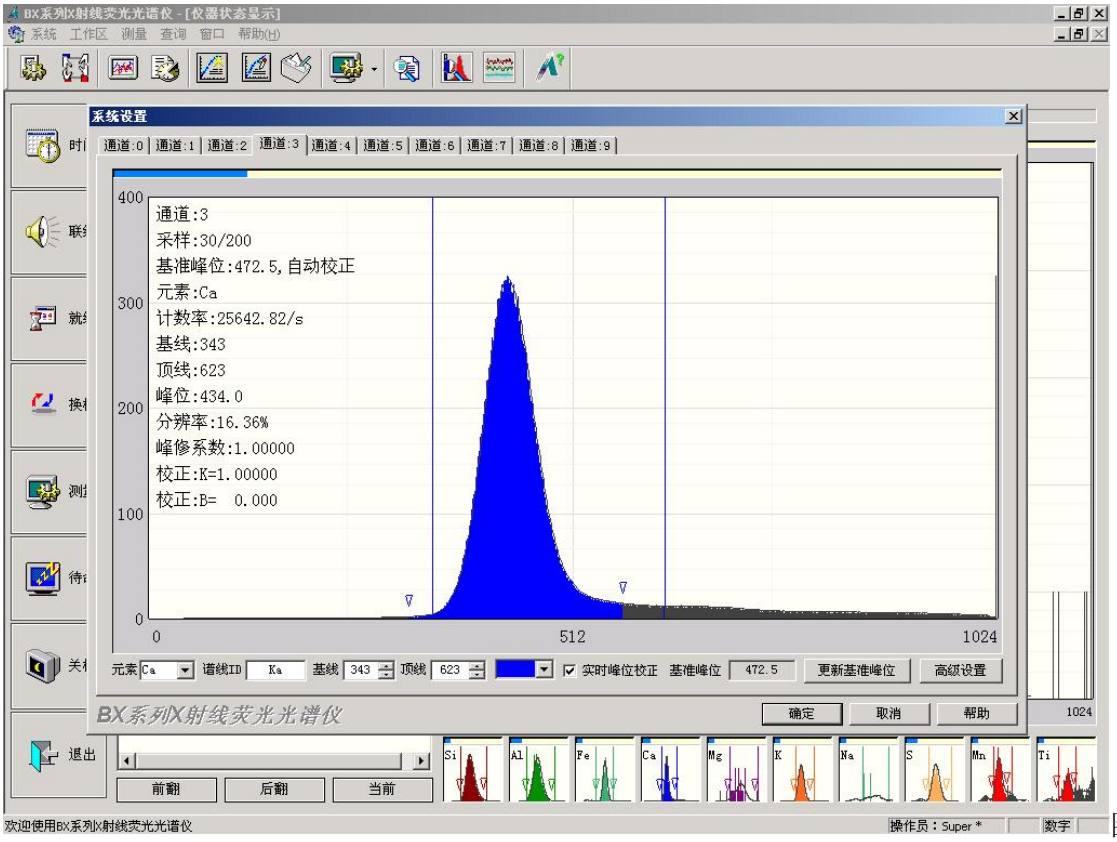


图 9-1

Below peaks display area, there are buttons and parameters view boxes in addition to view box of names of elements.

①Baseline move button and Baseline channel site view box: left mouse click the button on the right side of 基线 343 to see the baseline moves left. The baseline site value becomes smaller. If there is a continuous click of the button, then the baseline moves left ceaselessly. On the contrary, if we click the button, the baseline moves right and the baseline site value becomes larger.

②Top line movable button and top-line channel site view button 顶线 623: the operation is the same as the baseline movement.

③: This is the color option for the peaks. The left side shows the color now used by the peaks. Click the button to pull color selection box. Click a certain color in the box, and you can change the color of the peak.

④Real-time peak Correction: Click on the small box on the left side of the real-time peak correction box. Then is displayed, which suggests that a real-time correction is implemented on

the peaks of the elements; click on the box again,  is displayed. That means no peak correction would be done to the peaks of the elements.

⑤Benchmark peak position display box and update the BENCHMARK peak position button. The so-called benchmarks peak position is the peak position of a reference spectrum kept by the multi-channel spectrometer at the correction time of peaks of necessary elements. During the instrument tuning before leaving the factory, the benchmark peak positions of all the elements are preserved in the multi-channel spectrometer and they are displayed in the benchmark display box. If you click on the button update the BENCHMARK peak position button, the multi-channel spectrometer will remove the original benchmark peak position value, and change into the peak position value of the current peak displayed and saved in the peak display area, that is, to use the existing peak position as the benchmark.

⑥Advanced Settings button: Click the Advanced Settings button, and dialog as follows pops up:


As for the correction factor f, please do not change; the smooth width is generally determined automatically based on spectrum shape and count rate, which does not allow amendments; dead time is identified by the characteristics of the detector, the nuclear electronic circuits and the spectrum collection circuit. Users do not change it please. Only the tune personnel of our company have the right to decide and adjust the advanced settings. After the completion of various channels settings, click the OK button to save your settings and return.

Steps of setting the above parameters of the spectrometer are as follows:

① In case that the instrument has been preheated fully in the measuring state and has achieved stability, collect peaks of all elements at the set time, and adjust the peak position of the spectrum on the requirements of the last chapter.

② Click Channel Configuration button, enter the correct password and step into the system settings interface of the multi-channel spectrometer (9-1).

③ Click the update benchmark peak position button one by one by starting from the element peaks corresponding to channel 0. Re-establish the benchmark peak positions of all elements. At the same time, adjust the position of the baseline to remove noise signal; adjust the top-line position to remove the possible interference of peaks. In the absence of interference peaks, set the top line in the relatively flat part peaks in the right side skirts. Note: Baseline channel site may not be <150 ch; top Line channel site may not be> 950 ch.

④ Click on real-time peak correction box so that it is ☑.

⑤ Click the OK button and let the settings enter into force and get saved. Then return to the main interface.

At this point, the instrument tuning and the setting of the operating parameters are finished. You can enter the operations of performance appraisal and instrument calibration.

## §9.3 Instrument performance test

### 1. Conditions of performance test

Prerequisites for a performance test are: ① all working parameters have been set on the above requirements. ② the instrument has fully preheated in the measuring state and the oil temperature and the thermostat room temperature fluctuate in the vicinity of the setting values. ③ the vacuum degree of X-ray separation room ≤ 10 Pa, the vacuum degree of the sample room≤ 13 Pa. ④ change of environment temperature in the laboratory: setting temperature ± 2 ℃.

### 2. Performance test content

Performance test is to evaluate the main performance indicators of the instrument, which include:

① Stability of X-ray fluorescence count rate of various elements. The square deviation measurement value σ (actual) of count rates of all elements within eight hours are required to

be≤ 1.5 times the theoretical value σ (theory); within 24 hours, σ (actual) ≤ 2 times σ (theory).

② Stability of X-ray tube voltage and current: better than 0.1 percent in 8 or 24 hours.

③ Thermostat room Temperature control results : meeting the set value ± 0.2 ℃ in 8 or 24 hours.

④ Oil temperature control results: meeting the set value ± 0.5 ℃ in 8 or 24 hours

### 3. Operation Interface and Steps of performance test

Confirm the sample has been put into the sample room before conducting the performance test. If there is no sample, operate on the change the sample on the main menu. And then click on the performance test button on the main interface to enter the performance test interface (9-2).

The central part of the interface is the count rate data table of various elements. The bottom is the stability curve display area. The operation steps of the button and the setting are as follows:

① Set the time or number of times of a performance test

Performance evaluation tests can be carried out by the set time, such as 8 or 24 hours; you can also set the frequency of spectrum scanning to do this.

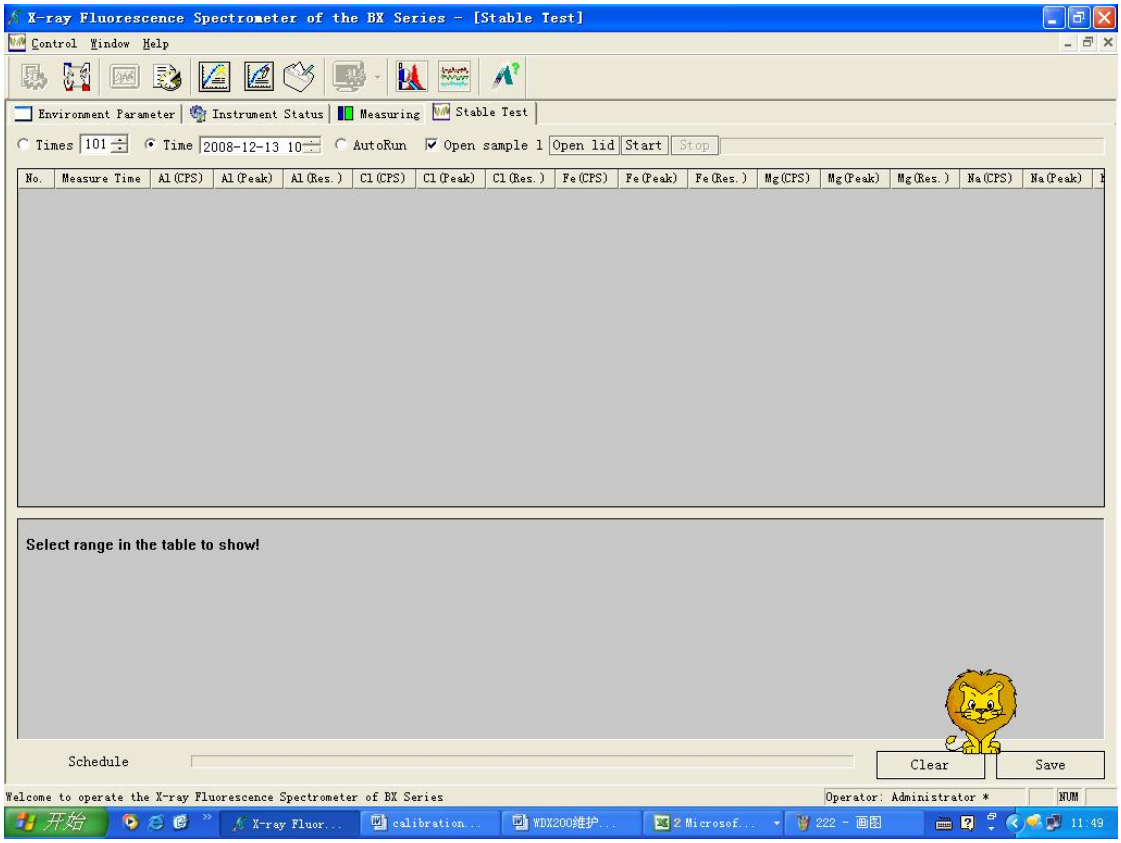



Figure 9-2 Performance testing initial interface

In the software, the default setting is to conduct the test in 24 hours. In the radio box to the specified time we see . Behind it, in the time display box, shows the date and time 24 hours later count from now on. If you want to amend the completion time of the test, use the keyboard or mouse to modify the time displayed in the box. If you want to do the test in accordance with the repetition times, use your mouse to click on the radio box set number of times. Make it be and then click the right arrow button to set the repetition times of the test.

② Select the test method

The check box Open sample 1 in the same line refers to when every repeated measurement takes place, the light gate is closed, the sample room is filled with atmosphere, the protective cover opens and shuts…… and another repeated measurement begins. The main objective is to test the reliability of the light gate mechanism and the flip device and the repeatability of the measurement. If this kind of test is not required and only stability test demanded, click the checkbox to change it into .

③ Begin testing

Upon completion of these settings, click the Start button to start performance testing. After each test, the results will be added to the list on the first half of the interface, which include tube voltage, tube current and other conditions (see Figure 9-3). This doesn't end until the set times of the tests or the set time run out. On the bar table in the bottom of the interface we can also see the time process or the number of times processes.

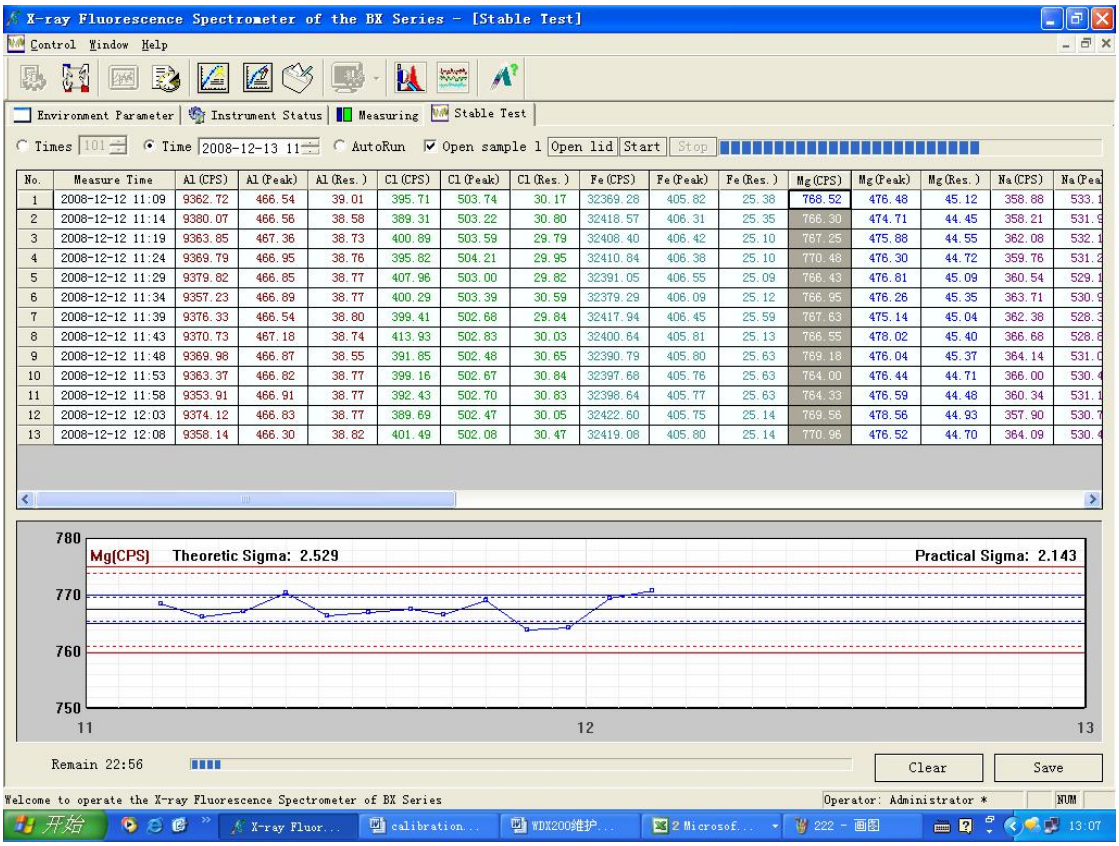



Figure 9-3 performance test interface

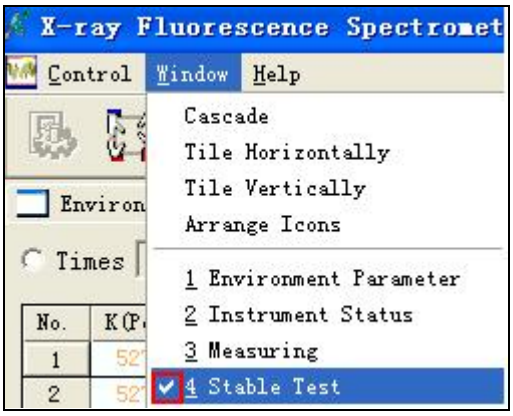
④ stability curve

If we need to observe the stability curve, click on the title bar in the list at any time. Trend curve corresponding to the column will be displayed in the bottom curve displaying area. In the trend line, the red dotted horizontal line stands for three times the standard deviation while the red dotted horizontal line 1 times. In the upper right corner of the trend line shows the standard deviation of the actually measured results. If we click on the column corresponding to the count rate of a certain element, a red horizontal line on behalf of three times variance of theoretically statistical fluctuation and a blue horizontal line on behalf of 1 times will be displayed. In the upper left corner of the trend line displays the

variance of theoretically statistical fluctuation. We can also drag the mouse in any starting position of a certain row to show the trend line in a certain period of time.

⑤ Performance test interface and the switch window for the main user interface

In the performance test process, if we are to observe the work state of the instrument and peaks of all elements, we can click on the window button at the top of the interface to pull out the small window menus:

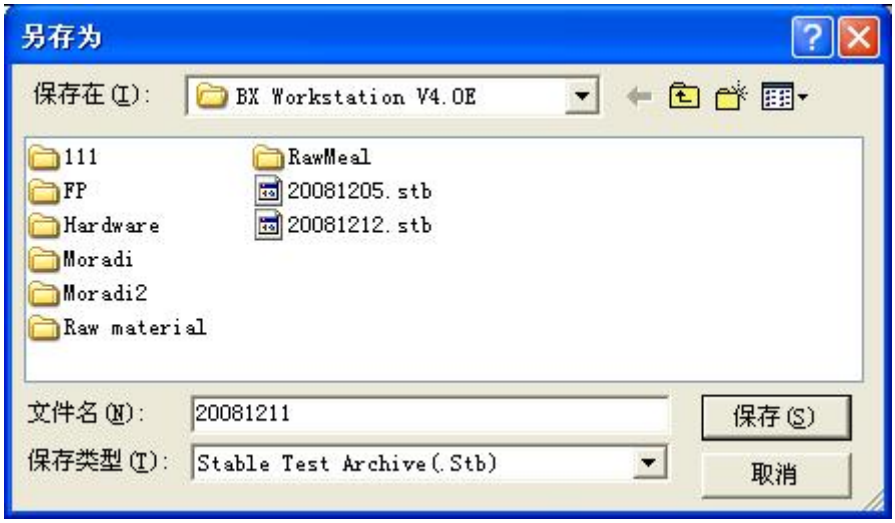



Icon ✔ indicates that we are currently in the performance test interface. Click the instrument status display on the top row. And then we return to the main interface.

It should be noted that in a performance test process, we need to guard against the various peaks and the operating parameters from suffering abnormal change or giving a fault alarm. So the test should be placed on the main interface. When there is a need to observe the performance test results, click the window button in the top row of the main menu, pull out the window menus and click on 3 performance test. Then you can enter the ongoing performance test interface and study the stability test results and stability curve.

⑥ Save the test results

After the performance test is finished, click the Save button on the bottom right corner of the interface. The following dialog pops up:



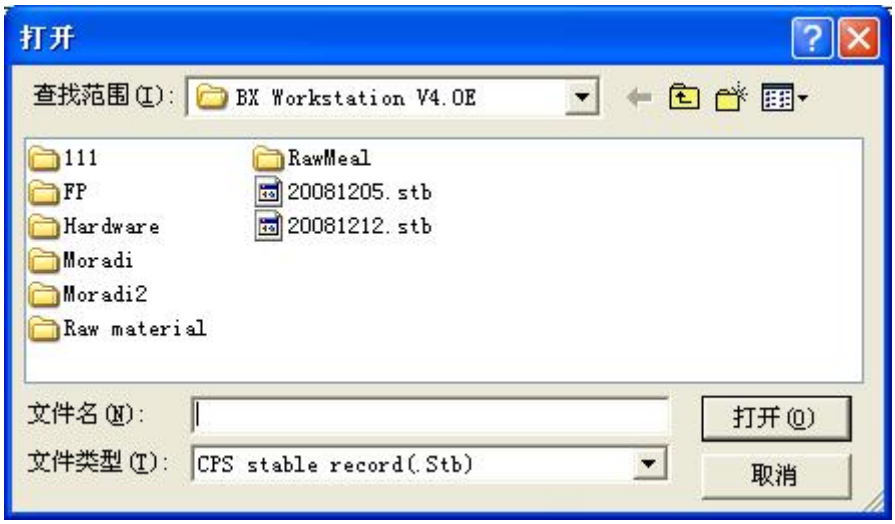

In the file name (N) dialog, enter the file name of the test results for the future enquiries. Operators can input a file name easy remember, e.g. the start and end dates of the test, time and so on. Click the Save button after entering the file name. And the test results are saved.

⑦Terminate the test

If we want to terminate the test in the process, click on the stop button in the line on top of the data table. To save the test results, do according to the above ⑥. If we do not need to save the results, click on the button ☒ below the upper right corner of the interface. Then we return to the main user interface.

⑧Inquire the previous test records

Click on the performance record button at the top of the main interface and we see the following dialog:



Click the left and right arrow key below the file table to look up the file name we want to. Reveal it in the blank box behind the file name (N). And then click the open button on the right side of the box to open the performance test results under the file names. Figure 9-4 is a performance test opened. Click the  button below the upper right corner of the interface to exit this interface and return to the main interface.

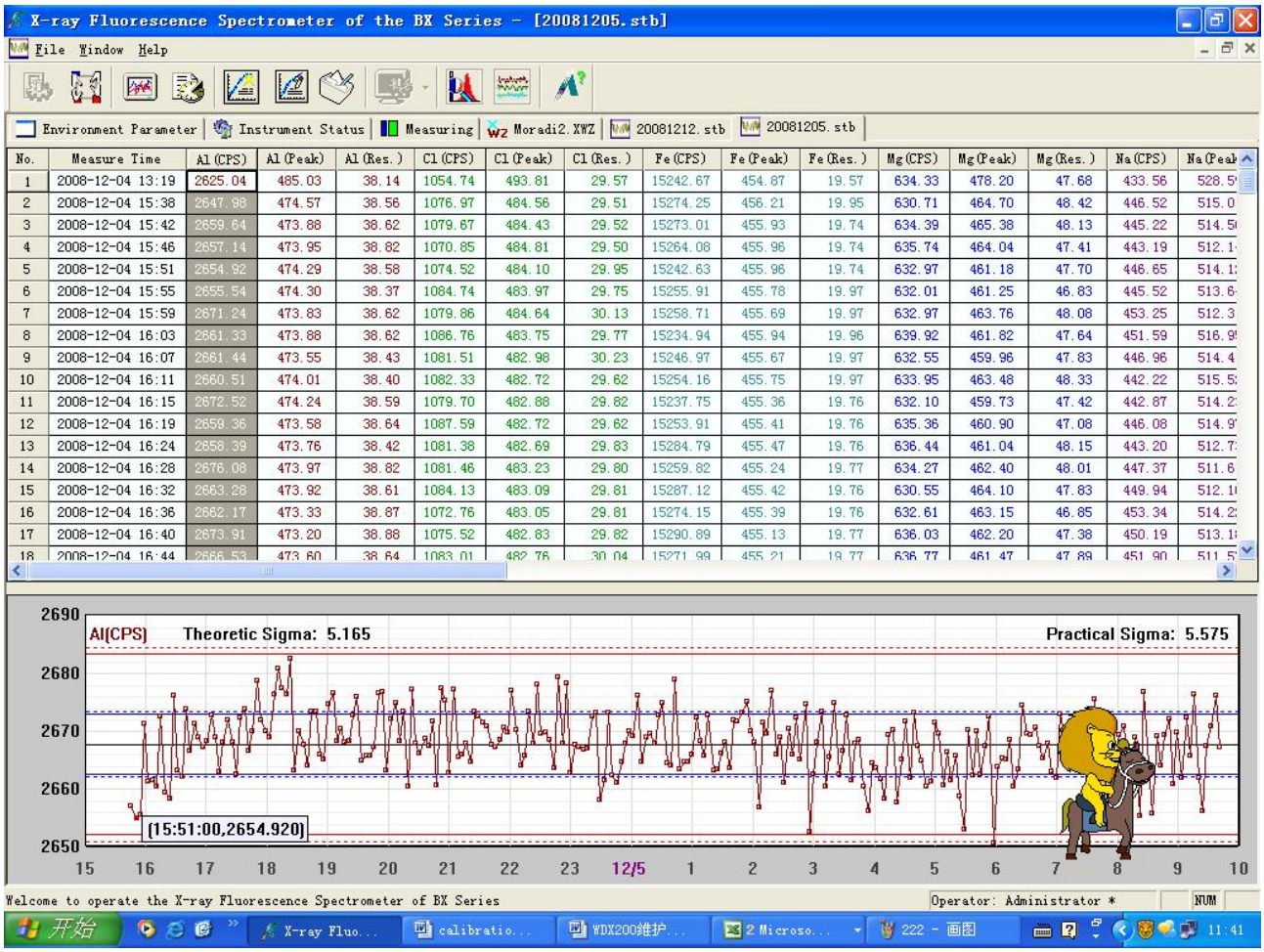



Figure 9-4 Performance Test Record

# Chapter 10 Operation rules of the instrument and routine maintenance

## §10.1 Operation rules

### 1、Start-up process

（1） Check power supply. Confirm two power supplies are working properly.

（2） Open the master valve of the PR10 gas bottle. Check the export gas flow pressure of the primary decompression gauge. It should not be less than 1 MPa. Otherwise replace PR10 gas bottle. The export pressure on the primary decompression gauge should be within 0.14 ~ 0.15 MPa. Otherwise, turn the adjustment Knob to make the export pressure be at 0.15 MPa.

（3） Switch on the power of the instrument. Let the instrument complete initialization and get ready to accept the master PC command.

（4） Open the power supply of the computer (including CRT power supply and that of the printer). Start WDX workstation software after entering the Windows operating system. We may have to wait a little time for WDX software to establish the communication links with the instrument. Wait until the instrument state is changed into free. At this time, the instrument automatically adjusts the gas flow pressure and will not reach 110 kPa immediately. But we can proceed to the next step. Take down the present time T.

（5） Click on the standby button to start the pump stations. The high-voltage power rises to about 10 kV, 0.5mA (20kV, 1.0mA). Wait until the instrument state change into the standby status. At this time, the constant temperature system adjusts automatically but will not reach the setting value immediately. We can proceed to the next step, but we mustn’t click on measure or click the start button at the top of the spectrum.

（6） Click the ready button. And the high-voltage power supply will rise slowly and steadily to the settings value. In the end, the instrument shows the ready state. At this time, the gas flow pressure and the thermostat room temperature in particular still need some time to rise to the setting value.

（7） Click change the sample button, put the sample, usually used to test the stability of the instrument, into the sample room and then click the ready button. Time counts from T on and four hours

later we can proceed to the next step.

（8） Click the performance test button on the toolbar. Enter the test procedures of count-rate stability. Click the start button on the interface to start measuring the count rate. Within two hours in general the count rate will change according to the regulation of temperature of the thermostat room. After that, the temperature reaches the set value and maintains constant. Count rate of each channel tends to stabilize. After the count rate is basically stable, click the Clear button at the bottom right corner of the performance test interface to remove the unstable records, which makes it easier for the follow-up observation. If the instrument stands by for a very short time (several hours), then generally after 2-3 hours the instrument will achieve stability. If it stands by for a long period of time (several days), it is better to conduct other operations after the 12-hour count-rate stability test confirms the system has really stabilized.

（9）Before the resumption of the use, we need to monitor the measured samples and compare it with the initial value. If there is a large difference, correct the count rate drift. Start daily work after the completion of measurement.

**2、Shutdown process**

If the spectrometer won't be used for a short period of time (for example, within 3 days), switch the spectrometer to standby state and do not shut it down. If it won't be used for a long period of time (above 3 days), shut it down completely. The approach to do this is to click the Standby button. Wait for one or two minutes and the spectrometer is changed into standby state. We can see the high voltage power decrease to 10 kV, 0.5mA (20kV, 1.0mA). If you need shut it down, then click the shut down button. When the instrument state changes into free, and vacuum pump stops working, we can then cut off the power to the spectrometer. After the spectrometer powers off, the gas flow system automatically cuts off. For a more-than-three-day halt, close down the PR10 gas bottle please.

**3、Standby recovery process**

Standby recovery process must start from step 6 in the start-up process of the spectrometer because the gas flow system does not cut off. Step 7, waiting four hours, is not necessary. Step 8 of the count-rate stability can be shortened to one hour or less. As long as the count-rate stabilizes, carry out step 9. Measure the monitoring sample and compare it with the initial value. If the difference is too large, execute count rate drift correction. Upon the completion, access to the measurement interface and

carry on the daily work.

**4、When must count rate drift correction be done?**

（1） When work shift comes, measure the monitoring sample in the "raw material testing zone". If the results differ considerably from the original value, do count rate drift correction. After the correction, use the monitoring sample to check whether the measurement results are in line with the original value.

（2）After the spectrometer reboots or recovers from the standby state and achieves stability, measure the monitoring sample before carrying out the conventional measurements and compare the results with the initial value. If they are in large difference, do count rate drift correction. After the correction, use the monitoring sample to check whether the measurement results are in line with the original value.

（3） Measure the monitoring sample if adjustment is made in the test conditions such as thermostat room temperature, tube voltage, tube current, cooling oil control temperature. Compare the results with the initial value. If they are in a large difference, do count rate drift correction. After the correction, use the monitoring sample to check whether the measurement results are in line with the original value.

（4）If the instrument was maintained and has achieved stability after its rebooting, measure the monitoring sample before carrying out the conventional measurements. Compare the results with the initial value. If they are in a large difference, do count rate drift correction. After the correction, use the monitoring sample to check whether the measurement results are in line with the original value.

**5、Which quantities should be paid attention to in daily observation?**

（1） We should pay attention to whether temperature of the thermostat room fluctuates in range of ± 0.2 ℃ the setting value and the oil temperature in range of ± 0.5 ℃. If they exceed the setting temperature for a longer period of time, reduce or increase the appropriate settings.

（2） We should pay attention to whether the pressure of gas flow fluctuates within the range of 110 ± 0.1kPa.

（3）Attend to the export gas pressure of PR10. See if it is less than 1 MPa. If it is, change the gas bottle.

（4）Check before putting samples. See whether the sample is firm enough or the surface is clean. Pay attention to the place where the sample lies in the sample cup. The sample must be rotated freely after putting into the sample room.

（5）Time is usually certain from issuing a measurement directive to completing the measurement. If it exceeds the normal time too much, switch to the status interface to see whether there is problem about the vacuum.

（6）After the measurement, check whether the content is reasonable or not, which comprises a good way to find the problem.

（7）When doing count rate drift correction, see whether the slope of the correction factor of various channels should be 1. If there is too much deviation, there is certainly a problem.

（8）See whether the shape of the spectrum of every channel is normal enough. The potential problem is that noises may occur in front of a spectrum, which will affect the accuracy of measurement.

### §10.2 The daily maintenance of the instrument

Our instrument is a large precision instrument. Note the following in the use process.

**1、Vacuum System**

- Every other week, take out the O-ring on top of the sample room. Wipe the ring and its installation slot with lens cleaning tissue with absolute ethanol. In the ring surface, coat it with thin vacuum silicon grease so as to ensure the sealing of the sample room.
- Inspect vacuum oil very month (observe the oil scale with flashlight). The oil should be maintained clear. If it is found cloudy, replace it immediately. In addition, replace the vacuum oil once for every other year in principle. During the replacement, use a small amount of clear oil to clean the pump after the dirty oil is disposed. After the washing oil flows out absolutely, add the clear oil till it reaches the middle of the oil scale window. The brand name of the pump oil is HFV-A or HFV-M (produced by Shanghai Huifeng Petrochemical Company Limited).

**2、Gas flow system**

- When the gas flow pressure in the bottle is ≤ 1 MPa, change the gas bottle. Before changing the gas bottle, shut down the instrument and cut off the mains power.

- If the gas flow are turned off for longer period of time or for other reasons, let the gas flow run for at least 2 to 4 hours before entering the measurement state when reopening it.

**3、X-ray tube cooling system**

Check the oil surface of the oil cup once every month. When the oil pump starts, there should be oil fluctuation on top of the oil surface and then it remains at a normal level. If the oil level is comparatively low, check whether there is a leakage point in the cooling system.

**4、Sample tablet**

The sample tablets of our instrument are usually the ones with substrate of boric acid. If there is crack or flake on the surface of the tablets, do not put it inside for measurement. This is to prevent the powder from falling onto the beryllium window of the X-ray tube. Suppose such a situation happens, remove them with aspiration ball. Then wipe off the powder with cotton wool with absolute ethanol. **It should be noted that no hard objects (including fingers) are allowed to come into contact with the beryllium window.**

**§10.3 Common failures analysis table**

| No | Occurrence | Cause of Failure | Approach |
|---|---|---|---|
| 1 | Gas flow pressure drops sharply and the vacuum deteriorates. | The thin window film of certain proportional counter breaks down | Stick the import and export hoses of the counter by turns. If there is an apparent increase in the pressure when the import and export hoses are stuck. It means that the thin film of the counter breaks off and we need to replace it. |
| 2 | The vacuum of the x-ray separation room | ①the oil in the vacuum pump deteriorates | Change the oil. |

| | | | |
|---|---|---|---|
| | deteriorates | ②leakage in gas charging valve and manual gas release valve | Replace the sealing parts or valves after checking these two valves one by one. |
| | | ③There is a leakage in X-ray separator or connector of vacuum tube. | Dip the brush in absolute ethanol and smear it on the sealing points. If alcohol at some point is inhaled and the vacuum deteriorates significantly, it means the gas leaks here. Replace the sealing parts there. |
| 3 | The vacuum degree of the sample room deteriorates but that of x-ray separation room remains good. | ①there is dirt on top of O-ring or on the protective cover. | Clean the surface below O-ring and protective cover. |
| | | ②Sealing O-ring of spin and drive axis wears off. | Replace the O-ring |
| | | ③A leakage occurs to the vacuum pipe of the sample room | Check the joints one by one by smearing alcohol on. Shoot the leakage problem If there is any. |

| | | | |
|---|---|---|---|
| 4 | Oil fog condenses between the Protective cover and the middle board. | ①a leakage occurs to X-ray tube cooling gasket | Tighten the sealing screw and replace the O-ring. |
| | | ②a leakage occurs to oil seal of X-ray tube cooling gasket | Take off X-ray tube cooling gasket and examine. Contact the Company for maintenance |
| 5 | Oil level in oil cup drops | a leakage occurs to oil pipe or oil pump. | Check pipeline joints and pumps |
| 6 | Unstable X-ray tube voltage or tube current | ①poor contact of high voltage cable plugs or filament cable terminals | Check the plugs and the terminal wiring in relevance |
| | | ②high-voltage power supply failure | Contact with the maintenance center of the Company |
| 7 | Protective cover can not be opened | ① Dislocation and damage of optical gate positioning photoelectric switch | Check the photoelectric switch of the optical gate |

| | | | |
|---|---|---|---|
| | | ②Dislocation and damage of protective cover positioning photoelectric switch | Check the relevant photoelectric switch |
| 8 | No spinning signal | Spin detection photoelectric switch is damaged | Inspect the relevant photoelectric switches |
| 9 | The oil temperature loses control and is apparently high | Cooling fan is damaged | Check and replace the cooling fan |
| 10 | The temperature of the thermostat room loses control and is apparently low | Ribbon heater or pressure adjustment controllable silicon is damaged. | Check and replace the ribbon heater and the pressure regulation controllable silicon |

Appendix:

**Tuning of slave computer in WDX series spectrometers**

**1、 The directory structure**

C:\XRFT Root

- CFG: Configuration files subdirectory
- OBJ: Object files subdirectory
- SOURCE: Source program subdirectory (No such a directory in the release version)
- TUNE: Tune and diagnostic subdirectory

**2、Sub-components tuning**

When slave PC starts up, it will run automatically C: \ AUTOEXEC.BAT. This is a batch file. The last line of C: \ AUTOEXEC.BAT is a statement to start the main program. There may be various mistakes in the initial power-on tuning, such as wiring error, photoelectric switch dislocation and so on. We suggest you not run the main program. The method to avoid this is to press the F5 key does not run through the computer started the process of holding down to skip C: \ AUTOEXEC.BAT files, directly to the DOS prompt C: \>, type C: \> CD \ XRFT ↙ (↙ representatives return to, the same below; the underlined letter is computer display content, need to type), the screen shows C: \ XRFT>, and then to carry out sub-components tuning.

（1）Samples covered tuning C: \ XRFT> TUNE \ TUNESLID ↙, into the sample covered tuneger.

F1-off samples covered; F2 - open the sample covered; F3 - to stop movement

Observe and adjust the block-and photoelectric switch positions, making the closure and open the sample covered all parked in the correct location.

（2）Commissioning-gate C: \ XRFT> TUNE \ TUNESHUT ↙, entered the gate-tuneger.

F1-closed-gate; F2 - open-gate; F3 - to stop movement

Observe and adjust the block-and photoelectric switch positions, making the closure and open-gate can be parked in the correct location.

（3）Spin tuning C: \ XRFT> TUNE \ TUNESPIN ↙, spin into the tuneger.

F1-start spin; F2-stop spin

Type F1 start spin, spin motor sports should be in the upper left corner of the screen figures should

be uniform uninterrupted 40 and 00 in change, if change uneven, photoelectric switch positions need to be adjusted so that it evenly.

（4）Vacuum pump station system tuning C: \ XRFT> TUNE \ TUNEPUMP ↙, tuneger into the pumping station.

F1 - open the vacuum pump; F2 - closed vacuum pump

F3 - open the valve 1; F4 - closed a valve (valve 1 - Room by switching valve)

F5 - open the valve 2; F6 - Close valve 2 (Valve 2 - sample room switch valve)

F7 - open the valve 3; F8 - Close valve 3 (valve 3 - sample room air valve)

Commissioning should be adopted after releasing vacuum manually, disconnect the power supply pumps, for safety reasons.

With the corresponding keys observation valve and relay (contact) the action is correct, incorrect wiring needs to be adjusted.

If the three-phase vacuum pumps, need to connect the power vacuum pump, open the valve 1, an instant type F1, will soon enter F2, check the electrical direction, if not correct, the need to exchange any two of the three-phase input of the line.

（5）X-ray tube high-voltage power calibration and tune C: \ XRFT> TUNE \ CALIHT ↙ into the process

**Key description:**

F1 - the importation of pressure, flow control voltage value, format, for example: 1.5,1 ↙, set up the pressure control voltage 1.5 V, the flow control voltage of 1.0 V.

F2 - the importation of pressure, the flow sheet value of the first show, for example: to read out the pressure to 15.2 kV, the flow is 1.25 mA, enter 15.2,1.25 ↙

F3 - started reading the pressure and flow of feedback voltage value, about 20 seconds later, type

F4, stop reading and an additional calibration data points.

F5 - with the calibration data points are calculated and document storage parameters.

F6 - keyboard type of pressure, the flow of settings, step by testing on the parameters are correct.

F7 - on / off high-voltage power

F8 - auxiliary cooling fan switch

F9 - cooling fan automatically

F10 - on / off counter (detector) high-voltage power supply.

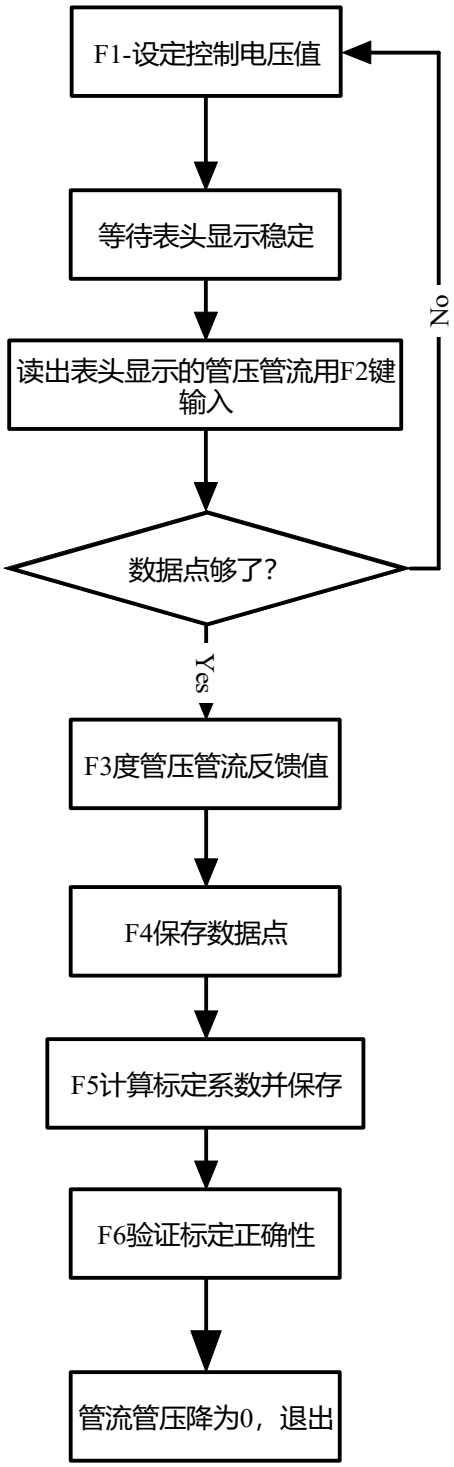
**Steps:**

（1）F8 - auxiliary cooling fan switch to observe the operation of fans is correct, and maintain in a state.

（2）F10 - switching counter (detector) high-voltage power supplies, observe the counting of the Power Plate indicator is correct.

（3）F7 - switch high-voltage power X-ray tube, first determine whether the correct action, and remains in a state.

（4） (first high-voltage power supply to the internal and external buttons for internal control, try to manually adjust the pressure and flow, the flow of care can increase pressure, the stability of the pressure on the screen of the AD-voltage with the value The correct flow of pressure changes, back to zero after the F1 key set by the pressure of the flow control voltage of 0,0, and then internal and external buttons for controlling the field.) Then begin calibration, flow chart is as follows:




**3、C:\XRFT> OBJ\XRF↙Overall tuning, running the main program C: \ XRFT> OBJ \ XRF ↙**

(1) into the process be conducted after the reduction, including the sample covered closed-gate closed, room air samples, the last state to free (Idle). Observation of the two temperatures, the pressure and flow, vacuum, the gas flow pressure on the show is reasonable. Start gas flow

pressure certainly is not setting (110 kPa), after a period of adjustment, will reach settings.

（1）F4 - into the standby (Standby), vacuum pump room open by the vacuum, fluorescent tubes and high-voltage power-up of pressure to increase the flow of security (10 kV, 0.5mA or 20 kV, 1.0mA), observation of cooling Oil temperature changes and changes in the vacuum, should increase the tropical heat.

（2）F2 - into the state-for-like (Change Sample), if necessary-gate will be closed and room air samples, the sample covered open.

（3）F1 - into the readiness of (Hilight), the increased pressure to slow the flow settings. With the other standby.

（4）F3 - into the test readiness of (Measure), closed room by switching valve, close the air valve, open the valve switch sample room, the sample room vacuum 30 seconds, the observation room by gas leakage rate, and open the room by switching Valve, from top to bottom at the same time vacuum, vacuum to be set at the value-open the gate to test readiness of the end.

（5）F6-admission into the spectrum of state (Sampling), the test readiness on the basis of open spin, and began collecting line.

（6）F10-enter the shutdown state (Idle).

All of these can freely switch between the states; only in the Idle state is the safety of the power is switched off.