MN-OES-R420-E

SPARK DIRECT READING SPECTROMETER (STANDARD TYPE)

OES-R420

MANUAL

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1 Overview

OES-R420 series spectromter, the latest generation of spark source optical emission spectrometer, is the combination of spark discharge technology and CCD full-spectrum acquisition technology, together with optics chamber flushing, separate exposure and intelligent curve selection technology.

1.1 Basic principle

Spark source optical emission spectroscopy is a kind of emission spectrum, whose basic principle is to evaporate, atomize and excite the analyte using the energy between the analyte (usually a solid metal or alloy) and the counter electrode generated by the spark source. After the light is split by the grating, the characteristic lines of different elements are obtained. Photoelectric conversion device is used as a detector to collect the characteristic lines of each element, and then qualitative and quantitative analysis is performed according to the wavelength and intensity of the line. Photomultiplier tubes
(PMT) and charge coupled devices (CCD) are currently widely used detectors. Traditional spectrometers use PMT as the detector. Since each PMT can only receive and detect the spectral line of a certain wavelength, and due to the structure and limited space for PMT layout in the optics chamber, the spectrometer can only analyze a finite number of elements simultaneously, the upper limit usually around 30~40. The application of the CCD detector approximately covers the full spectrum in the detectable range, so that the appropriate spectral lines of the analyzed elements can be selected from tens of thousands of optional lines to optimize the stability and accuracy of the instrument.

1.2 Performance characters

The OES-R420 series spectromter spectrometer uses a high-resolution linear array CCD as a detector to achieve full-spectrum coverage. The excitation source, an all-digital solid-state spark source with continuous tunable excitation energy and frequency, can be widely applied

for componential analysis to all elements of various matrix metal samples. Due to the specially developed optical path flushing protection system, performance of the instrument is more stable and its service period is prolonged; Thanks to the massive spectral information, the software can intelligently select the sensitive lines of each element matched with the best reference lines, which reduces the influence of relative intensity fluctuation to the stability of the instrument and reduces the third element interference; A perfect connection is achieved between different spectral lines of the same element, expanding the scope of analysis; New analysis elements and even new material matrixes can be easily added without new hardware at the user site, with a simple maintenance process.

OES-R420 series spectromter has the following features:

1) All-digital solid-state spark source with continuously adjustable excitation energy and frequency, suitable for various materials.
2) Full-spectrum detection of the spectral lines in the available range by using a multi-chip staggered array of specially coated linear CCDs.
3) Single-plate lens holder, greatly reducing the potential contamination of the optics in cleaning the lens.
4) Ethernet data acquisition and control, fast speed and versatility.
5) Programmable optics argon flush control system, much better long-term stability.
6) Aluminum copper spark stand base, improving heat dissipation and firmness.
7) Low argon consumption, no pressure fluctuations, no noise, and shorter cold start time.
8) Good scalability, easily adding new analysis elements and material matrix at user site.

1.3 Hardware

OES-R420 series spectromter spectrometer consists of spark source system, optics diffraction system, acquisition system and a control and data processing system.

1.3.1 Spark Source System

The spark source system is to provide energy for the evaporation, atomization, and excitation

of the analytical sample to produce characteristic lines. It mainly includes an excitation stand and corresponding circuit systems for power supply and signal control to the excitation stand.

1.3.2 Optics diffraction system

The optics diffraction system is to disperse the complex light beam generated by the spark source into monochromatic light, which is mainly composed of a grating, an optical path collimating lens, an entrance slit, etc. The composite light emitted by the spark source is irradiated onto the grating through the entrance slit through the collimating lens, splitting lights based on the principle of different diffraction angles for different spectral wavelengths.

1.3.3 Acquisition system

The acquisition system is to convert the collected optical signal into electrical signal by using photoelectric devices, and perform signal amplification processing. The acquisition system consists of detectors and corresponding control circuits. As mentioned above, the commonly used detectors are photomultiplier tubes (PMT) and charge coupled devices (CCD). The PMT is a photoelectric conversion device manufactured based on a secondary electron multiplication phenomenon, and is composed of a photocathode coated with a photosensitive material on its surface, multiple dynodes having a surface coated with an electron escaping functional material, and an anode. When incident light illuminates the photocathode, electrons are released, the electrons are multiplied stepwise between the plurality of dynodes until the electrons collect at the anode of the tube. CCD is a kind of semiconductor optoelectronic device that utilizes the internal photoelectric effect. When radiant energy acts on the photosensitive material in the device, the generated electrons usually do not leave the material, but produces a current relying on the free-moving photoconductivity of electron-hole pair generated after absorbing photons in the material (the resistance of the semiconductor decreases and the conductance increases after absorption of photons), converting the optical signal into electrical signal.

1.3.4 Control and data processing system

The control and data processing system are mainly composed of spectrum software and corresponding control circuits. Its main function is to execute the commands issued by the computer such as flushing, excitation, stop, data acquisition, etc., and the collected intensity data are calculated and converted into chemical content of various elements in the sample.

1.4. Auxiliary devices

1.4.1 Stabilized voltage supply

Stabilized voltage supply is often used for voltage regulation, because unstable input voltage will accelerate aging of the equipment, affect the service period or even burn out circuits. In serious cases, there may even be safety accidents, causing massive losses.

Currently commonly used stabilizers include fully automatic compensation regulators, AC purification regulators, and parametric regulators. In the actual selection, the power supply should be reasonably selected according to the specific conditions of the rated power, power factor and load type of the electrical equipment. There should be a proper margin for the output power especially for a heavy impact load. It is suggested to choose a voltage regulator with a total load of 1.5 to 3 times of the rated power of the spectrometer, but not too large to avoid wasting energy.

OES-R420 series spectromter is powered by a 220V single-phase AC power supply with a maximum power of 1.2kW. According to the principle above, an AC parametric voltage regulator is recommended with a power of 3kVA.

1.4.2 Sample preparation equipment

The sample needs to be processed before the analysis, in order to remove the oxide layer and inclusions on the sample surface, so that the analysis results can represent the true level of the analyzed sample. Different processing equipment will be used according to different sample materials: for the harder iron and steel samples, cast iron, nickel alloy and cobalt alloy

samples, the grinding machine (including two kinds: double wheel grinding machine and sandpaper grinding machine) is generally used; for the soft metals and alloys such as aluminum, copper, zinc and lead, the samples are generally processed on a lathe. For larger and irregular specimens, the specimen should be cut into a regular shape and size suitable for analysis then processed with grinding machine or lathe.

1.4.3 Argon supply facility

The sample should be excited under the argon atmosphere and the optics chamber need to be flushed with argon to reduce the interference of nitrogen gas to nitrogen detection and eliminate the absorption of spectral lines in the far ultraviolet region such as C, P, S, B, etc. by oxygen, water vapor, etc. It is recommended to use high purity argon with a purity greater than 99.999%.

An argon gas purifier can be used if the purity of argon gas is not satisfactory. The argon purifier works by passing the gas through a vessel containing titanium particles heated to 700 degrees, chemically removing oxygen and nitrogen, then removing hydrogen, hydrocarbons, carbon dioxide and water through a pipe containing copper oxide. The remaining carbon dioxide and water are removed as the gas passes through the molecular sieve.

If the amount of sample needed to analyze is large and the argon consumption is fast, a liquid argon tank can be considered for gas supply.

1.5 Scope of application

OES-R420 series spectromter can be widely used in the production process control of metallurgy, foundry, machinery, steel and non-ferrous metal industries, raw material, parts and product analysis in the fields of automobile manufacturing, aerospace, marine, mechanical and electrical equipment, engineering machinery, electronics and electrical, education, scientific research, etc. It is also widely used in the process research and product development of large metallurgical enterprises.

1.6 Storage and usage conditions

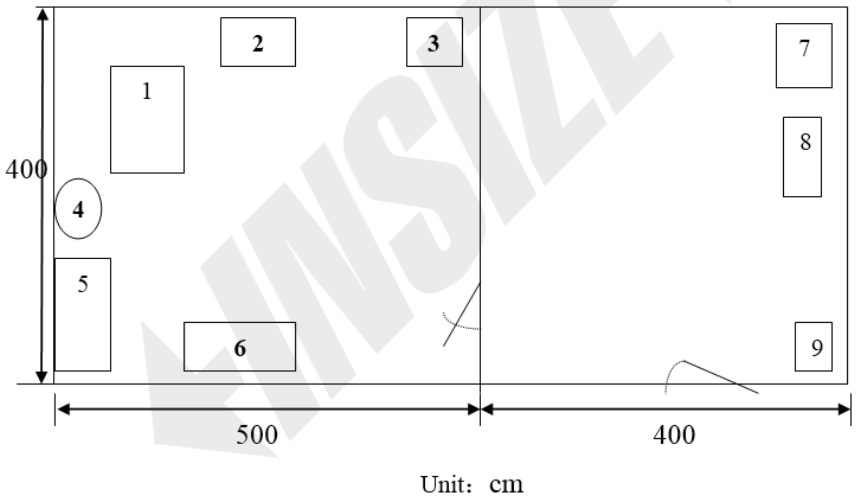
The working environment of OES-R420 series spectromter requires temperature (10~30) °C and relative humidity between (20~80) %. Vibration should be avoided as much as possible in the laboratory, and if necessary, use anti-vibration frame, rubber, etc. to reduce vibration impact. Storage temperature for the instrument is required to be (0-45) °C. The instrument should be installed or stored in a room with little dust and no corrosive gases.

2 Installation

It is the basic requirement for ensuring the normal operation of the instrument to install it according to certain specifications and sequences in a laboratory with suitable conditions. **It is strongly recommended that the instrument be installed by extensive experienced after service engineer.** If customers want to install it by themselves, it is only possible for the installation to be carried out by those who are **familiar with electrical equipment and computer software and hardware** and have **read through and completely understand the current part of this manual**. If anything abnormal occurs during installation, please contact Customer Service Department.

2.1 Preparation before installation

2.1.1 Lab conditions



1.spectrometer and bench 2. computer desk 3. stabilized argon supply 4. argon cylinder
5. file cabinet 6. work table 7. sandpaper grinding machine
8. floor-type double disk grinding machine 9. gap water and tank

Fig.1 Sketch map of lab layout

The spectrometer should be installed in a dedicated room, with a certain use area (about 15-25 square meters). There should be no flammable, explosive, toxic, harmful substances and no corrosive gases such as acid and alkali. The working temperature of the instrument is (10~30) °C, and the lab temperature fluctuation should be less than 2 °C per hour. Avoid installing the instrument in direct sunlight. Air conditioning is required in the laboratory. The relative humidity should be between 20% and 80%. A dehumidifier is necessary if the humidity

is too high. A thermohygrometer is needed to monitor temperature and humidity changes in the laboratory.

Fig.1 is the sketch map of lab layout. The laboratory should be clean with little dust, especially metal dust, to avoid polluting the optics, affecting the heat dissipation of the instrument or causing circuit system failure. Meanwhile, keep away from electromagnetic radiation sources (such as medium frequency electric furnaces, transformers, etc.) and vibration sources (such as forging workshops, railway tracks, etc.) to reduce their affection to the optical path and circuits, and ensure accuracy and service period of the instrument.

2.1.2 Power supply

The instrument is powered by (220±22) V, 50/60 Hz single-phase power supply. It is recommended to equip a magnetic saturation stabilized voltage supply of 3kVA, with dedicated and independent grounding wire. The grounding resistance should be less than 4Ω and the voltage between the ground wire and the neutral line should be 0 or less than 3 volts. For grounding method, refer to Fig.2 or Fig.3.

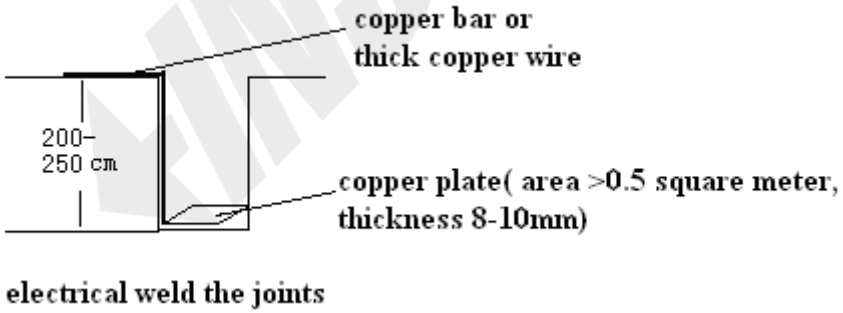


Fig.2 Grounding method（1）

An AC contactor with rated voltage of 220VAC and rated current of no less than 20A together with corresponding start and stop control switches are needed if power failure is frequent in the laboratory, in order to avoid the affection of the grid impact on the instrument when the power system is restored.

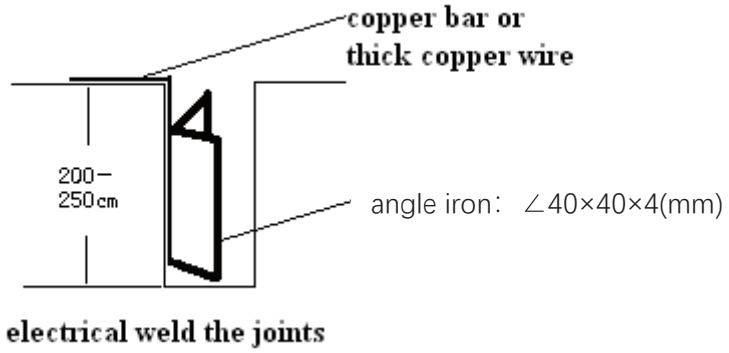


Fig.3 Grounding method（2）

2.1.3 Workbench and computer, printer

The size of OES-R420 series spectromter is: 84cm × 47cm × 44cm (width × depth × height), net weight: about 100kg. A workbench is needed with loan-bearing no less than 300kg and height between 55 cm and 65 cm.

Prepare a PC with a current configuration (with no less than 3.3GHz, 2GB RAM, 500GB hard drive) for installing analysis software and an inkjet or laser printer to print sample test reports.

2.1.4 Argon supply

At least one cylinder of high purity argon gas is needed, whose purity should be higher than 99.999%. The inlet pressure of the instrument (ie, the outlet pressure of the pressure reducing valve) is 0.5 MPa. The working flow rate is between 8 to 10 L/min in analysis and the maintained flowrate is between 70 to 80 mL/min in standby mode. An argon purifier can be equipped if necessary.

2.1.5 Sampling and sample preparation device

Casting sample can be casted into a cylinder or a cone with a diameter of 30 to 40 mm and a height of about 60 mm. The sample is processed after cutting 1/3 of the bottom. For the analysis of large samples or final products (non-cast iron), it is necessary to cut out a block sample with a diameter of bigger than 16mm and a height of more than 20mm for

successive processing.

Different sample processing equipment is recommended for different materials: A rotary grinder is suitable for hard materials such as steel and superalloys and lathe is recommended for soft materials such as copper, aluminum, lead, and zinc.

Fig.4 Rotating disk type grinding machine

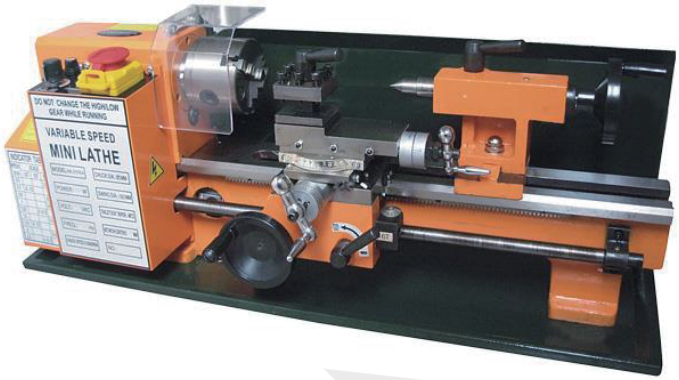


Fig.5 Mini lathe

Cast iron samples can be analyzed by OES only after sampling with a special mold and after the “whiten” process (transformed to white iron). The sampling mold for cast iron can be made of steel or red copper, with its dimensions shown in Fig.6.

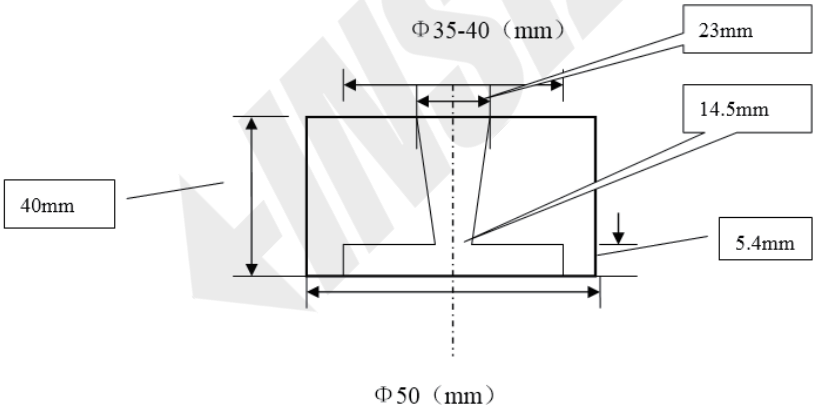


Fig.6 Sampling mold for cast iron

2.1.6 Reference materials

It is necessary to prepare standard samples or self-made control samples that are consistent with the user's product type (consistent with the metallurgical process of the product, having good uniformity and accurate value) for correction of instrument accuracy.

2.1.7 Spectrometer maintenance material and equipment

One bottle of absolute ethyl alcohol with a volume of 500mL is needed to clean the lens and a vacuum dust cleaner with rated power of 1200W is necessary for instrument maintenance.

2.2 Hardware connection

Confirm whether the outer packaging is intact when the instrument arrives at the customer's site. **Do not sign and immediately contact Customer Service Department if the package is found to be damaged or tilted upside down.** If the user installs it himself, please read this chapter carefully and strictly follow the rules.

Don’t open the outer packaging before service engineer arrives to avoid loss of spare parts.

Check and confirm the packing list after opening the packaging boxes of the spectrometer and related accessories. Then perform the following steps.

2.2.1 Hardware inspection

After placing the spectrometer on the workbench, open the upper cover and the front and rear panels of the instrument to check whether the PCBs, circuit connectors and mechanical parts are properly connected.

2.2.2 Argon path connection

Fix the pressure reducing valve on the argon gas cylinder, and connect the two ends of the argon gas pipe respectively to the pressure reducing valve and the argon gas inlet port of the instrument, and then fasten the joints with a wrench.

2.2.3 Electrics connection

Connect the air switch and AC contactor (including start and stop control switch buttons), then connect the output terminal of the AC contactor with the input terminal of the stabilized voltage supply. **Be careful not to reverse the phase line and the neutral line**. These steps should be done by professional electrician and the following steps be performed by engineer: connect the dedicated independent ground wire to the grounding terminal of the stabilized voltage supply and connect it to the ground terminal of the spectrometer. Connect the output end of the stabilizer to the dedicated socket, **pay attention to the one-to-one correspondence between the zero line, the phase line and the ground line at both ends.** Finally, plug the power cable of spectrometer, computer, monitor, and printer into the dedicated socket.

Insert the data cable (network cable) into the computer network card slot and set the computer IP address as 192.168.1.18 and the subnet mask as 255.255.255.0.

2.2.4 Spectrometer power on

Turn on the air switch, AC contactor, stabilized voltage supply and dedicated socket switch in sequence, then press the “ON” button at the back of the spectrometer to power on the spectrometer. The instrument heating system starts automatically. After about 4 hours, a stable standby state can be achieved.

Unrelated personnel should keep away from the laboratory when the instrument is powered on for the first time.

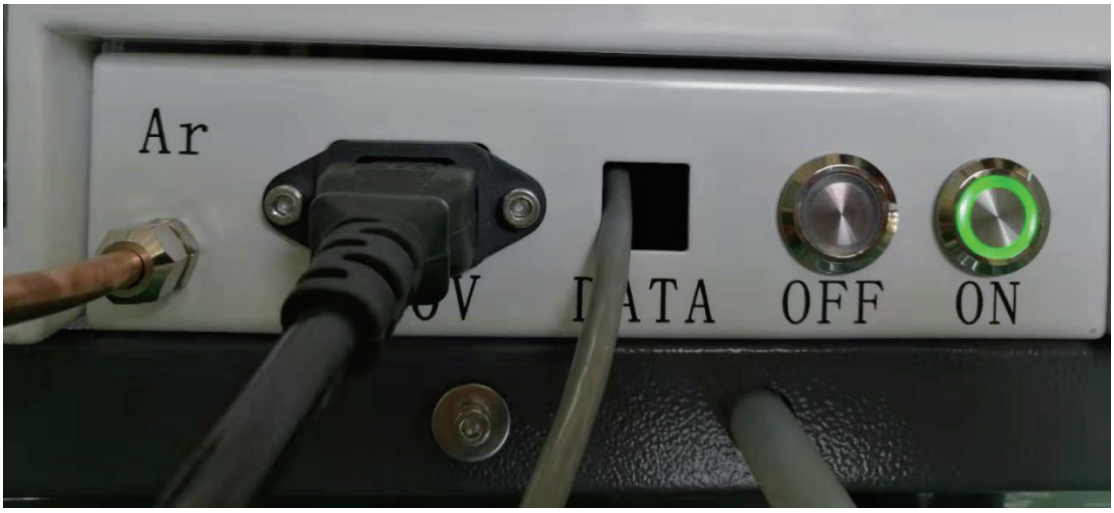


Fig.7 Back of instrument（left to right: argon inlet, main power, data cable, power off, power on）

2.3 Software installation

The software for OES-R420 series spectromter spectrometer software is a free installation version. Open the analysis software by clicking */OES-R420/CCD.exe* after copying the software package to the computer hard disk. Register the software according to the software registration code provided by the service engineer.

2.4 Instrument inspection

After the spectrometer is powered on, turn on the master valve of the argon cylinder, then turn on the computer and start the spectrum analysis software. Place a sample on the spark stand, then click the *Flush* item in the *Test* menu to set the pressure of the pressure reducing valve to 0.5 MPa. Open the rear panel of the instrument, and adjust the flowrate of the flow meter to (8~10) L/min. The flow meter on the right side is (70~80) mL/min. If there is a leakage at the pipe joint, check with foamed water and fasten the joint. Turn on the emergency button at the front panel of the instrument and click the *Spark* button in the *Test* menu to make sure the instrument is working normally and check whether the spark spot is good.

Fig.9 Pressure and flowrate checking

3 Software introductions

3.1 Main interface

Enter the main interface by opening*/OES-R420/CCD.exe*. The main interface consists of a menu bar, a top toolbar, a sample selection field, a data display area, a bottom toolbar, and an information bar. The sample selection field is used to select or input a sample name. The information bar is used to display the sample excitation progress, optics chamber flushing progress, functional status, working curve, spark times, product type, operator, etc.

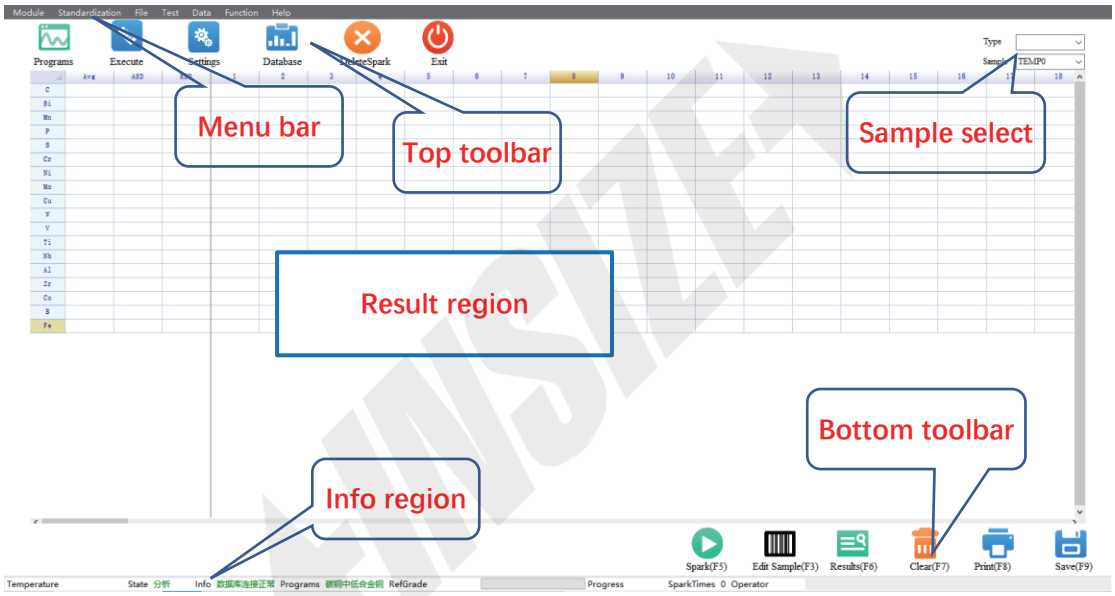


Fig.10 Software main interface

3.2 Menu

The menu bar is below the name of the software in the main interface. There are six menus, including *Module*, *Test*, *File*, *Data*, *Function* and *Help*.

Fig.11 Menu bar

3.2.1 Module

There are three items in the menu of *Module*, i.e., *Calibration*, *Standardization* and *Analysis*. *Calibration* is used to create or edit a work curve for quantitative analysis of metal materials. The work curves of the instrument were built-in before leaving the factory, and can be used directly by the user. Generally, this function is not needed by the user. *Standardization* is used for correction of the instrument, including three types of standardization, i.e., peak standardization, global standardization and type standardization, respectively used for the correction of pixel position, intensity drift and content error. *Analysis* item is used for analyzing customer samples.

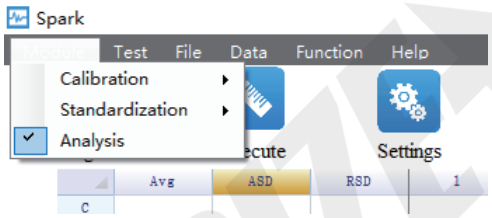


Fig.12 *Module* menu

3.2.2 Test

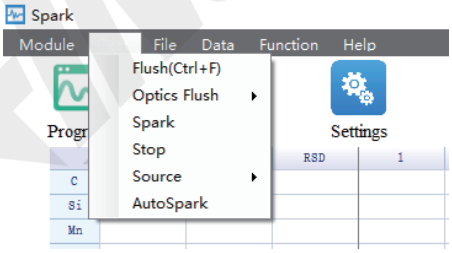


Fig.13 *Test* menu

The *Test* menu is mainly used for hardware checking and maintenance, among which there are items such as *Flush, Optics flush*, *Spark, Stop* and *Source*. *Flush* is used for gas path inspection. *Optics flush* is used to recover the environment inside the optics chamber when the instrument is not used for a long time. *Spark* is used to check whether the excitation function of the instrument is normal, *Stop* is used to end the flush or excitation process. *Source* is a switch used to turn on and off the spark source system.

3.2.3 File

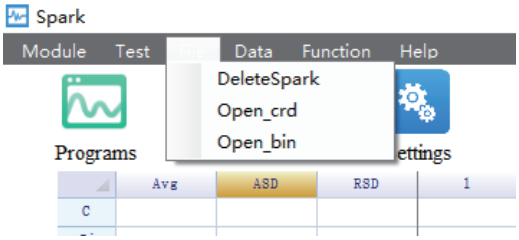


Fig.14 *File* menu

The *File* menu includes three menu items: *DeleteSpark*, *Open_ crd* and *Open_ bin*. *Delete Spark* is used to delete outlier of analysis result, *Open_ crd* is used to open previously saved .crd file, and *Open_ bin* is used to open previously saved .bin file.

3.2.4 Data

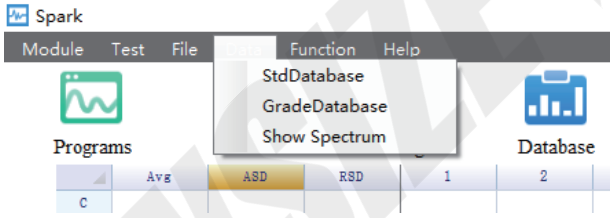


Fig.15 *Data* menu

There are items such as *StdDatabase*, *GradeDatabase*, and *Show Spectrum* in the *Data* menu. *Std Database* is used to store and query content information of standard samples. *GradeDatabase* is used to store and query the content range (the upper and lower limits) of different grades. *Show Spectrum* is used to view the stored spectrum.

3.2.5 Function

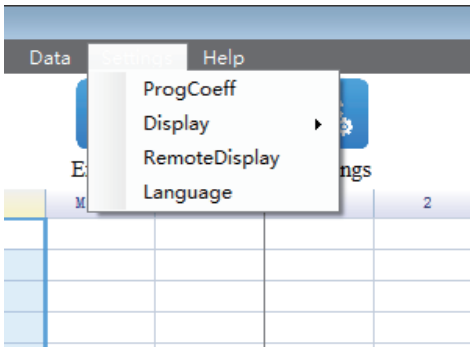


Fig.16

Function menu

Items such as *ProgCoeff*, *Display*, *RemoteDisplay* and *Language* are available in the *Function* menu. *ProgCoeff* is used to view the correction coefficients after global standardization and type standardization. *Display* is used to set the display mode of the result. There are three optional display modes: intensity, intensity ratio and concentration (intensity and intensity ratio are intermediate data, and **concentration is the final result**). The result can be shown in different integration number by changing the *Integral* item. *Remote Display* is used to transfer the analysis result to a remote display. The language of the software can be changed with the *Language* item. Currently there are four options: Chinese, English, Russian and German. Click *OK* after changing the language option and restart the software to apply the new setting.

The *Concentration* item must be selected in the *Display* setting when the sample analysis is finished, otherwise the reported number will be incorrect.

3.2.6 Help

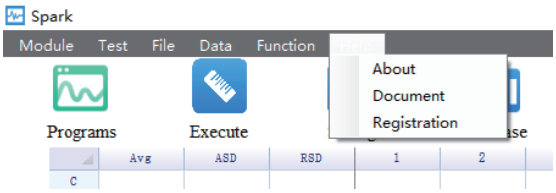


Fig.17 *Help* menu

The *Help* menu has items such as *About*, *Document* and *Registration*, which are used to view the software version and operation instruction file, and perform software registration.

3.3 Toolbar

The toolbar is divided into two parts: the top toolbar and the bottom toolbar. The top toolbar has six buttons: *Programs, Execute*, *Settings, Database*, *Help* and *Exit*. In the bottom toolbar, there are six buttons: *Spark, EditSample, Results, Clear, Print* and *Save*. The following is a brief introduction to the function of each button.

Fig.18 Top toolbar

Fig.19 Bottom toolbar

3.3.1 Programs

The button is used to select the current working program. Click the *Programs* button, double-click the program name in the program list of the *Programs* window to confirm selection, and the selected program is displayed in the info region.

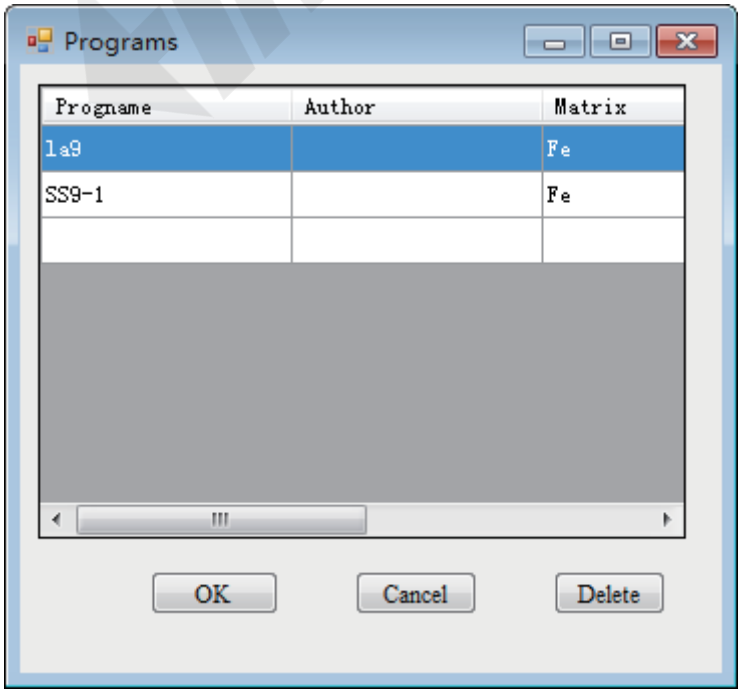


Fig.20 *Programs* window

Fig.21 Info region

For global standardization, type standardization, and sample analysis, the type of sample to be analyzed must match the selected analysis program, otherwise incorrect results will be obtained.

3.3.2 Execute

It is used to perform standardization calculations and is the final step of the peak standardization, global standardization, and type standardization procedures. Please refer to 4.3, 4.4 and 4.5 for details.

3.3.3 Settings

The button is used to set the instrument analysis conditions, spectral line table, display order and decimals of each element, print format, etc. Generally, it is set by service engineer according to user requirements during installation, without being set by the user.

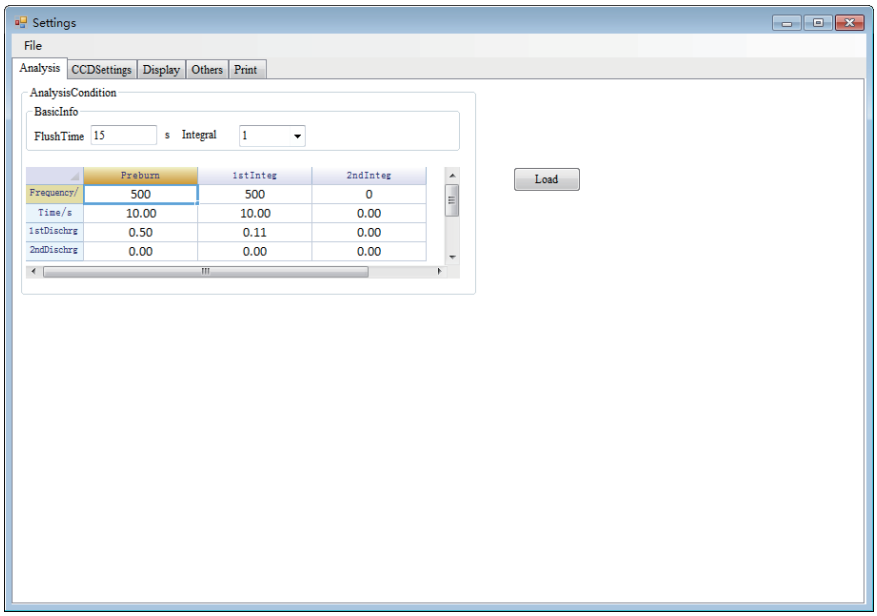


Fig.22 *Setting* window

KEY parameters in the *Analysis*, *CCD Settings* and *Others* tabs, DONOT modify them.

3.3.4 Database

Database The button is used to query and store the content information of standard samples and the upper and lower content limits of different brands. It has the same function with the two items of *StdDatabase*, *GradeDatabase* in the *Data* menu.

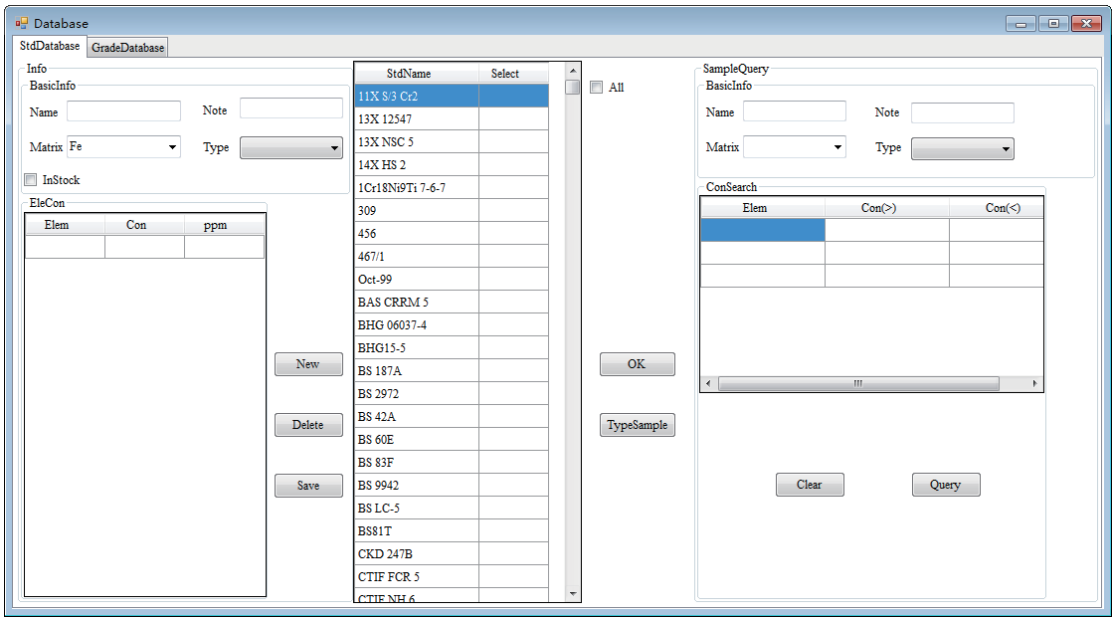


Fig.23 *Database* window

3.3.5 Help

This button is used to view the currently used software version and related help documentation.

3.3.6 Exit

It is used to exit the spectrum analysis software. When exiting the software, the spark source and maintaining argon flow are turned off at the same time.

3.3.7 Spark

Click *Spark* button to start the excitation process under peak standardization, global standardization, type standardization and analysis state after placing a sample on the spark stand properly. When it reaches the set time (usually 20~40s, set in the *Settings* button - *Analysis* tab), the excitation ends automatically.

3.3.8 EditSample

EditSample This button is used to input the sample name, operator and select grades of the sample.

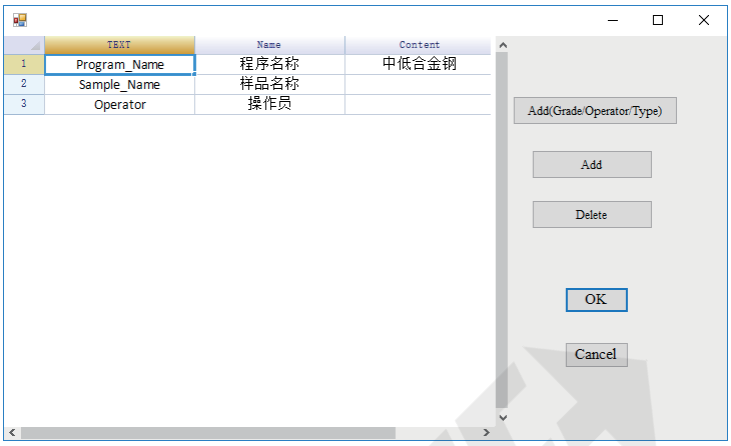


Fig.24 *EditSample* window

3.3.9 Results

Results It is used to view and query previously saved sample results. Click to enter the *Results* window, open the data by directly double-clicking on any row in the results list. The result can also be queried with the operator, sample name or test time as keywords.

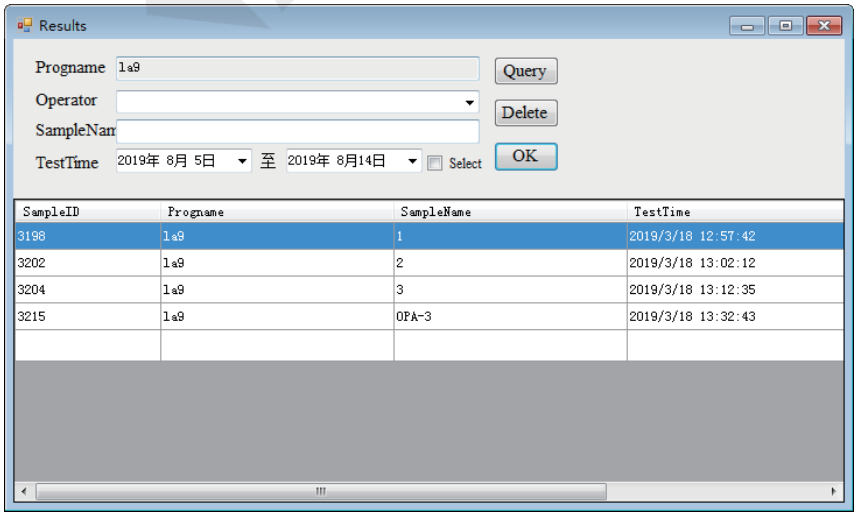


Fig.25 *Results* window

3.3.10 Clear

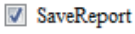
This button is used to delete all data in the result region in the current interface.

3.3.11 Print

It is used to print all the data in the result region in the current interface. The printed items and format are set in the *Print* tab under the *Settings* menu.

3.3.12 Save

This button is used to save the sample analysis results in the analysis interface. If the



checkbox is also selected in the *Other* tab under the *Settings* tool button, the data can also be saved as a test report in Excel spreadsheet besides being saved in the results list of the software. You can query the stored data in the software using the *Results* button introduced in 3.3.9. Test reports can be copied to a removable disk or other computer for data format editing.

4 Common use

Fig.26 is the flow chart of OES-R420 series spectromter for daily use, the detailed operation process is discussed in six sections in this part.

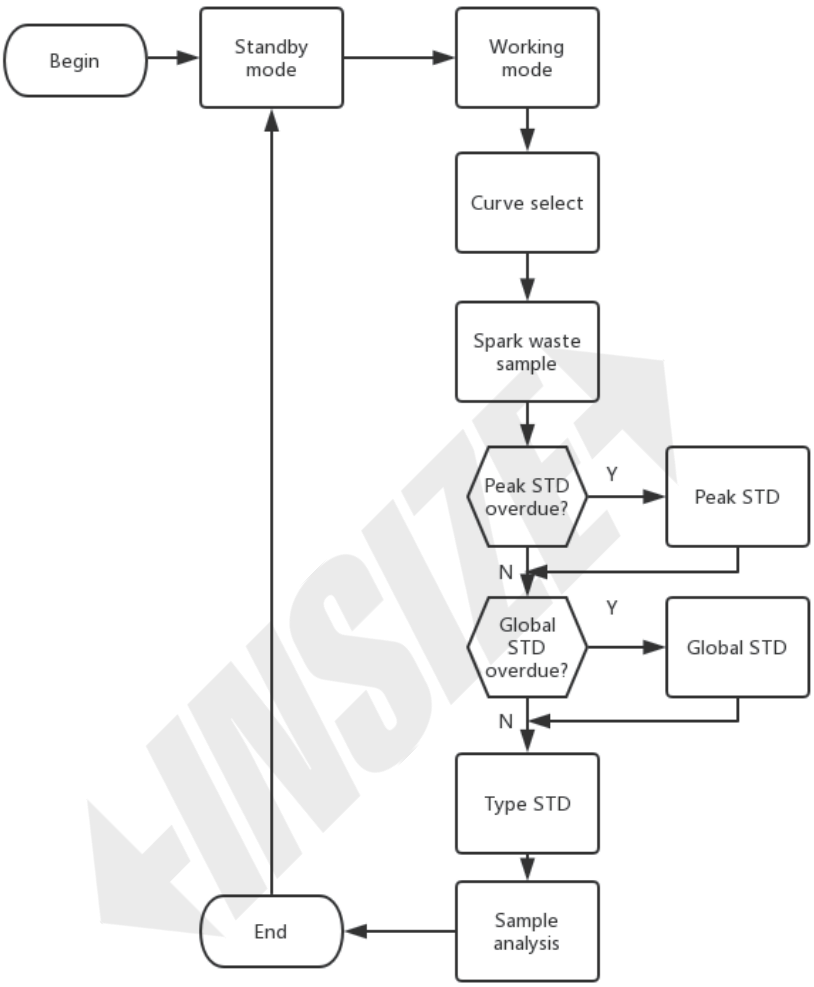


Fig.26 Flowchart of OES-R420 series spectromter for daily use

4.1 Turn on and off

Keeping the spectrometer in standby state all the time makes the instrument stable and ready to work, which helps to extend the life of the instrument. It is recommended to **turn the instrument to standby state every day after work**, and **turn it to work state when it is used again next time**. The instrument can be **completely powered down only if it is not used for a long time (such as in a holiday for more than three days), or in the case of**

expected power failure or during a thunderstorm.

Working state refers to the state in which the instrument can be used immediately. At this point, the instrument is powered on, the emergency stop button is switched on, the spectrum analysis software is open, the main valve of the argon cylinder is opened and the pressure reducing valve displays 0.5 MPa, the flow meter on the right side (maintaining flow) is (70~80) mL/min, and the flow meter on the left (analytical flow rate) displays (8~10)L/min when it is sparking or flushing state or displays 0 when there is no spark or flush.

When it is in standby state, the inner parts of the instrument are in an automatic constant temperature state. At this point, the instrument is powered on, both the computer and the main valve of the argon cylinder are shut down (no argon being consumed).

Completely shut down state means all the facilities in the laboratory are closed or powered off, including the instrument, power socket, stabilized voltage supply, AC contactor, air switch, air conditioner and dehumidifier.

4.1.1 Work to standby

The spectrometer should be turned from work state to standby state after the instrument is used and before the personnel leaves the laboratory. The specific steps are:

(1) Turn off the emergency button at the lower part of the front panel of the instrument (press, no light for off);

(2) Exit the spectrum analysis software (there should be a sound of the solenoid valve and relay in the instrument at this time, also the cooling fan for the spark source stops running), then turn off the computer and monitor;

(3) Close the main valve of the argon cylinder.

After the three steps above, the instrument is in standby mode, there is no need to turn off other switches.

4.1.2 Standby to work

The instrument should be turned to work state from standby state before being used for analyzing. The process is:

（1）Open the main valve of the argon cylinder;

（2）Turn on the computer and monitor, then open the spectrum analysis software (there should be a sound of the solenoid valve and relay in the instrument at this time, also the cooling fan for the spark source begins to work);

（3）Turn on the emergency button (rotate clockwise, red light for on);

（4）Restore the inner environment of the optics chamber by clicking *Optics Flush* in *Test* menu: It is recommended to use long flush when the instrument is out of service for a longer period of time (more than 5 days) and use short flush for short time of out of service (12 hours to 5 days). Check whether the pressure reducing valve and flowmeter number are within the required range during the flushing process;

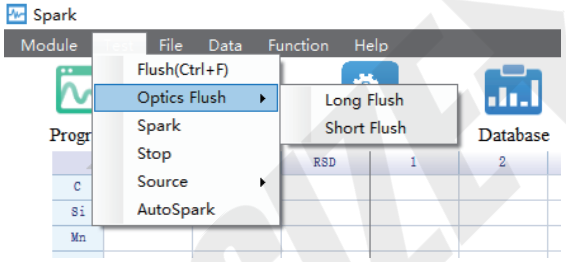
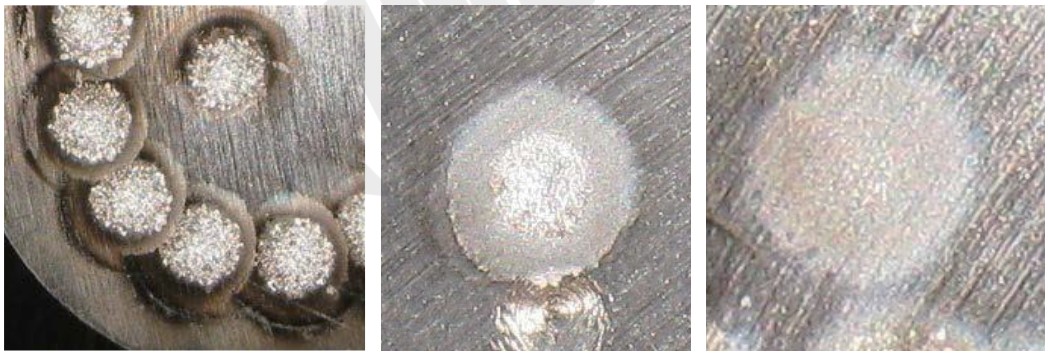


Fig.27 Optics flush

（5）Spark waste sample for 3 to 5 times to confirm the spark spot is normal.



Good　　Acceptable　　Bad

Fig.28 Comparison of different spark spots

Type standardization and sample analysis can be performed after completing the above five steps. Do peak standardization and global standardization first if necessary.

4.1.3 Work to power off

It is recommended to power off the instrument when it is not used for a long time (in holidays

for more than three days) or in the case of expected power failure or in thunderstorm. The process to power off the instrument from work state is:

(1) Turn off the emergency button at the lower part of the front panel of the instrument (press, no light for off);

(2) Exit the spectrum analysis software (there should be a sound of the solenoid valve and relay in the instrument at this time, also the cooling fan for the spark source stops running) and turn off the computer and monitor;

(3) Close the main valve of the argon cylinder;

(4) Turn off the main power supply of the spectrometer (press the “OFF” button at the bottom of the instrument's rear panel, the red indicator of this button is on);

(5) Turn off the dedicated socket switch, stabilized voltage supply, AC contactor and air switch in sequence. If necessary, take off the plugs of the spectrometer, computer, monitor and printer power cable on the socket;

（6）Turn off the air conditioning.

4.1.4 Power off to standby

The spectrometer needs to be turned to standby state before reusing after a period of power off. The process for this is:

(1) Turn on the air conditioner and set the temperature to 20~25°C. Turn on the dehumidifier at the same time if the indoor humidity is too high;

(2) After the indoor temperature and humidity becomes stable, turn on the air switch, AC contactor, regulated power supply, and dedicated socket in sequence;

(3) Turn on the main power supply of the spectrometer (press the “ON” button at the bottom of the instrument's rear panel, the green indicator of this button is on).

The instrument needs a period of time for temperature constant system of the optics chamber after the spectrometer is powered on. About 4 hours later, the instrument can be turned to work state according to the process in 4.1.2.

4.2 Sample preparation

The standard used for instrument standardization and actual samples should be freshly prepared before analysis to ensure a smooth surface, clear texture and uniform orientation, and no defects on the prepared surface such as fingerprints, water or oil stains, pores, inclusions, trachoma, cracks, etc. Please refer to 2.1.5 for the method and facilities of sample preparation.

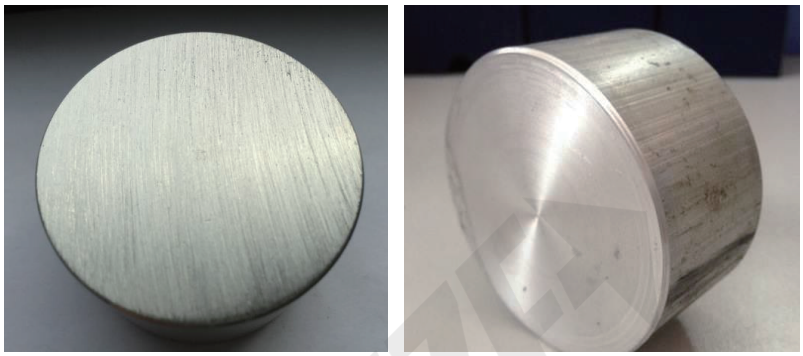


Fig.29 Well prepared sample

4.3 Peak standardization

After the instrument is used for a long time, the peak position of the element's spectral line may shift as the ambient temperature, humidity, vibration and other conditions change. In this case, the peak standardization is needed to correct the spectral line position shift. The specific steps are as follows:

(1) Click *Standardization* in *Module* menu and select *Peak*, there shows the *Peak Standardization* window;

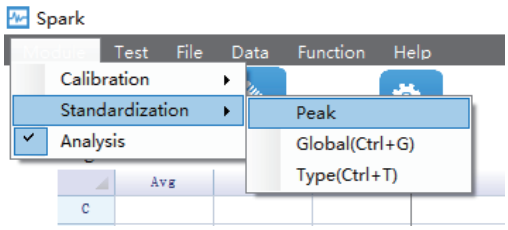


Fig.30 Peak standardization

	SetPixel	SetIntensity	LastIntensit	Drift	CurrentInten	1
CCD-0	2563	1218048	1220736	0	1459712	
CCD-1	1535	1948672	1961856	0	2312320	
CCD-2	917	5595520	5609088	0	6273408	
CCD-3	3486	4989696	5022592	0	5553280	
CCD-4	1970	4957056	4972160	0	5458432	
CCD-5	1635	3540480	3601536	0	3890432	
CCD-6	2981	2101632	2190336	0	2205184	
CCD-7	1318	3062016	3123968	0	3127680	
CCD-8	2356	4948480	5056512	0	5167616	
CCD-9	926	738816	745216	0	726272	
CCD-10	2989	61696	67328	0	92800	
CCD-11	1000	0	0	0	0	

Fig.31 *Peak standardization* window

（2）Place the prepared profile sample on the spark stand after cleaning the electrode with electrode brush;

（3）Click *Spark* button;

（4）Finish peak standardization process by clicking *Execute* button at the top tool bar after sparking.

Perform peak standardization only after confirming the spark spot is normal. Doing peak standardization with abnormal spark spot will lead to error in analytical result.

Perform peak standardization only with the specialized profile sample packed with the spectrometer, it will lead to wrong analytical result if using other standard or sample.

The recommended period for peak standardization is once per month, it is adjustable according to the laboratory environment and instrument stability.

After peak standardization, you can click *Settings* button

then click *Others* tab to check *Drift* column of the *Pixel Correction*.

The normal range of the peak drift should be between -5 and 5.

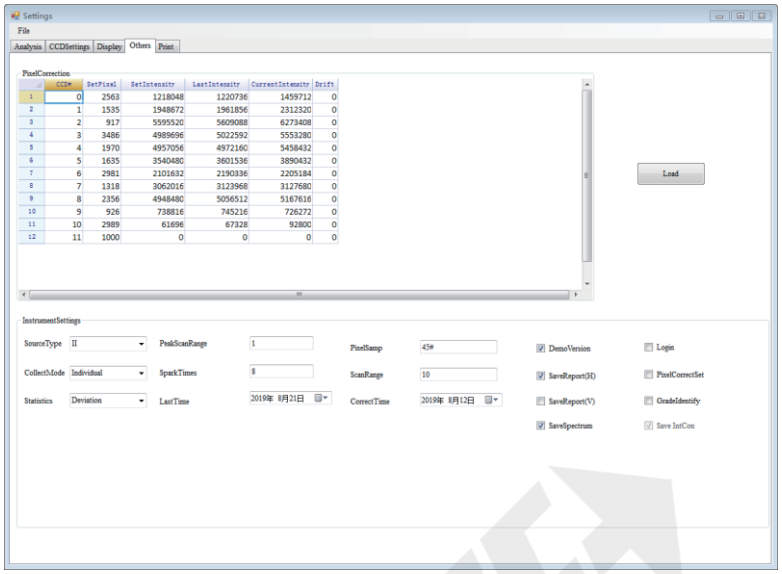


Fig.32 Pixel correction

4.4 Global Standardization

Working curve is the basis of the spectrometer for quantitative analysis. The working curve is usually drawn in factory by the commissioning engineer. The relationship is established between the spectral line intensity and content of each element when the work curve is drawn. However, the intensity of each element is not static, it can be affected by vibration, temperature fluctuation of the optics, and contamination of the optical components. In this case, global standardization is needed to correct the instrument intensity change. The specific steps are as follows:

(1) Click *Standardization* in *Module* menu and select *Global* function;

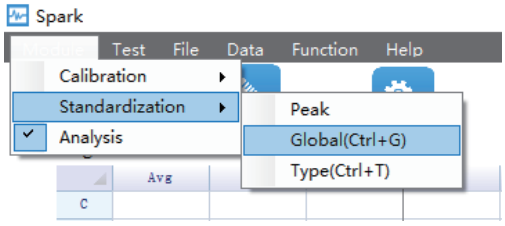


Fig.33 Global standardization

(2) Select the curve for correction and the mode of global Standardization (*IntExpectCoeffCalculation* or *GlobalCoeff*, *GlobalCoeff* for general use). Click *OKSave* after

selection;

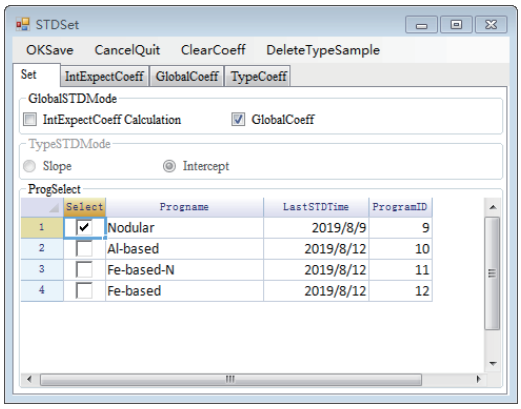


Fig.34 *STDSet* window (global)

（3）Select the standard sample in the sample select region of the main interface and place the respective standard sample on the spark stand, then click *Spark* button;

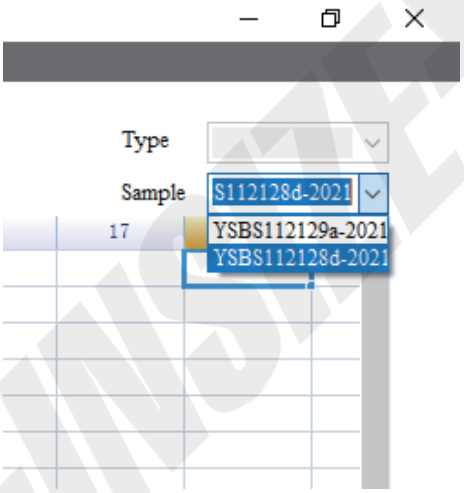


Fig.35 Sample select

（4）After sparking, clean the electrode with electrode brush and change the sample position, then spark for another time. Each standard sample should be sparked for at least two times, removing outliers;

Fig.36 Brushing electrode

（5）Select another standard sample in sample select region and spark it according to step (3) and (4), repeat this step until the standard samples are all sparked. Then go to next step;

（6）Finish global standardization by clicking *Execute* button in the main interface.

Perform global standardization only after confirming the spark spot is normal. Doing global standardization with abnormal spark spot will lead to error in analytical result.

The recommended period for global standardization is once per week, it is adjustable according to the laboratory enviroment and instrument stability.

4.5 Type standardization

4.5.1 Principle and operation

In theory, customer samples can be accurately analyzed after the process of peak

standardization and global standardization. However, this is not always the case. There is still a certain error in the analytical results of customer samples because of a common difference in metallurgical process, internal structure, and physical state between the standard used for the working curve and the customer samples. It is necessary to perform type standardization to eliminate the above error using a standard sample or a user-made control sample having the same metallurgical process, internal structure, physical state and chemical composition with the customer sample.

The process for type standardization is as follows:

(1) Click *Standardization* in *Module* menu and select *Type* function;

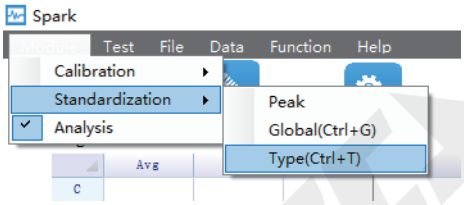


Fig.37 Type standardization

（2） Select the type standard to use and the mode of type standardization(*Slope* or *Intercept* or *Slope+Intercept*, Intercept for general use), then click *OKSave* button;

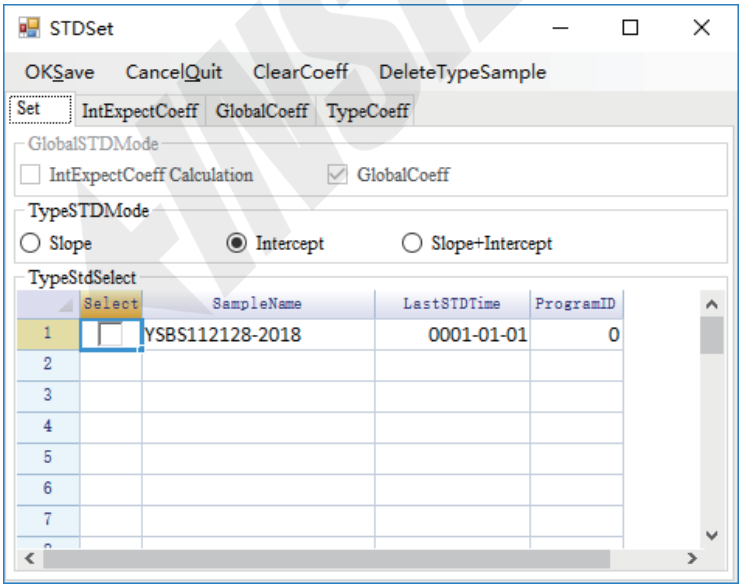


Fig.38 *STDSet* window (type)

(3) Place the selected type standard(prepared) on the spark stand after cleaning the electrode with electrode brush. Spark for at least two times and delete outliers;

(4) Finish type standardization by clicking *Execute* button in the main interface if the results are acceptable.

Perform Type standardization only after confirming the spark spot is normal. Doing type standardization with abnormal spark spot will lead to error in analytical result.

The recommended period for type standardization is once per shift (every 8 to 12 hours), it is adjustable according to the laboratory environment and instrument stability. Redo type standardization if the type of the sample to analyze was changed.

4.5.2 Add type sample

If the type standard to use can't be found in *STDSet* window(type), it is necessary to add the standard to the type standard select list of this window before it can be selected. The specific steps for this are:

(1) Open the *Database* window by clicking the *Database* button in the toolbar or clicking the *StdDatabase* item in the *Data* menu;

(2) Under the *StdDatabase* tab, select the material matrix corresponding to the curve to be added;

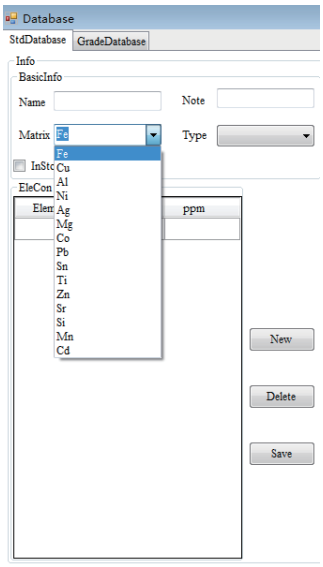


Fig.39 Matrix selecting

(3) Find and click the name of the standard to be added in the central standard list, confirm the element name and content of the standard according to certificate or test report of the standard;

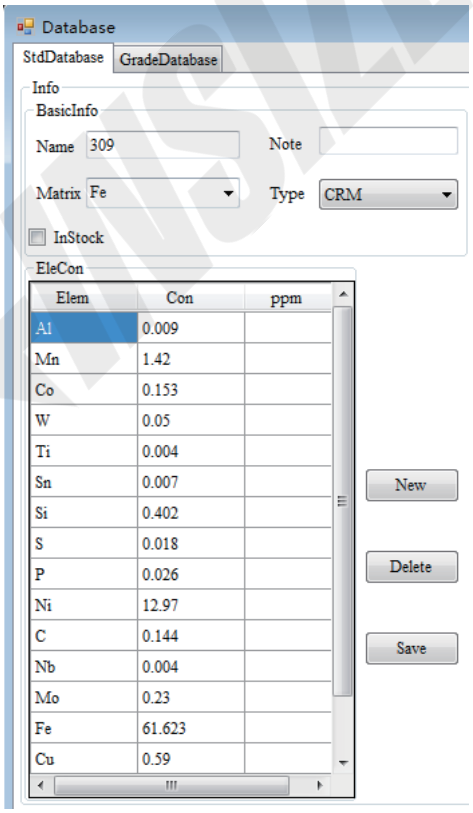


Fig.40 Content list of standard samples

(4) Click the *Select* bracket on the right of the standard name and then click *TypeSample* button to add the standard to the type standard selecting list in Fig.38.

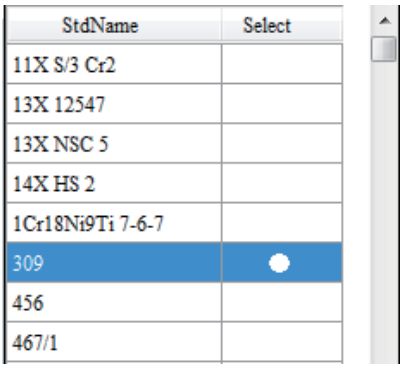


Fig.41 Standard sample selected

If the standard to be added cannot be found in the standard list of the *StdDatabase*, you must first add the standard to the standard list before selecting it as a type sample.

The process for this is to insert three steps between (2) and step (3) in 4.5.2:

(a) Click *New* button under the *StdDatabase* tab;

(b) Input the element symbols and contents of the standard according to the certificate or test report of the standard. There is no need to input the content of the matrix, which is calculated automatically by the software.

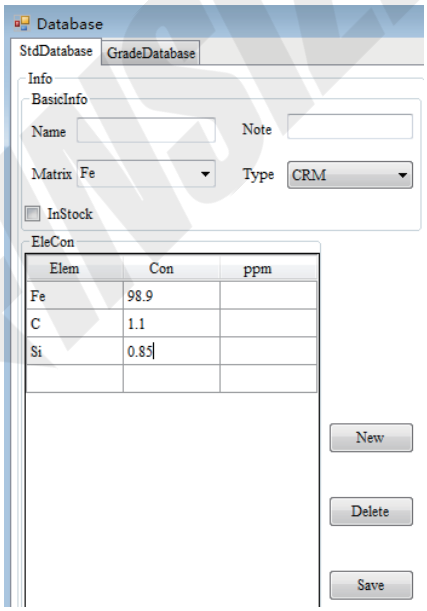


Fig.42 Add new standard sample

(c) Click *Save* button to add the new standard sample to the standard database after completing inputting of the elements and contents.

4.6 Analysis

The spectrometer can be used to analyze customer samples (consistent with the current using

type standard).

To analyze a customer sample, clean the electrode with electrode brush, then place the prepared sample on the spark stand. Click *Spark* button, and the analysis result is obtained at the end of the excitation. It is generally recommended that each sample be excited at least two points, deleting the outliers and report the average value of remaining data as the final result. Refer to the 6.1 of this manual if the analysis results are too volatile. To save or print the analysis results, refer to 3.3.11 and 3.3.12.

5 Maintenance

Maintaining the instrument according to a certain period and specification is an important basis for ensuring the normal performance of the instrument. It will lead to poor performance of the instrument or affect the accuracy and stability of the analysis results, or even shorten the service period of the spectrometer in severe cases if the maintenance period is prolonged or not complying with specification. And stability, in severe cases may shorten the life of the instrument.

For the saftey of the operating staff, it is recommended to exit the analytical software of the spectrometer and turn off the emergency button(push the button, a closed light indicates its off) at the bottom of the intrusment's front cover before maintenance process.

5.1 Common maintenance

There are three parts for the common maintenance of OES-R420 series spectromter, i.e. cleaning spark stand, cleaning the optical lens and cleaning the dust filtering cartridge.

Take off the hose between the dust collecter and the water tank first before cleaning the spark stand or dust collecter with a vacuum cleaner to avoid water from absorbing into the instrument and damage of the instrument.

Fig.43 Unplug the hose of the water tank before cleaning with vacuum cleaner

5.1.1 Cleaning the spark stand

The spark stand is a device where the sample is excited and light is emitted. Most of the ash generated by the sample excitation is carried into the exhaust gas filtering device by argon gas, but a small amount of ash remains in the spark stand, so the spark stand needs to be cleaned regularly.

The cleaning process is as follows: Remove the fixing screws at the four corners of the cover of the spark stand and carefully take off the cover. Use a vacuum cleaner to clean the accumulated ash on the upper cover and in the cavity of the intermediate copper plate while cleaning with a brush (the hose of the water tank has been removed).

Assemble the spark stand cover as it is after cleaning.

It is recommended that the spark table be cleaned after changing the sample substrate or using an argon cylinder.

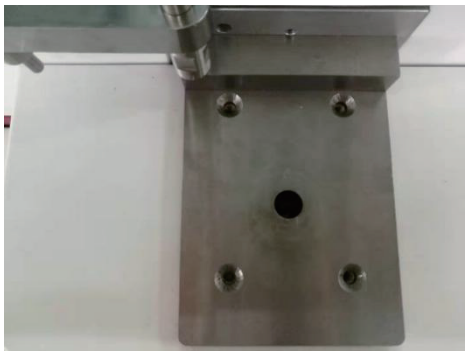


Fig.44 Take off screws of the spark stand cover

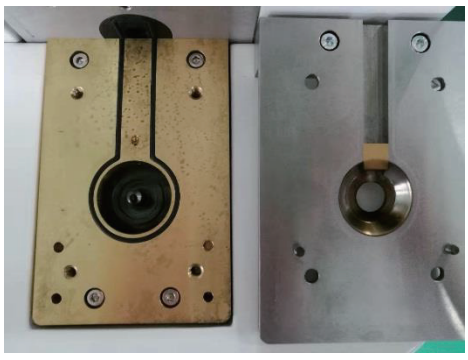


Fig.45 Take away the spark stand cover

5.1.2 Cleaning the optical lens

The light generated by the sample in the spark stand enters the optics chamber through the lens. Whether the lens is clean relates to the overall strength of the instrument. As the instrument is long time used, the lens will become dirty. When the intensity reduces to a certain level, the lens needs to be cleaned to restore the overall strength of the instrument.

The cleaning process of the lens is as follows:

（1）Open the upper cover of the instrument, pull out the lens holder vertically and remove the mobile plate of the lens holder horizontally to the left;

（2）Place the mobile plate with the O-ring facing down, and press the lens from top to bottom through the special lens paper.

（3）Dip the lens into absolute ethanol(purity higher than 99.7%) for 3 to 5 minutes, then **clean with lens paper carefully**. Distinguish the plane and convex surface when cleaning the lens. If there is obvious oil film on the lens surface and cannot be cleaned with absolute ethanol, it can be wiped with translucent toothpaste (no obvious particles inside) and rinsed with water, then dip into absolute ethanol, and finally wiped with lens paper.

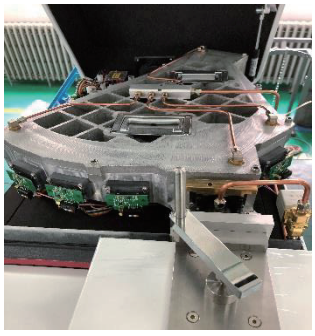


Fig.48 Open the instrument cover

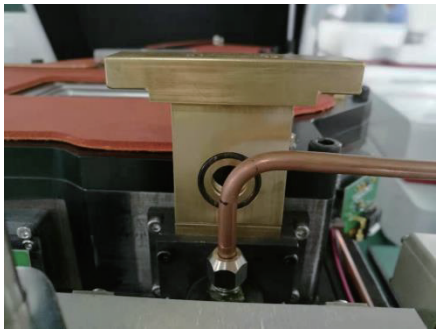


Fig.49 Pull the lens frame upward

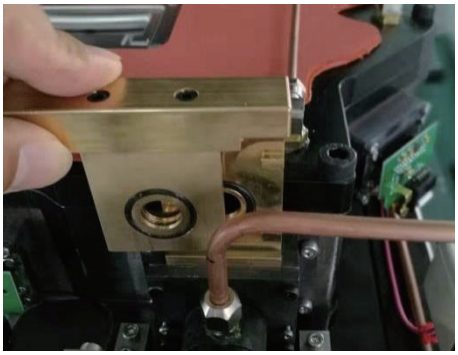


Fig.50 Take off the mobile part

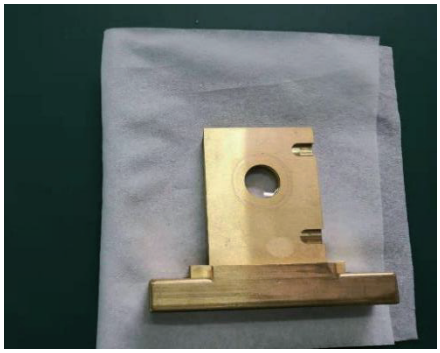


Fig.51 O-ring side down

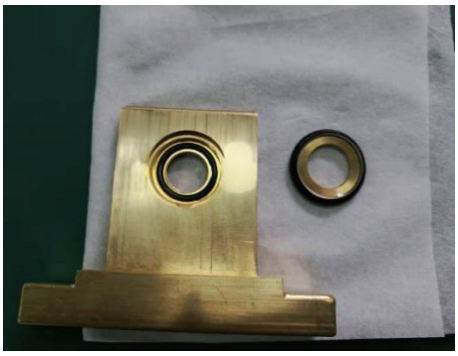


Fig.52 Take out the lens

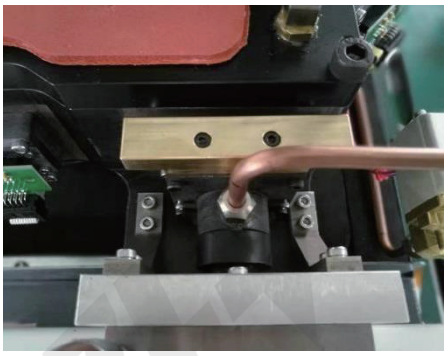


Fig.53 Mount back the lens frame

（4）Confirm the O-ring is located in the positioning groove of the mobile plate, then place the lens into the positioning groove with its convex side upwards. Gently tap the edge of the mobile plate to let the lens fall into the positioning groove correctly.

（5）Put the copper ring above the lens and press the O-ring on the copper ring carefully so that the copper ring and the O-ring are flush with the surface of the mobile plate;

（6）Match the mobile plate with the fixed plate of the lens holder, then mount back the lens frame.

It is recommended to clean the lens after changing the argon cylinder for 3 times.

The lens of the N-containing instrument is made of different material, and can not be dipped into absolute ethanol regardless of rinsing with or dipping into water. It can be directly wiped with lens paper wetted by a small amount of absolute ethanol.

5.1.3 Cleaning the dust filter cartridges

The exhaust gas filtering system is used for collecting ash generated by the excitation and filtering the exhaust gas. It mainly consists of three parts: exhaust hoses, a dust collector and a filtering water tank. When there is too much ash accumulated in the dust collector and the exhaust pipe, the instrument will be exhausted poorly, which will affect the pressure in the spark stand and cause data instability. Too much or too little water in the water tank may affect the filtration of the dust. Therefore, it is necessary to regularly clean and maintain the exhaust gas filtering system.

The main steps of the maintenance process are as follows:

（1）Filtering water tank: check and keep the water level constantly between 1/3 and 1/2 of the tank height. Replace with gap water if the water in the tank is black and dirty;

（2）Dust collector: Open the front cover of the instrument to take off the dust collector. Open the upper lid of the collector and clean dust inside the collector with brush and vacuum cleaner (the hose of the water tank has been unplugged). Close the upper lid of the dust collector tightly after cleaning;

（3）Exhaust pipe: unplug different segments of the exhaust pipe, clean the dust inside with vacuum cleaner. Plug the pipes on the water tank and dust collector, not to knot the pipe while connecting.

Fig.54 Water tank(red line for water level)

Fig.55 Dust collector

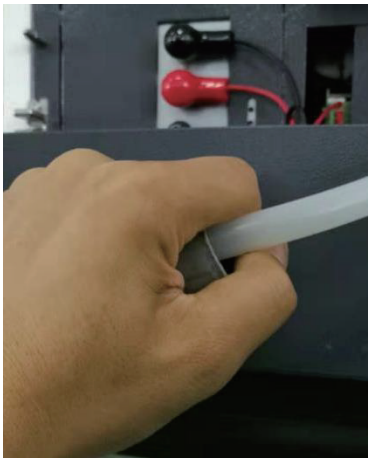


Fig.56 Cleaning hose

It is recommended to check the water level in the water tank every day and refill or change the water in time if necessary. The dust collector and exhaust pipe are recommended to clean after using 10 cylinders of argon gas.

5.2 Scheduled maintenance

There are two parts of scheduled maintenance for OES-R420 series spectromter, i.e. maintenance and replacement of electrode and maintenance of argon filter cartridge.

5.2.1 Cleaning/changing the electrode

The electrode is a component that generates a discharge between it and the sample. When the sample is excited, especially for soft metals such as Zn, Al or Pb, a small amount of sample will be deposited on the electrode surface, and the accumulation of deposit will affect the excitation of the sample. Therefore, the electrode needs to be maintained regularly.

The process of maintaining electrode is:

Fig.57 Electrode gauge

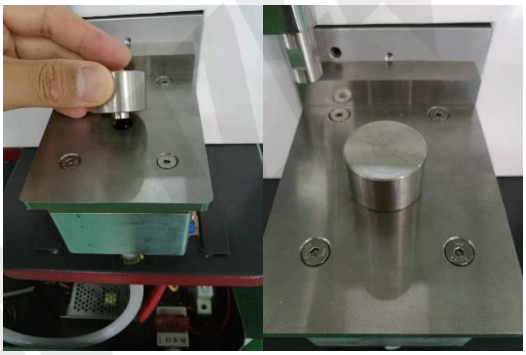


Fig.58 Cover the electrode gauge

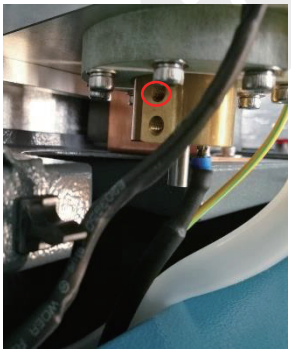


Fig.59 Electrode fixing (red circle)

Fig.60 Electrode

(1) Take off the electrode: Open the front cover of the instrument by pushing the protruding buttons on both sides of the cover. Cover the spark hole of the spark stand with the electrode gauge, then release the fixing screw of the electrode with an Allen key and take it off;

Fig.61 Open the front cover

（2）Maintaining the electrode: Carefully grind or scrape off deposits on electrode surface with a blade or fine sandpaper of 200 mesh or more along the original angle of the electrode.

（3）Electrode height positioning: Place the electrode flat head down to the electrode fixing hole of the spark stand, cover the electrode on the spark hole and push the bottom of the electrode upward, so that the electrode tip is just flush with the bottom surface of the pole gauge. Fasten the electrode fixing screw.

（4）Replace the electrode: If the electrode angle is obviously blunt and cannot be recovered by electrode maintenance, replace with a spare electrode and position electrode height according to step (3).

It is recommended to maintain and positioning the electrode after 30,000 sparks. The maintenance period should be shorter if soft metals are often analyzed.

5.2.2 Changing argon filter

The argon filter, which locates between the argon inlet port of the instrument and the inlet port of the optics chamber, is used to filter the small amount of oil and water vapor in the argon gas. There are absorbent and indicator in the filter cartridge and the indicator is blue before absorbing water. Replace the argon filter cartridge immediately if the indicator turns to pink.

Fig.62 Argon filter (far right)

6 Troubleshooting

The resolving methods of simple problems that OES-R420 series spectromter may encounter are listed in this part. If any problem occurs during operation, please distinguish fault type and check according to the steps respectively. Necessary tools such as Allen keys, screwdrivers, test pencil, multimeter, should be prepared before inspection. **Those must be inspected by a professional electrician for the troubles relevant with circuit units. Please be careful during the inspection for your own safety.** If you are still unable to solve the problem after completing the prompt inspection, please contact Customer Service Department.

6.1 Inaccurate or unstable results

6.1.1 Sample preparation problem

Check whether the surface of the prepared sample is flat, whether there are oil or water stains on the surface (It is **strictly forbidden to touch the prepared surface or rinse with water or clean with rag.** The surface of the sample must **have a clean grinding texture, not too smooth**, otherwise it may lead to a bad spark and result). Make sure the spark sound is normal. If the sound is hoarse or shaking, check for cracks, inclusions or defects in the spark area. If the spark sound is harsh, check the flatness of the sample surface and make sure the spark hole of the spark stand is covered completely.

6.1.2 Abnormal spark spot

Please refer to 4.1.2 for the checking criteria of spark spot. Possible causes and solutions for abnormal spots are as follows:

(1) **Sample problem:** The sample itself is not suitable for spectral excitation (such as gray iron, ductile iron sample without “whitening” treatment or powder metallurgical sample with insufficient internal density);

(2) **The pressure or flow rate in the argon pipe not in the normal range:** check the

pressure in the argon cylinder and reducing pressure, confirm the maintained flowrate and working flowrate;

(3) **Possible air leakage in the gas path:** check whether the joints are locked in the gas path, and whether the screws are tight on the upper cover of the optics chamber;

(4) **Poor environment inside the optics chamber:** flush the optics chamber with a short process or a long process;

(5) **Air mixed with argon in the spark stand:** spark waste sample for 3~5 points;

(6) **Insufficient purity of argon**: replace argon cylinder with better purity or equip with argon purifier.

6.1.3 Inadequate cleaning and maintenance

The analytical results may get worse if the instrument has not been cleaned for a long time, or is not cleaned according to the specifications (excessive ash accumulation in spark stand, clogging of the exhaust hose, abnormal water level of the water tank or dirty lens). Please clean the instrument according to the maintenance specifications.

6.1.4 Lab condition problem

Fig.63 Optics temperature display (OES-R420)

The light chamber temperature of OES-R420 is generally set at 30°C, and the actual temperature is displayed at the bottom left of the software interface (Figure 6-1 above). The light chamber temperature of OES-R420 is usually set at 32°C, and temperature control is achieved through the high-precision temperature control module at the back of the

instrument (Figure 6-1 below, the green numbers at the bottom of the figure indicate the set temperature, while the red numbers at the top indicate the actual temperature). Check if the displayed actual temperature of the light chamber matches the set temperature. If the actual temperature does not match the set temperature, it may be due to insufficient stabilization time of the instrument, high or low laboratory temperature, or excessive fluctuations, in which case the stabilization time of the instrument may need to be extended or the air conditioning settings adjusted to maintain the room temperature constant between 20~25°C.

In addition, when there is a significant vibration source around the laboratory, internal optical components of the instrument may be displaced, resulting in data instability and inaccuracy. Vibration reduction measures should be taken in this situation.

6.1.5 Standardization problem

Check if the peak pixel offset, global correction coefficients and type correction coefficients are within the normal range. New correction is required if the normal range is exceeded or the recommended correction period is exceeded. During the standardization process, it is necessary to pay close attention to the spark spot, whether the standard used is consistent with the selected standard, whether the standard used for the type calibration is the same or similar to the actual analysis sample: it may lead to a huge error if the grade difference is too large or the metallurgical process and physical state are inconsistent.

If you want to check global or type standardization coefficients, click *ProgCoeff* item in *Function* menu, then click *GlobalCoeff* (or *IntExpectCoeff*, depending on setting when doing global standardization, refer to 4.4) or *TypeCoeff* respectively.

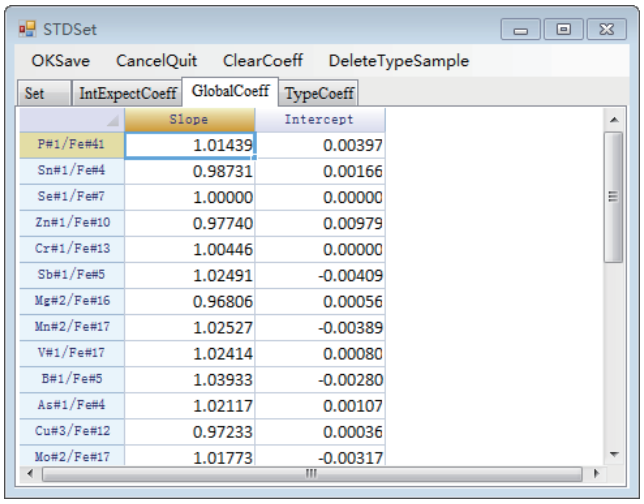


Fig.64 Check Curve Coefficients

6.1.6 Sample inhomogeneity

When results of the test sample or standard sample are unstable, you can continuously spark the global standard for 3 to 5 points, and check whether the stability of the result meets the requirements. If the stability of the global standard is good, but the results of test sample or other standards are not, it is due to the unevenness of the test sample or other standards. You are suggested to replace the sample (or standard sample) or average multiple sparks of the test sample or other standard.

6.1.7 Electrode inspection

Check whether there are many deposits on the electrode surface and it needs to maintain, confirm whether the electrode distance is normal. Replace with a spare electrode if the electrode angle is obviously passivated.

6.2 Spark error

6.2.1 Confirmation communication between spectrometer and computer

If there is argon flowing in the gas path of the spectrometer after clicking the *Spark* button, it

indicates that the communication between spectrometer and the computer is OK, you can directly transfer to 6.2.2. Otherwise, you should check according to the following steps:

(1) Check whether the data cable (network cable) is plugged in at both ends, and whether the indicator of the network card in the computer is normal. If necessary, replace the network cable, network card or reinstall the network card driver.

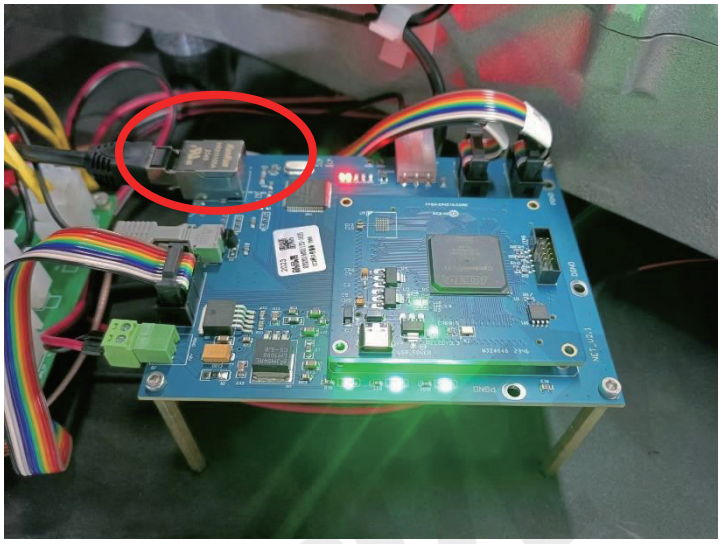


Fig.65 Fixing data cable (top left)

(2) Confirm the setting parameter of the network card in the computer, the right setting for IP address is 192.168.1.18 and the subnet mask should be 255.255.255.0.

6.2.2 Checking Emergency button

Check whether the emergency button is turned on at the bottom of the front cover. Rotate clockwise to turn it on if it is closed, a red light indicates its working state.

6.2.3 Checking spark source switch

Check whether the *Source* switch in the *Test* menu is in *ON* status.

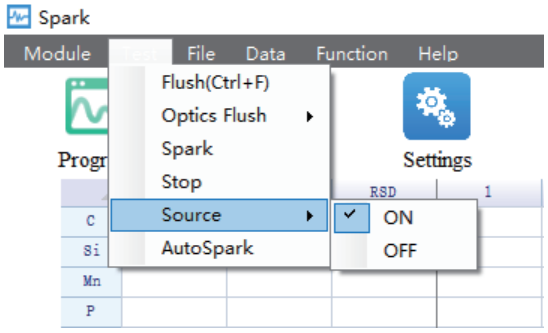


Fig.66 Check spark source switch

6.2.4 Checking spark source fuse

Turn off the spark source and emergency button and take off the front cover of the instrument, then open the white fuse box and take out the fuse. Check whether the fuse is burnt out with a multimeter, and replace it with a spare fuse if it is not conductive.

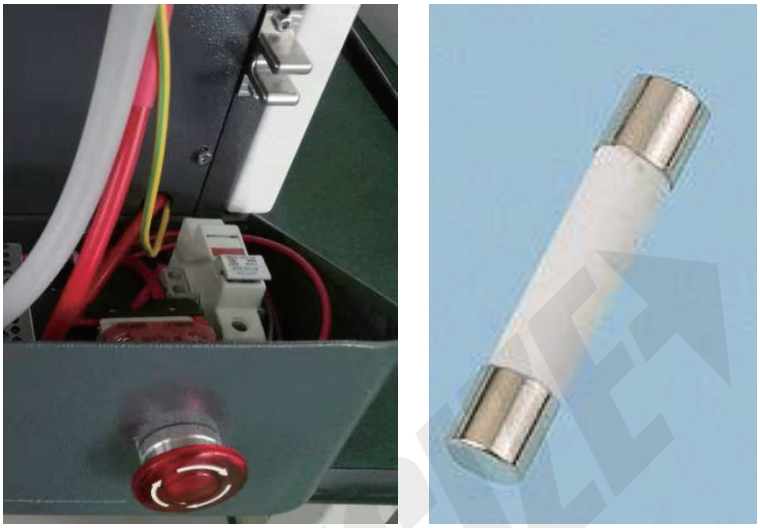


Fig.67 Fuse box (white) and fuse

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