# DW-ICP-OES3000 User Manual



Please read operating manual before installation and operation.

# **Drawell International Technology Limited**

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# Chapter 1 General

Drawell International Technology Limited. is a high-tech international enterprise specialized in R&D, manufacturing and sales of the analytical testing instruments of spectroscopy, chromatography and mass spectrometry. We provide customers with competitive solutions and services, rank No.1 of sales and varieties in the world analysis instrument industry. Our products have been widely applied in electrics, jewelry, toy safety, building materials, metallurgy, petroleum, chemical, geological mining, commodity inspection, quality inspection or even human body trace elements testing, etc.. With advanced R&D team, we will provide global customers with better quality, more cost-effective products and services.

#### 1.1 Product general

ICP3000-Spectrum Direct Reading Inductively Coupled Plasma Emission Spectrometer-is a high-tech precision instrument, mainly applied in the rare earth industry, silicon industry, petrochemicals, mineral analysis, metal smelting, geological research, drug safety, laboratory research, environmental testing and food.

#### 1.2 Working environment

Item	Range
Store&transport Temp.	15°C-25°C
Store&transport relative humidity	≤70%
Atmospheric pressure	86-106 kPa
Power	220±10V 50-60Hz
Working humidity	≤70%
Working Temp.	15°C-30°C

# Chapter 2 Structure & Principle

# 2.1 Basic structure

# 2.1.1 Appearance



#### 2.1.2 Internal structure



- 1. Polychromator
- 2. Computer control board module
- 3. Auxiliary board module
- 4. CID Detector
- 5. Auto-match box
- 6. Torch collimation adjustable platform
- 7. Water/gas interface module
- 8. Solid power box





#### 2.1.3 Sampling system



- 1. Torch coil
- 2. Torch
- 3. Inlet tube
- $4. \ N_2 \ purge \ quick \ connector$
- 5. Plasma gas quick connector
- 6. Supporting gas quick connector
- 7. Carrier gas quick connector
- 8. Binocular spray chamber
- 9. Nebulizer
- 10. Waste container



# 2.1.4 Torch collimation adjustable platform

Adjust platform: computer-controlled, adjust the flame to the optimal observation position.



2.1.5 Water/gas interface



# 2.2 Basic working principle

RF generator applies the high frequency power onto the tri-concentric quartz torch via induction coil to generate a high-frequency oscillating electromagnetic field; Ar is purged from the outer layer of the quartz tube and charged by HV discharging, those charged particles reciprocate in the high-frequency electromagnetic field and collide with other Ar atoms to produce more charged particles, finally generate Ar plasma(6000K $\sim$ 8000K) with a rise of temperature. The aerosol generated by aqueous sample passing through the nebulizer is excited the characteristic lines of the elements under high temperature by entering the quartz torch center channel, which is correctly positioned to CID detector pixels by using a Plasma source and a polychromator for spectroscopic imaging and transform the spectral intensity into signal, transmit to the computer for data processing,finally print the result out.



#### 2.3 Principle of key components

#### 2.3.1 Inductively coupled plasma(ICP) formation principle

High-frequency generator (oscillation frequency 27.12MHz, max. output 1600W) is used to produce a high-frequency electromagnetic field to provide plasma energy. The torch is a tri-concentric quartz glass tube (or made of other materials). Purge the cooled Ar into the outer layer of the tube to avoid the tube being damaged by the plasma torch. The middle layer is made into a "horn"shape, purged Ar to maintain the plasma. The inner layer is  $\varphi$ 1mm-2mm,the aerosol is introduced into the plasma by using carrier gas. When the load induction coil (2-5 turns of the water cooling coil winding by round or square brass tube) around the plasma torch is powered,the high frequency induction current flows through the coil and generates the axial high frequency magnetic field. Meantime, purge the cooling gas tangentially into the outer tube and the supporting gas(Ar) axially (or tangentially) into the middle tube of the torch ,excite the charged articles by using high-frequency igniter, as the articles increase enough for a gas conduction, a circular eddy current will be shaped on the section perpendicular to the magnetic field. A few hundreds of induced current instantly heat up the gas to 6000K-8000K, generating a torch-shaped plasma at the top of the tube.

#### 2.3.2 Solid-state generator and auto-match box

#### Solid-state generator

ICP3000 high-frequency generator is a full solid-state RF generator with separate-excited oscillation circuit (max. output power 1600W, frequency 27.12MHz) manufactured by Drawell International Technology

Limited. Compared with the self-excited oscillation electron tube RF generator, it has the advantage of smaller size, higher output, stabler frequency and more efficiency, etc..

#### Auto-match box

In general, the output impedance and the load impedance are different, inductance capacitance is distributed in the circuits, therefore in order to get a max. heating power for the plasma load, an impedance matching is needed between the load and power source. ICP3000 auto-matching box, by using Drawell self-developed auto-matching technology of  $\Gamma$ -shaped matching network, has the advantages of fast matching, high precision, and saving the troubles of manual matching, etc...

#### 2.3.3 Polychromator

Polychromator is composed by the light chamber, illuminating path, slit, collimator, echelle, prism, focus mirror and CID detector. The light chamber focal length is 440mm. The complex light emitted by ICP light source focuses in the illuminating path and incidences onto the collimating mirror through the slit, the reflected light passing through the prism is then diffracted by the echelle ,passes the prism and finally takes image on the focus mirror, the CID detector is used to measure the spectral signals.



#### 2.3.4 Electronic measurement and control circuits

The circuit system has 4 functions, including communication, gas path control, stepper motor control and signal acquisition.

#### 1. Communication

RJ45 net port is used as the communication interface. Net port communication has the advantages of stability, fast communication, strong anti-interference,etc..

#### 2. Gas path control

ICP3000 gas path adopts the advanced MFC (mass flow controller) as the control unit for plasma gas, carrier gas, supporting gas control. With the advantages of high control accuracy, fast response, flow stability and provided with the flow feedback function, it ensures the stability of injection system and improves repeatability and stability by real-time monitoring the actual gas flow.

# **Chapter 3** Technical Specifications

#### 3.1 High-frequency generator

- 1. Circuit Type: Full-solid RF power supply with auto-matching function.
- 2. Working Frequency: 27.12MHz
- 3. Frequency Stability: <0.1%
- 4. Output Power: 800W  $\sim$ 1600W
- 5. Output stability:  $\leq 0.2\%$

6. Electromagnetic leakage radiation intensity: at 30cm away from the instrument case, electric field: E < 10V/m; magnetic field: H < 0.2A/m

- 7. Input Power: 220V, 30A
- 8. Output coil inner diameter 25mm, 3 turns, with tri-concentric quartz torches with outer diameter

20mm

- 9. Coaxial nebulizer outer diameter: 6mm; binocular spray chamber outer diameter: 35mm
- 10. Gas flow:
  - Plasma gas flowmeter:  $(1 \sim 20)$  L/min Supporting gas flowmeter:  $(0.05 \sim 1.0)$  L/min
  - Carrier gas flowmeter:  $(0.05 \sim 1.0)$  L/min
- 11. Cooling water: temp. range 20°C  $\sim$  25°C, flow >7 L/min, water pressure > 0.1MPa, resistivity > 1M $\Omega$ .

#### 3.2 Polychromator

- 1. Optical path: Echelle + cross dispersive prism
- 2. Focal length: 440mm
- 3. Specification: Echelle, Zerodur® base, groove density 52.67lp / mm, blaze angle 64°; ruled area
- $(80 \times 110) \text{ mm}$ 
  - 4. Resolution: at 200nm<0.007nm; at 257.61nm<0.010nm
  - 5. Numerical aperture: >F/8
  - 6. Slit: customized as needed
  - 7. Wavelength Range: 175nm  $\sim$ 870nm
  - 8. Thermostatic temperature: 35±0.1°C
  - 9. N2 purge: High-speed 6L/min, normal 2L/min

#### 3.3 CID Detector

- 1. Size: 1024×1024, 27.6mm×27.6mm
- 2. Potential well depth: >500000e-
- 3. Read noise: <24e- @128NDRO
- 4. Detection wavelength range: 165nm~900nm
- 5. Quantum efficiency: >30%@200nm
- 6. Refrigeration temperature: -45°C
- 7. N<sub>2</sub> purge: 1L/min

#### 3.4 Communication circuit: net port communication

#### 3.5 PC system

- 1. Brand computer: 1 set, operating system:XP, WIN7
- 2. Printer: Panasonic KX-P1121

#### 3.6 Technical parameter

- 1. Wavelength Range: 175nm~900nm
- 2. Repeatability: RSD≤0.5%
- 3. Stability: RSD≤1%
- 4. Detection limit  $(\mu g/L)$

# Chapter 4 Installation&Connection

# 4.1 Pre-preparation

Please read the *Installation Notice* carefully and make the preparations as required before our technicians' arrival.

# 4.2 Installation flowchart



#### 1. Site selection

Dimensions: 1300mm(L)×840mm (W) ×740mm (H) ,weight:230kg. See *ICP3000 Installation Conditions* for the installation.

#### 2. Unpack&Check

Uncover the instrument(top and rear), take out the accessory case, check any missing based on the packing list and make sure there's no damage on the instrument case.

#### 3. Cooling water installation

Take the cooling pipes and gas path plastic pipes out from the accessory case. Connect the cooling pipes to the cooling water ports at the instrument lower back(Note: the cooling tank outlet pipe is connected to the instrument inlet, return pipe to the outlet. Then turn on the pump power, check the cooling water joints (including solid state power supply cooling water connections, induction coil connections, CID cooling water joints) for water leakage and the pull-in of the water switch(note the pull-in sound). There are 3 gas pipes, in which two for Ar, connecting to plasma gas and carrier gas inlet respectively with the other ends to the gas reducer of Ar cylinder; for the third one, one end connects to  $N_2$  inlet with the other end to the reducer of  $N_2$  cylinder.

Note: Ar and N<sub>2</sub> can not be connected reversely, otherwise lead to ignition failure; frequent ignition may damage the solid state power.

#### 4. Injection system installation

Take the quartz torch, spray chamber, nebulizer, waste bottles out from the accessory case. Quartz torch is installed in the center of high frequency induction coil and keeps concentric with the coil. The lowest coil should be 3-5mm higher than the central nozzle of the quartz torch .Plasma gas plastic pipe and supporting gas plastic pipe are respectively connected to the quartz torch upper inlet and second inlet. Spray chamber top joint is connected to the quartz torch bottom joint prior to clip firmly. Nebulizer inlet is connected to the carrier gas plastic pipe and insert the nebulizer gas nozzle into the opening of the spray chamber. The capillary/nebulizer joint is leakless. Use  $\Phi$ 8mm plastic hose to connect the peristaltic pump latex tube to the back of the spray chamber.A cotton cord in the waste hose is used to discharge the waste outside freely ,after the injection system is installed, the burning torch output wire (red HV wire)is sleeved at the quartz torch plasma gas inlet.

#### 5. Gas adjustment

Open  $N_2$  cylinder, adjust the outlet pressure to 0.2MPa; open two Ar cylinders, adjust both plasma gas and carrier gas outlet pressure to 0.2MPa. Open the gas by computer-control and check the leakage.

#### 6. Optical path debug

Uncover the thermostat insulation cotton, open the cover plate on top of the polychromator, observe whether the illumination path reflector, collimator and focusing mirror have drop-off; install the echelle base onto the polychromator backplane, recover and tighten the nut.

#### 7. Circuit connection

Open the accessory case. Take out the black power cord, with one end connected to the air switch of the power supply (>32A) or regulator (>5kW), [Note: both green wire twisted together are connected to the fire wire!]. Computer net port is connected to the communication output at the rear of the instrument, computer and printer are connected according to the instruction. High-frequency ground wire is connected with the ground terminal. Computer ground wire is connected with the instrument ground wire.

Note: Please finish the above steps under the guidance of our technicians.

# **Chapter 5 Operation**

#### 5.1 Instrument operation process

Note: Before the instrument is turned on,first make sure: 1) stable working conditions under constant temperature: Thermostatic indicator has flashed for more than one hour; 2) CID  $N_2$  has purged for more than one hour.CID is an precious apparatus, which will be damaged if start the refrigeration operation without

adequate purging.



#### 1. Turn on the air switch

There's fan rotation sound when turn on the main switch, indicating the power is working properly.

#### 2. Startup the cooling tank

There's pull-in sound of the water switch when power on the cooling water pump. If the cooling tank is not turned on,the ignition of the instrument is unavailable.

#### 3. Turn on the instrument power

Switch on the control power at the rear of the instrument.

Note: switch on control circuit $\rightarrow$ air switch $\rightarrow$ CID refrigeration. To turn off the instrument, turn off the air switch $\rightarrow$ control circuit.

#### 4. Ignition

After online success (including CID connection, temperature display), directly click Ignition.

For ignition airflow settings, recommends the default.

Ignition gas purge time is 30s.

If the ignition fails, first flameout, then check the gas path according to the prompted.

After the ignition, click *Injection preparation*. Note: avoid the air inlet during injection, otherwise, lead to flameout. While not testing, put the injecting tube into the washing fluid.

#### 5. Start the test

Prepare methods for the test.

Note: 1 CID normal temperature is -45°C, the measurement is only allowed below -30°C;

2 For normal operation of CID refrigeration ,it is required that the water tank works properly,  $N_2$  purge is turned on(flow>0.8L/min);

3 Instrument warm-up for 20min.

#### Additional notes:

1 Before the ignition, observe the cooling tank level, and the cooling system operation condition.

2 Observe the pressure gauge on the Ar cylinder, make sure Ar is enough for the test. Plasma consumption is about 1.1MPa/H, the carrier gas consumption is about 0.1MPa/H.

3 Observe the pressure gauge on the  $N_2$  cylinder,make sure  $N_2$  is enough for the test; if  $N_2$  purge is not turned on,  $N_2$  consumption<0.1MPa/H; otherwise, $N_2$  consumption<1MPa/H.

4 Cooling air: 10L/min~12L/min.

5 The optimal pressure of carrier gas is determined according to the nebulizer lift volume(normally about 0.7L/min).

6Ignition is not allowed if the indoor humidity > 70%, or room temperature > 30°C.

#### 5.2 Instrument shutdown process

The instrument should be shutdown strictly according to the standard, to avoid damage to the instrument caused by the malfunction and affect the regular test. The correct shutdown procedure is showed below. Please read carefully before operation.



Note: shutdown CID N2 purge source until CID temp. returns to normal.

#### 5.3 Software operation

After the ICP torch is stable and ensure a continuous injection, the customized software is available for testing. For detailed steps, please refer to "*Software Manual*".

# **Chapter 6** Software instructions

ICP analysis software is provided with this instrument.

6.1 Software start-up

Double-click the desktop shortcut using icon to display the welcome interface as shown in Figure 6.1.





Choose a user name to login and enter the corresponding password, click *Login* to enter the main interface as shown in Figure 6.2, click *Cancel* to exit the login interface.



Fig.6.2

The main interface is divided into 5 parts, including menu bar, toolbar, status bar, data display area and analysis measure&control. Analysis measure&control consists of 3 parts, including analysis measurement, analysis method, auto-analysis measurement as shown in Figure 6.3.



# 6.2 Analysis method

#### 6.2.1 New method

As shown in Figure 6.4, enter the name of the new method and save it, click OK to create a new method.

Wethod Nam	ne:		Results Name:	Add
4	MethodName	Results	Creator	Date
<u>۲</u>	0122	1	Admin	2019-01-22 14:44:53
	1220z	1	Admin	2018-12-20 14:59:32
	1219z	1	Admin	2018-12-19 14:46:06
	1218z	1	Admin	2018-12-18 14:48:16
	1217z	1	Admin	2018-12-17 15:07:24
	12121 z	1	Admin	2018-12-12 15:27:30
	1212z	1	Admin	2018-12-12 11:15:02
	1211z	1	Admin	2018-12-11 15:01:25
	1210z	1	Admin	2018-12-10 14:47:26
			C 109-000	0010 10 07 14 11 07



Click OK, the Add interface of analysis spectra will pop up as shown in Figure 6.5.



Fig.6.5

Click the element name in the periodic table of elements to show its spectral line in the list below, select the line, click *Add*, it will be added into the *Selected wavelength* list; click *OK*, it will be added to the method being edited and return to the main interface.

Delete: Remove the currently selected analytical lines.

6.2.2 Analysis parameter settings

Click *Method> Analysis Parameter*, setup the repeat times, sample rinse time, integration time (Figure 6.6).

🗊 ICP0122		
Instrument Method Analysis	Data Management – User Manageme	nent Instrument Diagnostics Language Help
🛔 🖬 🗢 🖵 🗹 🕯	i 📐 🕺 📾 🛒 i	‰ ⊙
Analysis «	Analysis Preference	
Andyzis Preference Standards Diamon Lineart Zn [213.856] Ni [231.604] Ni [231.604] Ni [231.604]	Measurement Options:	3
Cr {267.716}	#sample Repeat:	3
Method Reports	Delay time:	1 5
	Flush Time:	10 s
	Flush Speed:	0.5 ml/min
	Precision:	3
	Normalized:	Mormalize
	Integration Time:	15

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Fig.6.6

#### 6.2.3 Standard

Click *Method> Analysis Parameters*, you can add or delete the standard, select the elements and the desired lines contained in the standard, setup and modify the element content (Figure 6.7).

	v 🖄 🕺 🗐 🖗	0				
Analysis X « Analysis Preference Standards Element	Standards -					
Zn {213. 856}	Standards Name		Standar	ds Name: 1 Element	Content	1
Mn (257.61) 	1			Zn{213.856}	0	
Cu{324.754}	2			Ni {231.604}	0	
■ Ba{455.403} ■ <edit elements=""></edit>	3			Mn {257.61}	0	
🍘 Method Reports	4			Cr {267.716}	0	
				Cu{324.754}	0	
		Add		Ba{455.403}	0	



1. Add: Click *Add* to pop-up the Figure 6.8 to add new standard.

New Standard Sample	e	x
Standard Name:	DalibStdl	
Default:	10.00	
Ok	Cancel	

Fig.6.8

- 2. Delete: Delete the standard.
- 3. Edit: Click the left standard name, check the analysis lines to be measured at right.





#### 6.2.4 Select methods

To open a method, either click *Analysis method -> Open method -> OK* as shown in Figure 6.10, or by double-click a method. You must select a method and open it prior to analysis.

1	Method	Results	Creator	Date	-
	winni	1	Admin	2019-02-12 15:48:16	
	0122	1	Admin	2019-01-22 14:44:53	
	1220 z	1	Admin	2018-12-20 14:59:32	
	1219z	1	Admin	2018-12-19 14:46:06	Ok
	1218z	1	Admin	2018-12-18 14:48:16	L
	1217 z	1	Admin	2018-12-17 15:07:24	
	12121 z	1	Admin	2018-12-12 15:27:30	Cane
	1212z	1	Admin	2018-12-12 11:15:02	
	1211 z	1	Admin	2018-12-11 15:01:25	
	1210z	1	Admin	2018-12-10 14:47:26	
	1207j	1	Admin	2018-12-07 14:11:37	



# 6.3 On-line

#### 6.3.1 On-line

Switch on the power of the main control board, click *Instrument Control -> Online*, as shown in Figure 6.11, if online success, display *Online success* at the lower left of the status bar, otherwise, display *Online failed*, then you need to check the instrument is connected properly to PC prior to click *Instrument Control -> Online*.

			COMPANY OF THE OWNER WATER	and Million	Real Road Real Prod.	-		- 6 <b>x</b>
Instrument Method Analys	is Data Management	User Management	Instrument Diagnostics	Language	Help			
💧 🖬 🦘 🖵 🗹	🔤 😣 🖄 🕷	🛒 🗶 🥥						
Analysis «	[							x
Progress of analysis								
Analysis measurement								
Standard Analysis								
Blank Analysis								
Sample Analysis								
Group of results								
Analysis Lines								
Liement Wavelength								
Analysis [interned   Auto sar 4 ]						CID T-	Adulta A	(010)
Connect railure:						Cito remp:	Admin	00

Fig.6.11

#### 6.3.2 CID link

After online successful, the system will automatically link CID and refresh CID temperature in the status bar, the measurement begins when the temperature  $<-30^{\circ}$ C. If the link fails, click *Instrument Control -> link CID* to relink.

6.3.3 Plasma Control

Click Instrument Control -> Plasma Control or to pop-up Figure 6.12. Open the valve of plasma

gas, carrier gas and supporting gas, setup the gas flow and power. Click to real-time refresh and view the actual gas flow (as shown in Figure 6.13). Before ignition, please make sure:

1. The temperature of light chamber is stabilized at about 35 °C;

2.CID  $N_2$  purge for more than one hour to prevent CID detector chip from frosting leading to damage to CID detector;

3. Check and make sure the injection system properly installed;

4.Open the exhaustion system.

5. Mount the peristaltic pump holder, put the sample tube into the flushing fluid.

	Commissi (R.G. (alla))		When the (a)	
	carrier or (Lymin).		ridsh fime(s).	
	0. 70 🚔		30	
Gas Conti	rol			Plasma On
	Plasma GF (L/min):	12.00	On Off	
	Auxiliary GF(L/min):	0.60 🜲	On Off	Sampling Ready
ampling	Control			
	Carrier GF(L/min):	0. 70 🌲	On Off	Plasma OFF
	Peristaltic Pump:	2.0	On Off	
	Rotate Speed:	Normal	Fast	Default GF
ower Se	tting			

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Fig.6.12

1. Gas control

Control the valves of plasma gas, Auxiliary gas, and setup their gas flow.

2. Sampling Control

Control the carrier gas and the peristaltic pump, setup the flow rate of carrier gas, the speed of peristaltic pump. Select "Normal"during the measurement, shift to "Fast" for discharging the waste in the spray chamber.

3. Power Setting

Operation power:800-1200W.

4. Flame On

Flame On: include prep. process and Flame process, display Current state in the status bar.

Flame preparation: setup the related parameters for the measurement, re-open the carrier gas and peristaltic pump.

- 5. Restore the default gas flow
  - The gas flow( plasma gas, carrier gas, supporting gas) is restored to the factory default value.



Fig.6.13

## 6.4 Analysis measurement

Analysis measurement consists of 3 parts, including standard measurement, blank measurement, sample measurement. Setup Data storage database, click *Select*, start *add/select* operation in the pop-up dialog(Figure 6.14). The selection for the result database facilitates the costumers to screen and classify the sample measurement data using the same method.



Fig.6.14

#### 6.4.1 Standard measurement

In the drop-down box, select Measure standard sample, click *Analysis standards* to start the standard measurement. The real-time measurement spectra, single measurement data and mean data are displayed in Fig.6.15.



Fig.6.15

#### 6.4.2 Standard curve

Click *Method -> Element -> Standard* to view the spectra linear relationship and related coefficients to determine if the standard curve is available. If not, check *Add* or *Remove* in Figure 6.16.



Fig.6.16

#### 6.4.3 Blank analysis

Click *Blank analysis* to start the blank measurements. The default blank name is BlankSample1, or manually edit in the text box. The blank can be measured repeatably, take the last blank measurement for calculation.

#### 6.4.4 Sample management

Before the sample measurement, first edit the sample info.(click *Analysis method-> Sample management*), as shown in Figure 6.17:

Sample Name	Supplier Unit	LotNo.	Weight (g)	Constant Volumn(ml)	Dilute Ratio	Cup ID	Uni t	
81 ank			1	1	1	1	mg/l	
2			1	1	1	2	mg/l	
3			1	1	1	3	mg/l	
í.			1	1	1	4	mg/l	n.
Sample01			1	1	1	7	mg/kg	
Sample02			1	1	1	8	mg/kg	
Sample03			1	1	1	9	mg/kg	C
SampleO4			1	1	1	10	mg/kg	
ample05			1	1	1	11	mg/kg	
Sample06			1	1	1	12	mg/kg	
Sample07			1	1	1	13	mg/kg	
Sample08			1	1	1	14	mg/kg	
Sample09			1	1	1	15	mg/kg	
21-10			1	1	1	16	mg/kg	

Fig.6.17

#### 6.4.5 Sample measurement

After editing, all the sample names are displayed in the *Sample name* drop-down box, select the sample to be measured, click *Analyze sample*, the measurement results will be displayed in the *Data Display Area*.

#### 6.4.6 Spectra calibration

After the measurement, click *Analysis measurement -> Spectra calibration* for the calibration of the characteristic lines.

#### 6.4.7 Qualitative Analysis

The application of optical measurement technology and computer technology in the spectral instruments makes the qualitative analysis of the spectrometers simpler and easier. Finding out three or more sensitive lines of this element in the spectrum indicates this element's presence.

Click Analysis measurement -> Qualitative Analysis to pop-up the interface as shown in Figure 6.18.

Qualitative Analysis	X
Select Storage Directory D:\Frameing Select Frame Name	
Run Full Frame	d ▶ Qualitative Analysis 🛛
Hethod Name: Francing V Sample F Blank Bom Full France V Sample F Blank 10 •	Open Zdit Group All Zdit Group Analysis Save
Spectrum Interference View Zoom In(+) Zoom Out(-) Und Gray False Color	Element Fercent (%)
X-Pos: Y-Pos: Intensity: Genss Fitted X-Pos: Y-Pos: Pk Intensity Nevelength: Order: Offset: X-Pos: X-Pos:	
Contrasts(+) Contrasts(-) Contrasts(Auto) 0k	
	CTD T 43.1. 🔺 🧰

Fig.6.18

1 Method name: Enter the name of the new method.

2 Select the directory: Click Select Directory, select the store location(except C drive) for the documents.

3 Full-size photo: check *Sample* or *Blank*,take a exposure photograph of the full-size 2D spectrum, and save it into the above directory.

4 Open: Check *Open sample* or *Blank*, select *Integration time* to open the corresponding full-size 2D spectrum(Figure 6.19).

5 Qualitative analysis: determine the presence of the elements based on 2D spectrum and display.



Fig. 6.19

#### 6.4.8 Full-size management

Click Analysis measurement -> Full-size management and edit in the prompted interface as shown in Figure 6.20.

🖉 Frame Name	Time	
Frameimg	2018-07-10 14:39:06	
		Delete
		Cancel

Fig.6.20

1.OK: Select the method, either click OK or double-click it to open it.

2.Delete: Select the method, click *Delete*.

3.Cancel: close the window.

# 6.5 View data and export report

Click Data Management -> Search results to pop-up the window below:

M.	ethod Name:	0122		· 1	Results Name:	1		•		
Ē	Sample Name				Accurate	Obscure				
	Date:	2019 -	02 - 12	·	2019 - 02 - 12	-				
<b></b>		[[								( <b>.</b>
	Query	Delete		xport Keport	Export E	xcel			Select All	Inverse
							ar. 14	nan		[a.a ]
-	Kesults	Element	Wavelength	Sample	Times	Content	Unit	RSD	Date Date Date 17.07	Selected
	1	Zn	213.856	Blank	3	0	mg/i	9.7	2019-01-22 14:47:27	
	1	INI .	251,604	Didrik.	3	0	nig/i	03.33	2019-01-22 14:47:27	
	-	MIL Cr	207.01	Didrik.	3	0	nig/i	65.25	2019-01-22 14:47:27	
	-	Cr.	207.710	Didrik.	2	0	mg/i	51 94	2019-01-22 14:47:28	
		Cu Pa	J24.754	Diditik	2	0	mail	52.00	2019-01-22 14:47:28	
	1	70	955.905	Didrik	2	1	mg/l	2.41	2019-01-22 14:47:28	
	1	LII	213.030	2	2	1	mg/i	2.41	2019-01-22 14:49:20	
	-	Mo	251.004	2	5	1	mail	2.51	2019-01-22 14:49:21	
	1	Cr	257.01	2	3		mail	2.2	2019-01-22 14-49-21	
	-	0	324 754	2	3	-	mail	2.63	2010-01-22 14:40:21	
	1	Ra	455 403	2	3	1	mg/l	2 44	2019-01-22 14:49:22	
	1	Zn	213,856	3	3	2	mal	1.75	2019-01-22 14:51:09	
	1	N	231,604	3	3	2	mal	1.49	2019-01-22 14:51:09	
	1	Mn	257.61	3	3	2	mal	1.45	2019-01-22 14:51:10	
	1	Cr	267.716	3	3	2	mal	1.61	2019-01-22 14:51:10	
	1	Cu	324.754	3	3	2	mg/l	1.21	2019-01-22 14:51:10	
	1	Ba	455.403	3	3	2	mg/l	1.14	2019-01-22 14:51:11	
	1	Zn	213.856	4	3	5	mg/l	2.97	2019-01-22 14:52:58	
	1	N	231.604	4	3	5	mg/l	2.3	2019-01-22 14:52:58	
	125			1.27				2.40	2010 01 22 14 52 50	

- Fig.6.21
- 1. Search:search the data by conditions;
- 2. Delete: Delete the invalid measurement data;
- 3. Export report: select the valid data, export the report (Figure 6.22), or print it out;
- 4. Export EXCEL: Save to Excel (Figure 6.23).

Report Preview							in the second	
B B K ← → H B S B B +								
Main Report								
		1	CP3000 A	nalysis	Report			
	Method:	20170328						
	Instrument Mo	de:ICP3000						
	Operator:	Admin						
	Sample Name:	Blank						
	LotNo.:							
	Supplier Unit	:						
	Test Date:	3/28/2017 2:3	24:14 PM					
	Element	<b>Vavelength</b>	Intensity	Content	Unit	RSD		
	Sn	189,989	2.194	0	ng/1	64.59		
	Sn	283.999	4.694	0	ng/1	30, 25		
	Sample Name:	CalibStdl						
	LotNo :							
	Sumplier Buik							
	Supprier onic							
	lest Date:	5/28/2011 2:2	10:20 FM	-	-			
	Element	wavelength	intensity	content	unit	RSD		
	Sn	189.989	45.778	1	ng/l	4.22		
			10.000	*	n6/1	7. 94		

Fig.6.22



Fig.6.23

## 6.6 Shutdown

#### 6.6.1 Flameout

After the analysis, rinse the sample for 5-10 min , click *Instrument Control -> Plasma Control* or to pop-up the window below. Click *Flameout*.

Carrier GF(L/min):		Flush Time	e (s):	
0. 70 🛓			30	
s Control				Plasma On
Flasma GF(L/min):	12.00	On	Off	
Auxiliary GF(L/min):	0.60	On	Off	Sampling Ready
mpling Control				
Carrier GF(L/min):	0. 70 🌲	On	Off	Plasma OFF
Peristaltic Pump:	2.0	On	Off	
Rotate Speed:	Normal	Fast		Default GF
wer Setting				
Remark (900-1200W) -		000	Satting	

Fig.6.24

#### 6.6.2 Turn off the instrument

Switch off HV power, close Ar valve until the torch cools down 1 min later; View Camera temperature by clicking *Instrument state*, turn off N<sub>2</sub> until the temperature returns to normal; finally turn off the host power,

AC stabilized power.

#### 6.7 Data management

#### 6.7.1 Data reprocessing

Data reprocessing is mainly to reprocess the measured data by setting the spectral peak position and width, as well as background position and width to improve the accuracy of the measurement results. The matrix matching is optimal for ICP spectrometers, but unrealistic in many cases. The differences in the components varied by samples or standard, continuous spectra or spectral tailing may lead to background interference, therefore, background correction is inevitable for a correct analysis results. As shown in Figure 6.25, the data reprocessing can be divided into 3 parts, i.e.spectra display, standard curve, data select & edit.Left-click to modify the position of peaks and background.



Fig.6.25

1. Peak Settings: Check *Peak*, move the curser onto the spectrum, setup the left/right peak by clicking at the right position(Figure 6.26);

2. Right Background: Check *Right BG*, move the cursor on the spectrum, left-click (click repeatably is allowed) at the right position to setup the right background (Figure 6.27);

3. Left Background: Check *Left BG*, move the cursor on the spectrum, left-click (click repeatably is allowed) at the right position to setup the left background;







Fig.6.27

4. After the settings for peak position and background are finished, click *Calculation* to recalculate the result.

5. Check the valid data and right-click, select *Report Preview* to export the report (Figure 6.28).Click *Data Management -> Search result* to view the data.

Calculatio	n						4
Method	Name:	20170328	-	Results	Name:	muhtar	-
Analysis	Line:	Sn:189.98	9 🔽	<+	->		
Peak	W	idth: 3	•	🔽 Smo	oothed		
📕 Left	BG W	idth: 2	•	🔽 Left	BG Effect	ively	
🔽 Right	t BG W	/idth: 2	•	🔽 Rigi	ht BG Effe	ctively	Excel
Select	all	Inverse	Ca	alculation	Sav	e	Clear
Selected	Color	Name		Left BG	Right I	3G	Pk Intensity
V		Calib	Std4 5	59	48.667	1	.071.778
1	1	Calib	Std5 6	67.667	51	2	2062.334
1		Calib	Std6 8	39	52.333	4	127.555
1		H20-1	. 6	35.833	50.5	2	245.889
		H20-2	: 6	38. 333	50.5	2	334.333
V		Н20-3	ι ε	39	51.167	2	358.333
V		0.1M}	CI 6	39	51	2	317.555
J		0.2M}	ICI 7	73	51.333	2	311.333
V		0.5M)	CI 1	71.333	49.667	2	286.889
1							•
San Nan	nple Ne	Waveleng	BG Intensity	Net Intensity	Content	Uni t	Content
H20-	-2	189.989	59. 417	2274.917	55.229	ppm	55, 229
H20-	-3	189.989	60.083	2298.25	55.811	ppm	55, 811
0. 1	NHCI	189.989	60	2257.556	54.796	ppm	54.796
0.2	NHCI	189.989	62.167	2249.167	54.586	ppm	54.586

Fig.6.28

#### 6.7.2 Tracing

The functions of Add, Delete, Edit for all lines are provided in the Periodic Table(Figure 6.29).

Li	Be			N-M	etal	T-M.	etal	0-M	etal			B	0	N 7	0	F	Ne
Na	Me			A-M	etal	A-E-N	letal	K-E-I	letal			Al	Si	P	S	C1	År
11	12				TUNE 3.	UL SOL						13	14	15	16	17	18
K 19	Ca 20	Se 21	Ti 22	У 23	Cr 24	Mn 25	Fe 26	Co 27	Ni 28	Cu 29	Zn 30	Ga 31	Ge 32	As 33	Se 34	Br 35	Kr 36
RЪ 37	Sr 38	Ү 39	Zr 40	МЪ 41	Mo 42	Те 43	Ru 44	Rh 45	Pd 46	Ag 47	Cd 48	In 49	Sn 50	SЪ 51	Te 52	I 53	Xe 54
Cs 55	Ba 56	*	Н£ 72	Та 73	¥ 74	Re 75	0s 76	Ir 77	Pt 78	Au 79	Нg 80	T1 81	РЪ 82	Bi 83	Ро 84	At 85	Rn 86
lx 37	Ra 88	** **															
L	antha *	n	La 57	Ce 58	Pr 59	Nd 60	Pm 61	Sm 62	Eu 63	Gd 64	ТЪ 65	Dy 66	Но 67	Er 68	Tm 69	¥Ъ 70	Lu 71
A	ctini **	d	Ac. 89	Th 90	Ра. 91	บ 92	Ир 93	Pu 94	Am 95	Cm 96	Bk 97	Cf 98					
.ine	Posi	tions	Pl	ease	inspi	re bla	unk so	lutio	n								



Fig.6.29

Left-click to enter the edit interface (Figure 6.30).

E	lement	Ionization State	Wavelength	Detection Limit	Intensity	Interference Elements	X-Pos	Y-Pos	X-Subarra	Y-Sub ar
Zn	<b>`</b>	II	202.548	0	1000000		809.25	563, 24	13	3
Zn	1	II	206.2	0	900000		808.48	538.79	13	3
Zn	ı	I	213.856	0	3000000		790.48	493.12	13	3
Zn	1	I	328.233	0	10000		674.87	201.73	13	3
Zn	1	I	330.259	0	25000		472.93	198.56	13	3
Zn	1	I	334.502	0	40000		646.43	194.71	13	3
Zn	1	I	472.216	0	18000		452.36	108.21	13	3
Zn	1	I	481.053	0	30000		715.23	106.49	13	3
Zn	1	I	481.053	0	30000		715.23	106.49	13	3

Fig.6.30

1.Add the analysis line: select the analysis line and click > to move it into the right area in the interface(Figure 6.31), click *OK*.

2.Delete analysis line: Select the analysis line, click *Delete*.

ul of the	e available ar	nalytical lin	ne		Selected			
Element	Wavelength	Intensity	State		Element	Wavelength	Intensity	State
Zn	171.325	150000	II		Zn	173.79	150000	II
Zn	171.576	180000	II					
Zn	173.561	240000	II	(				
Zn	173.689	180000	II					
Zn	174.712	225000	II					
Zn	176.219	240000	II	•				
Zn	177. 404	225000	II					
Zn	179.076	240000	II				_	
Zn	179.764	300000	II		4		2	*
Zn	181.105	300000	II				_	
	1					Ok	Cancel	

Fig.6.31

# 6.8 User Management

#### 6.8.1 Rights Management

As shown in Figure 6.32, user rights are divided into 3 levels: Administrator, Expert, Operator, setup the operating rights by different user level, this function is only owned by Admin.

ccount List: Administrator		
Operate	Display	Save
Carrier GF(L/min):		
Instrument Control-Scan Zero		
Analysis Method-Edit Standard		
Exit	<b>V</b>	Exit
Continue		
System Setting-Zero Param		
Analysis-Tracing		
Analysis	<b>V</b>	
Analysis Method-Basic Param		
Instrument Control-Online	V	
History Data	V	
Data Reprocessing-Calculate All	<b>V</b>	
Negative HV(v):	V	
Data Reprocessing-Export Partiti		



#### 6.8.2 User Management

Administrator (Admin) has the rights to add/delete users, change user rights/passwords, the other user levels only have the rights to change password, as shown in Figure 6.33.

User Name:	Admin
Authority:	Administrator
Admin Password:	
User Password:	
Confirm Password:	
Modify	Remove

#### 6.8.3 User switching

As shown in Figure 6.34, select the account you want to log in, enter the corresponding password and click *Login* to start the user switching.

Fig.6.33

Log In	×	J
6	ICP analysis software of Skyray Inc.	
	User Name: Operator	
	Password:	
	Log In Cancel	

Fig.6.34

# 6.9 Language

#### 6.9.1 Edit language

Add, delete, modify UI language in the interface(Figure 6.35).

Drawell International Technology Limited.

Id	CN	EN	Add Language
ICP.FrmProfi	设置	SET	Full Name:
ICP.FrmProfi	预览	PREVIEW	
ICP.FrmProfi	打印	PRINT	Short Name:
ICP. FrmProfi	自动定峰	AutoFixPeak	Add Same
ICP.FrmProfi	返回	Back	Add
ICP.FrmProfi	X轴:	X :	Modi fy
ICP.FrmProfi	Y轴:	¥ :	T
ICP.FrmProfi	至	to	Language lype: en
ICP.FrmProfi	至	to	
ICP.FrmProfi	设置	set	Remove Save
ICP.FrmProfi	设置	set	
ICP.FrmSelec	选择方法	Select Methods	
ICP.FrmSelec	全选	All	
ICP.FrmSelec	反选	Inverse	OK
ICP. FrmSelec	上一步	Previous	

Fig.6.35

#### 6.9.2 Language switching

All the above language shall be added as a sub-menu to the language menu in the main interface, which is used for language switching.

#### 6.10 Supportive functions

#### 6.10.1 System initialization

Click *System Settings -> System Initialization* to pop-up the dialog (Figure 6.36) for the initialization of the characteristics (this parameter is setup before leaving the factory).

Integration Time(s): 10 Run Full Frame Restore Line Position					
🖌 🖌	to Save			Open	
Spectrum	Structure	Correction of wavelengt	h Wavelength		

Fig.6.36

#### 6.10.2 Torch debug

Click *Instrument Control -> Torch debug* to pop-up the dialog as shown in Figure 6.37, Click *Up/Down/Left/Right* to adjust the flame observation height by controlling the torch motor, observe the intensity change trend in the spectrum(this parameter is setup before leaving the factory).



Note: all the supportive functions are operated exclusively by our technicians.

# 6.11 Help

#### 6.11.1 About

The software version, ownership are displayed below(Figure 6.38).

关于	x
	ICP Version: ICP3000_1.0.0_20140520_Beta Skyray Instrument 2014 Skyray
Kanthiosee Allettere	通定

Fig.6.38

# 6.12 Attached diagram

6.12.1 Software operation flowchart



# Chapter 7 Maintenance and repair

ICP-OES is capable of analyzing the samples with concentration from a few ppbs to several percent or even tens of percent ppbs.Without strict control of the analysis environment will inevitably lead to inaccuracy of the test; it's also required to conduct strict maintenance for the instrument in service, thus guarantee the analysis quality and prolong the instrument life.

#### 7.1 Lab glassware cleaning

Commonly used lab glasswares, such as beaker, flask, need to be cleaned before use. Polytetrafluoroethylene (PTFE) and borosilicate glassware shall be washed with soap or detergent prior to rinsed with water, then soaked in (1+1)HNO3 for 24 hours (or boiled),first wash with water,then deionized water for 3 times. Some glasswares are seriously contaminated by oil-stain, rinsed with solution(concentrated sulfuric acid compounded with the dichromic acid) and then thoroughly flushed with water.

#### 7.2 Use and maintenance

#### 7.2.1 Lab.requirements

Generally, the temperature needs to be fixed at  $20 \sim 25$  °C( $\leq \pm 1$  °C). Indoor humidity  $\leq 70\%$  (preferably 45%~60%, equipped with an air purifier).

#### 7.2.2 Power lines

In order to ensure the safe operation of this instrument, the power lines capacity must be sufficient to load the voltage,otherwise excess voltage will affect the life of the instrument. For the specific requirements of the circuits, please refer to *Instrument installation requirements*.

#### 7.2.3 Dust-proof

The exhaust fan exhausting the heat and toxic gases arising from the operation of the instrument leads to a pressure difference in the lab and outside, so outdoor dusty air flows into the lab through the slit of doors & windows because of the negative pressure in the lab and greatly accumulates in various parts of the instrument, leading to the firing of HV components or connectors, short circuit or current leakage of the circuit board ,wiring, sockets. Therefore, it's necessary to conduct frequent dust removal, especially the

computer ,electronic control circuits, high-frequency generator, monitor, printer and disk drives need to be regularly disassembled or opened to clean with a small brush, remove the dust in all parts by using a vacuum cleaner. For the negative HV lines of the photomultiplier, HV lines and ports of the computer monitor, use gauze stained with a little absolute alcohol to wipe out the accumulated coke and dust. The disk drives and printers shall be dropped with a little instrument oil at their mechanical moving parts after the dust is cleared.Remove the printer head, sweep with a soft brush and wipe with a cloth to prevent the pinhole clogging by paper scrap,then adjust the print pressure according to the instruction. The dust removal is usually conducted by the electronics, repairing or computer technicians; the operator or administrator having no knowledge of the instrument or electronics should not touch the instrument to avoid accidents;The dust removal shall be conducted after shutdown the instrument and power off.

#### 7.2.4 Nebulizer Maintenance

Nebulizer needs good maintenance for it's the most vulnerable key part in the injection system. It's necessary to conduct a regular cleaning, especially after measurement of the high salt solution, salt accumulation in the torch nozzle leads to poor aerosol channel, decreased intensity, increased reflection power. The coke or dust accumulation on the torch will affect the stability of plasma torch and the reflection power, therefore, it shall be cleaned regularly with acid and water, finally rinsed with absolute ethanol and dried, and keep injection system and torch clean.

#### 7.2.5 Minimize On/Off times

Be prepared before the measurement. Do not turn on the instrument frequently in short time for it may cause damage to the instrument, when turn on the instrument, the instant current is much higher than that of normal, the instant pulse is likely to cause damage to the power tube, vacuum capacitor and other chips.

#### 7.2.6 Other precautions

- 1. Injection system pre-check;
- 2. The injection system check and clean after the measurement;
- 3. Frequent clean of the waste barrel;
- 4. Torch/nebulizer/spray chamber clean;
- 5. Regular renewal of the cooling water;

6. Before each start-up, please note: When plasma gas < 1Mpa and firing for only an hour, recommend to replace the cylinder;

7. Do not freely adjust the plasma gas flow meter and pressure gauge after the ignition, otherwise it will damage the quartz tube;

8. After the ignition, the capillary must be placed into the solution, the capillary leaving out of the liquid (exposing to the air)shall not >10 s when replace the solution, otherwise lead to flameout;

9. If find the quartz tube dirty, please clean it in time;

10. Remove the torch tube, nebulizer and spray chamber with great care (quartz glassware is fragile).

# **Chapter 8** Failure analysis and troubleshooting

The following is used for our technicians offering remote assistance . If any problems occur, please refer to the following and contact us. We always hope to solve the problems for you ASAP.

- 1. Software online failure
- ① Check whether the net-cables contact properly;
- 2 Check whether the instrument is powered on.
- 2. Ignition failure

① Check whether the cooling tank is open, the water pressure is normal (no less than 0.1MPa); this is the reason for not hearing the relay "Pick-up" sound in the ignition process.

2 Check the software tip. If indicate insufficient airflow, please check the gas path or replace the cylinders if necessary.

- ③ Check whether the firing gun connects to the torch.
- ④ Check whether the injection tube is inserted into the solution to form a liquid seal.

3. Click Injection, prepare for flameout

- 1 No liquid seal;
- 2 Excessive gas pressure(0.2MPa-0.3MPa), please check the cylinder pressure within this range.
- 4. Injection tube bubbles

This is caused by the fusing of the torch center channel due to short of the supporting gas/carrier gas or no gas at all. In such cases, it is necessary to replace the torch and ensure the gas path normal.