

# Chemistry Lab Cookbook

## ICP Maintenance v 210.0

**Instrument:**

Jobin Yvon Ultra-Ace 2000  
 ODP #: 05669  
 Model #: TX-TF722

**Website:**

<http://www.jyemission.com/>

**Software:**

Jobin Yvon Proprietary, V 5.10

**Documentation Available:**

Document Name/Title/Number	Source	Type	Associated Instrument(s)	Associated Measurement(s)
ICP Installation/Setup Binder	Jobin Yvon	hardware	ICP	elemental analysis
ICP Instrument Manuals Binder (Final Control Report; User Manual; Image Navigator User Manual; Sampler AS421 User's Manual; Schematics)	Jobin Yvon	hardware, software	ICP	elemental analysis
ICP Reprints Binder	scientific journals	data	ICP	elemental analysis
ICP Software V5.1 Binder (User Manual)	Jobin Yvon	software	ICP	elemental analysis
ICP Standards Cert./MSDS Binder	misc.	data	ICP	elemental analysis
ICP Technotes, Software Notes, Concepts, Spreadsheets, Etc. Binder	in-house	hardware, software, data, reprints	ICP	elemental analysis
JY Service Manual	Jobin Yvon	hardware	ICP	elemental analysis

## Maintenance (v 210.0)

### 1. Cleaning/Aligning the Torch

Clean the torch each week (minimum every two weeks). Dismount it and put the quartz tubes in Aqua Rega (1/3 conc. HNO<sub>3</sub>, 2/3 conc. HCl) for example on Friday. On Monday, clean the tubes with DI, then dry it.

Replace top and bottom O-rings at least two times a year. If the O-rings are cracked, the flame will bounce and may go off. If there is a leak at the torch assembly, the flame will go off. When there is a leak, more often than none it is at the torch. Also check that the teflon at the bottom of the torch assembly is still there.

In reassembling the torch, make sure that there is 2.7cm from the top of the injector tip to the teflon disk. The inner tube should be pushed in all the way and there should be 2-3mm between the top of the inner tube and the coil. The flame will be proper if the bullet is 2-3mm above the coil. If you burn a lot of quartz outer tube, the torch is not centered and the flame will glow orange.

### 2. Cleaning the Chamber

Clean it each week (minimum every two weeks). Dismount the small orings and replace them if you prick them. Put inside 3 pills of KOH (crushed), add DI in the chamber and shake during 20 s (there will be a hot reaction so be carefull!!). You will see a film in the chamber. Afterwards, dispose of the liquid and clean the torch with acetone and rinse again with DI. Do not clean with KOH pills if you analyze traces of K (ppb).

### 3. Cleaning the Nebulizer

Disconnect entirely, put in in Aqua regia (1/3 HNO<sub>3</sub>, 2/3 HCl - caution: generates lots of gas and heat) each week.

### 4. Cleaning the Grating Mount Gear and Worm-Gear

The large gear underneath the grating assembly and its worm-gear should be cleaned at least once a year by either the chem tech or ET. To access these parts, remove the blue "JY 2000 Ultrace" panel and lower the panel that covers the three pressure gauges and temperature control switches. Remove this inner panel to access the internal parts of the ICP. Once this panel is removed, a box-shaped aluminum dust cover can be seen towards the center of the ICP. This dust cover must be removed to access the gear assemblies. Wipe the gear with a kim-wipe that has been sprayed with WD-40 or some other light oil. The motor can be turned in the software to ensure that all of the gear is cleaned and oiled. Clean the worm gear the same as the motor gear. Reassemble the pieces in reverse order.

Note: There should be some play in the grating. One should not move the position of the grating or mirrors. If the mirrors have been moved: a little, you will see flattened peaks; a lot, you will see no peaks.

### 5. Cleaning the Lens

The lens should be cleaned if it appears that the intensities are decreasing across the spectrum (sensitivity is bad and background high). Remove the blue "JY 2000 Ultrace" panel, then remove the torch assembly. Once the torch

assembly is gone, remove the black plastic air line that goes to the aluminum cylinder with quartz tube. To the right of this cylinder is a matte aluminum cylinder that spans the torch box and the inner ICP box. Loosen the four thumbscrews (two on each side) and slide out the aluminum cylinder with quartz tube. Slide the matte aluminum cylinder all the way to the right (the left set of thumb screws can be removed). This will expose the lens assembly. Removal of the lens requires a special knurled knob that fits this assembly. Wear nitrile gloves when handling the lens. Once the lens is removed, carefully blow dirt away with Argon and then clean it with trace-metal grade acetone – NEVER USE WATER as water will destroy the lens crystal. To clean, use a Q tip and start with the center then work your way to outside limits. Carefully but the lens back into its holder, making sure it is flat and then put the lens holder back into the cylinder making sure it is centered. The lens assembly should only be finger tight (finish by tightening with nail). Reassemble the pieces in reverse order.

## **6. Adjusting or Changing Grating Mount Spring**

The spring mount assembly is underneath the grating mount gear. Following the above procedure, remove all the pieces necessary to have access to the gear. The spring mount is attached below and directly in the middle of the grating mount gear. Remove the three screws from the bottom of the assembly to have access to the spring. Replace the spring if needed. The spring can be tightened and adjusted in it slips by loosening the two (?) screws on the side of the spring assemble and turning it with a small screwdriver in the adjustment holes (also on the side of the assembly). There should be only the smallest amount of play in the assembly when it is adjusted properly.

## **Special Instructions**

### **1. Tip for using JY Windows Software**

#### TIP1:

When switching from the Windows version ( Analysis v5.01 ) to the DOS version ( Analysis v4.09 ):  
you need to refresh the port by  
clicking on 'shortcut to system'  
selecting device manager  
clicking on refresh to refresh the serial port

#### TIP2:

The software 'Plasma Control' is used to start the plasma with the DOS version ( Analysis v4.09). For the Windows version ( Analysis v5.01 ), you must click on 'Analysis v5.01' and let this software open the Plasma Control software.

### **2. Avoiding Blockage of Grating**

After a power outage or after a lost communication with the Spectralink if you want to avoid being stuck on the maximum switch with your grating, you can :  
reset the spectralink ( switch it OFF and ON );  
launch the DOS version ( Analysis v4.09 );  
select 'shift F3' and then Esc to detect the four boards and communicate with the spectralink;  
select 'shift F1' in main screen ( no zero order search );

select F6 (Diagnostics): your grating will be lost and the position will be something like -387nm  
select 'shift F1' and type 0 to reset the position and then F2 -50 to clear the switch ( if needed )  
then you can just go back to the main screen and select 'SHIFT F2' to make a zero order .

### 3. Calculation of Angle Correction Coefficient

To change the Angle Correction Coefficient, in the main menu of the JY DOS software, select shift F3 (Instrument Set Up), then Instrument and ESC. The next screen will show all the communication boards and an instrument Configuration window. In that window, you can enter a new value in the "Correction Included Angle" field. To calculate the new one, proceed as follows:

l of C should be at 193.01, on Leg 192 C was found at l=193.049  
l of Ar should be at 772.421, on Leg 192 Ar was found at l=772.512

Angle Correction Coefficient=  $X_{moy} \times 1.00061$  (default coefficient) = 1.0001665364, where

$X_{moy} = X_c + X_{ar}/2 = 1.00010553$ , and

$X_c = l \text{ of C found} / l \text{ of C theoretical} = 1.0000935$

$X_{ar} = l \text{ of Ar found} / l \text{ of Ar theoretical} = 1.00011781$

Note: If the reference offset is greater than 0.1, the angle coefficient needs to be recalculated.

### 4. Optimization of Sensitivities

The value of Pb BEC gives in general a good idea about the sensitivity of the instrument.

BEC is defined as the Background Equivalent Concentration and corresponds to the Intercept on the Calibration Curve for Pb.

Two different methods were given to test for Pb BEC

1. Jerome Thomas suggested to do the following to find the value of Pb BEC.

create a Pb method with the following parameters l=220.353, slit 20/16, mode 5, inc min 0.0002 nm, no background correction, calibration with 2 standards (0-5ppm);

calibrate with a 0 and 5ppm, look at the intercept of the calibration curve. With the parameters listed above, the BEC (intercept) should be 0.234 ppm.

2. Paul Thellier suggested to do the following:

create a Pb method with following parameters l = 220.353, slit 20/16, mode 2, time=0.5s, inc = 0.002 nm, calculation on 3 pts, no background correction, calibration with 2 standards (0-5ppm);

do an autosearch on 5ppm Pb solution, note peak intensity and background intensity (signal to noise ratio (s/b));

$BEC = (\text{Background Intensity} / \text{Signal Intensity} - \text{Background Intensity}) \times \text{conc. Pb}$  (i.e here 5ppm), assuming that BEC for Pb is 0.15 ppm, the signal to noise ratio should be:

$s/b = \text{conc} / BEC$

For example, for a 5ppm solution s/b will be 5/.15, i.e around 33, for a 10ppm solution, the s/b will be 10/.15, i.e around 66 (if that ratio is below 15, clean the torch and if it does not improve clean the lens).

On 192 we did several autosearch with 10ppm and found a s/b always around 65 (1644/35) , looks good!

To improve sensitivity, Paul suggested two strategies:

- change sheath flow;
- change torch position.

This optimization is done using the Manual Operation option:

first, autosearch on the 5ppm Pb solution, it will give you the current position of Pb (I), e.g 220.377nm vs theoretical 220.537;

escape to the main menu and select F6 Diagnostic, this will open the Manual Operation for Sequential window; select F2, enter your target position (e.g. 220.377) and in Channel 1 (signal on detector), enter a value around 10000;

play with Integration Time (F3) or Voltage (F6), then go to F4 graphics to see the plot of Signal (# of counts) vs Time. For example, with integration time of 1 and 600 volts, we got a channel 1 count of 13000;

to optimize, change the sheath gas (G1) flow first. G1 should be between 0.15-0.4 (<0.15 will clog the injector). While in graphics mode, adjust the flow to see if the signal improves, then check again the signal to noise ratio. It should be higher if there was improvement. On 192, we got a good signal at Time=1 and Channel= 15000 with a s/b of  $2391/31 = 77$  (must be higher than s/b of theoretical BEC). You can also play with the position of the torch assembly(up and down adjustment. Do not touch the screw at the bottom of the torch).

Other BEC for following elements are (in ppm):

- As = 0.11
- Pb = 0.15
- Co = 0.025
- Cr = 0.025
- Cu = 0.045
- Al = 0.3

## 5. Calculating Exhaust Vent Flow

The ICP should have a flow through the exhaust vent of between 200 and 300 cubic feet per minute (cfm). The vent air-speed can be checked inside the vent hood with a hand-held anemometer and on Leg 192 it gave a value between 2.4 and 3.5 cubic meters per second (m/sec) through an 11-cm tube. To calculate cfm, one needs to know the air-speed and the area of the pipe.

To calculate the area of the 11-cm exhaust pipe:

$$P(r^2) = 3.1416(0.11-m)^2 = (3.1416)(0.012) = 0.038-m^2.$$

To calculate the air-speed from m<sup>3</sup>/s to m<sup>3</sup>/mn:

A flow of 2.4-m<sup>3</sup>/sec equals 144-m<sup>3</sup>/mn

To calculate flow:

$$(144-m^3/mn)(0.038-m^2) = 5.472 m^3/mn$$

Conversion factor for m<sup>3</sup>/mn to cfm = 35.314

$$\text{So: } (5.472 m^3/mn)( 35.314) = \mathbf{193 cfm}$$

## Document Information:

Version Number, Original: 192

Revision History:

210: Chris Bennight

Signature (Department Supervisor)