

4. Be sure that you have selected **Startup enabled** and set the date and time.

**D. To exit the software:**

In the **File** menu, click on **Exit** to leave the software.

## **10.0 MAINTENANCE**

Routine maintenance is required to maintain the system in proper working condition and to ensure the highest possible level of performance. In particular, the torch glassware and RF coil must be well cared for.

### **10.1 Daily Checks:**

The following items should be checked daily, before igniting the plasma:

#### **10.1.1 Argon Supply:**

Make sure that an adequate supply of argon is available and connected to the system. Argon output pressure: 70-120 psig.

#### **10.1.2 Purge Gas:**

Make sure that an adequate supply of nitrogen purge gas is available and connected to the system. Nitrogen output pressure: 29-120 psig.

#### **10.1.3 Shear Gas Supply:**

The shear gas used in compressed air; Shear Gas output pressure: minimum 60 psig.

#### **10.1.4 Chiller:**

Cooling water pressure: 30-80 psig.

#### **10.1.5 Torch and RF Coil:**

Visually inspect the torch, glassware, and aerosol injector tube. The glassware should be clean with no traces of deposits or signs of melting.

#### **10.1.6 Nebulizer:**

The nebulizer must be checked for clogging, and the sample capillary tubing must be clean and in good condition.

#### **10.1.7 Drain:**

Be sure the drain tubing is firmly attached to the spray chamber and liquid flows smoothly through the pump. Keep the drain tubing clear of debris.

**10.1.8 Peristaltic Pump:**

Inspect the pump tubing daily and replace if necessary. Remove the tubing from the peristaltic pump when it is not in use to prevent flat spots from forming. Check each pump roller to ensure that it is not binding. Binding can cause poor precision.

**10.1.9 Flush:**

At the end of each working day, it is recommended that you flush out the system for five minutes with the torch on. Use 1%HNO<sub>3</sub>.

**10.1.10 Background Equivalent Correction (BEC) test:**

Serves as an indication of instrument sensitivity.

**10.1.11 Precision Test:**

Indicative of a problem with the sample introduction system.

**10.2 Weekly Maintenance:**

**10.2.1 Waste Reservoir:**

Empty the drain bottle and rinse with DI water.

**10.2.2 Sodium Bullet Test:**

This test allows you to visualize the sample flow in the plasma, so that you can check that the sample introduction system is working correctly.

**10.2.3 Waste Pump Tubing:**

Check all pump tubing weekly and replace as necessary.

**10.3 Monthly Maintenance:**

The frequency of maintenance required will be determined by the amount of use the system receives, the cleanliness of the environment and the number and nature of samples being analyzed. These factors are the responsibility of the user, and a suitable maintenance schedule will be implemented.

**10.3.1 Nebulizer:**

To maintain optimum performance the nebulizer and drain must be kept clean and free-flowing. Check the nebulizer spray pattern with deionized water. You should see an aerosol of uniform droplets. Clean or replace the nebulizer tips as necessary.

To check the performance of the nebulizer, aspirate a standard and note the

intensity reading. If the intensity is significantly lower than it should be, first check the following:

- ! The pump tubing is in good condition (has no flat spots).
- ! The sample capillary is in good condition (has no kinks) and is clean.
- ! The horizontal plasma viewing position is set properly.
- ! Flush out the nebulizer by aspirating for several minutes with 1% HNO<sub>3</sub>.  
Recheck the intensity of the standard. If the intensity is still too low, you should check the nebulizer spray pattern.

**A. Removing the Nebulizer/End Cap:**

1. Turn off the plasma if it is on.
2. Open the front door to the sample compartment.
3. Loosen, but do not remove, the two knurled screws in the nebulizer end cap. Support the spray chamber with one hand as you twist the end cap and end cap ring, together, off the spray chamber.
4. To check the spray pattern, leave the sample capillary tubing and the nebulizer argon tubing connected.

**B. Checking the Spray Pattern:**

1. Set the nebulizer argon flow to 1.0 L/min.
2. Place the sample capillary tubing in a beaker of DI water start pump at a rate of 1.0 mL/min.
3. Check the spray pattern on a paper towel. You should see a fine, even spray of uniform sample droplets.

Note: If the spray pattern is not even or the nebulizer is sputtering, the nebulizer tips should be inspected for clogging, and cleaned or replaced as necessary.

**C. Disassembling The Nebulizer:**

Disassemble the nebulizer only if it is necessary to clean or replace the sample tips. The Cross-Flow nebulizer has two Gem Tips. The sample tip is a clear sapphire and the argon tip is a red ruby. These tips are not interchangeable. First remove one GemTip, clean or replace it, then remove the other.

1. Remove the nebulizer/end cap assembly from the spray chamber.
2. Disconnect the sample capillary tubing and the nebulizer argon tubing.
3. Inspect the inside of the end cap. If deposits are found, clean the cap using a dilute acid solution.
4. Remove the sample tips one at a time. See Figure 10.1.
5. Inspect the sample tip. Clean the tip using soap and water or 1% HNO<sub>3</sub>. You may need to replace the tips; be sure to replace a clear GemTip for sample inlet

and the red GemTip for argon inlet.

6. The O-rings should be cleaned with soap and water, or replaced if necessary.

7. To reassemble the nebulizer, push the sample tip in as far as possible.

Examine the two GenTips in the end cap. These tips should almost butt up to each other at an angle of 90 degrees.

***Before replacing the end cap:***

- # Check the end cap O-ring for nicks or cracks. If you need to replace the O-ring, remove the two knurled screws and the end cap ring. When placing the end cap ring back on the end cap, check that the small circular indentations are on the side facing you.
- # Connect the sample capillary tubing and the nebulizer argon tubing to the nebulizer and check the spray pattern.

**To replace the end cap:**

1. Moisten the O-ring to make replacing the end cap easier. Make sure that the two knurled screws that hold the end cap and the ring together are loose.
2. With one hand supporting the far end of the spray chamber, push and twist the nebulizer/end cap onto the spray chamber so that the end cap seats firmly onto the spray chamber.
3. Alternately tighten the two knurled screws until the end cap ring is pulled snug against the end cap.
4. If you have not already done so, connect the sample capillary tubing and nebulizer argon tubing.

**10.3.2 Spray Chamber:**

Check the spray chamber periodically for leaks around the nebulizer end cap and drain fitting. Examine the end cap O-ring for cracking and wear and replace if necessary. Deposits and plated out metals can accumulate in the spray chamber, usually aspirating a strong acid will clean out these deposits. If not, remove the spray chamber and clean it in a 1% $\text{HNO}_3$  acid bath.

**A. Remove the Spray Chamber:**

1. Place the sample tubing in deionized water and turn on the pump to flush out the spray chamber. Remove the sample tubing from the deionized water and continue to run the pump a few minutes until the spray chamber is fully drained.
2. Disconnect the spray chamber drain tubing from the pump drain tubing. Be careful not to have liquid spill out of the disconnected drain tubing. Spilled liquid should be cleaned up immediately.
3. Disconnect the sample tubing from the nebulizer inlet.

4. Disconnect the nebulizer argon fitting from the gas fitting on the mount.
5. Gently twist the spray chamber off the base of the torch adapter.

### 10.3.3 Delete Out Dated Data Files

Delete data files for sample batches which have been recorded in SQL\*LIMS.

## 10.4 Semi-annual Maintenance:

### 10.4.1 The Plasma Torch Assembly and RF Coil:

Clean the torch components periodically to remove accumulated deposits. Replace any cracked or worn O-rings on the torch assembly.

Note: Always wear gloves when handling the torch assembly.

The RF coil must be kept clean to prevent arcing across the coils. Inspect the RF coil for any deformations or carbon buildup. Contract a Perkin-Elmer Service Engineer to replace the RF coil if there are any signs of pitting. Pitting causes weakness in the coil which can result in a hole in the coil.

#### A. Disconnect the Torch Tubing:

1. Disconnect the spray chamber drain tubing from the pump drain tubing.
2. Disconnect the sample tubing from the nebulizer inlet.
3. Disconnect the nebulizer argon tube from the nebulizer.
4. Remove the argon gas fitting from the torch inlets.

Note: Wait five minutes after turning off the plasma before you begin these maintenance procedures.

#### B. Removing The Torch Assembly:

By moving the torch on its mount, you will have repositioned the relative location of the torch to the coil. Doing this causes variations in the analytical performance. Therefore, it will be necessary to readjust the position of the torch, in addition you will have to rerun the View-X procedure.

1. Loosen the thumbscrew below the torch block (see Figure 10.2). This will release the tension on the torch mount.
2. The torch assembly is mounted on a plastic dovetail block that slides out. Slide the assembly out of the block, being careful to maneuver the torch glassware out of the coil.

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Caution: When the torch glassware is removed from the coil, the bonnet will be on the coil with nothing securing it. Be careful not to knock it off and break it.

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3. Gently twist the spray chamber and nebulizer assembly off of the torch adapter.

**C. Disassemble the Torch Glassware:**

1. Loosen the three thumbscrews holding the torch clamp. See Figure 10.3.
2. Remove the three thumbscrews on the torch adapter.
3. Carefully pull and twist the torch out of the torch block and clamp.
4. Slide the torch adapter out of the torch block.
5. Pull the injector tube out of the torch adapter.

**D. Replace the Torch Glassware:**

1. Inspect the silicone O-rings on the torch adapter for cracks and wear. If the O-rings need to be removed, carefully remove them from the adapter and replace. See Figure 10.4.
2. Grasp the top of the glassware and slowly push and twist the torch into the torch block. Do not hold onto the glass inlets. See Figure 10.5.
3. Notice the decal mark on the torch glassware. Line up the decal on the glass with the matching mark on the torch clamp. The glass inlets should face to the front of the instrument. Continue to carefully push and twist the torch all the way in to the torch block until the glassware is seated in the block and the decal marks are lined up.
4. Push the injector tube onto the torch adapter until is fully seated.
5. Moisten the O-rings on the torch adapter. Then push the torch adapter into the torch block until it seats. Screw in the three small thumbscrews.
6. If the glassware is set correctly, tighten the three large thumbscrews on the torch clamp. Tighten them evenly. From a top view of the torch, the injector should be centered inside the torch glassware.

**E. Adjust the Torch Injector Tube:**

If the injector tube is protruding out beyond the inner quartz tube, an adjustment must be made. See Figure 10.6.

1. Loosen the three bigger thumbscrews on the torch clamp.
2. Make the adjustments as necessary. The injector must be 0.0 to 0.5 mm inside the inner quartz tube. See Figure 10.6.2.
3. If the injector is sticking out beyond the inner quartz tube (and you are sure it is seated completely), drop in a O-ring against the torch adapter before sliding in the torch glassware. This will push out the torch enough so that the injector is

more inside the rim.

4. From a head-on view of the torch, the injector should be centered inside the torch glassware. See Figure 10.6.3.
5. Tighten the three larger thumbscrews on the torch clamp.

**F. Assemble Torch to Torch Mount:**

1. Slide the plastic dovetail mount into the AV $\equiv$  on the torch mount. At the same time, carefully slide the glassware through the RF coil and bonnet.
2. Note that the inner quartz tube should be approximately 1.0 mm to the left of the first turn of the coil.
3. Tighten the screw underneath the torch mount to secure the dovetail mount in place.

**G. Adjusting the Position of the Torch:**

There are three adjustments available for the torch position.

To center the torch in the RF coil by adjusting the **vertical position**:

1. Loosen the securing screw on the dovetail block.
2. Slide the dovetail block up or down until the torch is centered in the RF coil.
3. Tighten the securing screw. See Figure 10.7.

To center the torch in the RF coil by adjusting the **horizontal position**:

1. Using a wrench, loosen the two securing screws on the dovetail block.
2. Slide the dovetail block back and forth until the torch is centered in the RF coil.
3. Tighten the two securing screws. See Figure 10.8.

To adjust the position of the torch relative to the **RF coil**:

1. Loosen the securing screw on the dovetail block. Loosen the lower rear thumbscrew securing the spray chamber support bracket.
2. Adjust the torch position so that the inner quartz tube is approximately 1 mm to the left of the first turn of the RF coil. *Note: The injector should be slightly to the left of the inner quartz tube.*
3. Tighten the securing screw on the dovetail block. Tighten the lower rear thumbscrew to secure the spray chamber support bracket. See Figure 10.9.

**H. Lateral and X/Y Torch View Optimization:**

The Lateral (radial view) and X/Y (axial view) torch view optimization procedure is used to set the plasma viewing position of the spectrometer entrance optics for the highest signal intensity.

Perform the X/Y torch view optimization procedure when:

- X the instrument is first installed
- X the torch is removed or replaced
- X the RF coil is replaced

To perform the Lateral and X/Y torch view optimization:

1. Make sure that the plasma is lit and the system is warmed up.
2. Open the Spectrometer Control window by selecting **Spectrometer Control** from the **Tools** menu.
3. To view the Mn spectra during the optimize procedure, you must open the Spectra Display window before performing the next step.
4. If you are using axial viewing, click on **Optimize X/Y**.  
-or-  
If you are using radial viewing, click on **Optimize Lateral**.  
The Spectrometer Alignment dialog is displayed.
5. Aspirate a 10 mg/L Mn solution.
6. Click on **OK**.

There is an automatic read delay of 60 seconds. The torch view optimization will take several minutes. Because the optimization procedure must run uninterrupted, the software intentionally keeps the user from doing anything other than canceling the procedure while it is taking place. When the procedure is complete, the Spectrometer Alignment dialog will disappear and the **Align** on the **Optimize** button will be dimmed.

7. On the Optima 3000 Dual View instrument, the optimization needs to be performed for both viewing modes. Change to the other viewing modes. Change to the other viewing mode using the command in the System menu, for example, Change to Radial Viewing (if you were previously using Axial Viewing). Then repeat Steps 4 through 6.

## 10.5 General System Maintenance:

Regularly check the following:

**10.5.1 Air filters:** You should check the air filter at the rear of the Controller at periodic intervals (about every three months) and if necessary change it when it is loaded with dust. If the filters are not too damaged, rinse with DI H2O and dry with compressed air.

### A. Changing the Air Filter:

1. Switch off the Controller.
2. Prize up and remove the filter cover.

3. Check the filter and replace it if necessary.
4. Press the cover firmly back into place.

**10.5.2 Pneumatic filters:**

Check the argon dryer filter, and argon and nitrogen filters for moisture.

**10.5.3 Change Radial View Window:**

Change radial view window as needed.

**10.5.4 Chiller Maintenance:**

Turn off ICP and chiller power supplies. Drain chiller and refill with distilled water only. Clean filter on chiller by using vacuum to remove residue, rinse with deionized water and dry with compressed air. Chiller maintenance should be performed every three months, or more often as needed.

**10.6 Annual Maintenance:**

Annually check the following:

**10.6.1 Inter-element Correction (IEC):**

Annually perform inter-element corrections. See section IEC SOP for instructions.

**10.7 Annual Preventive Maintenance Visit:**

At least annually, preventive maintenance will be performed by a factory trained service engineer from Perkin Elmer.

**10.7.1 Preventive Maintenance Visit:**

The following maintenance will be performed:

- Optics cleaned and inspected
- Sensitivity checked
- Wavelength checked
- Mechanical assembly cleaned
- Mn BEC checked
- Torch changed
- Injector changed
- Nebulizer tips changed
- View aligned
- Purge window cleaned and inspected

## 11.0 TROUBLESHOOTING

### 11.1 Performance Problems:

#### 11.1.1 Checking the Sample Introduction System:

Many performance problems (such as poor precision or loss of signal) can be traced to the setup and condition of the sample introduction system.

The sodium bullet test is also a good way of visually checking the sample flow in the plasma and, therefore, indicates problems in sample introduction.

##### **A. Pump and Pump Tubing:**

*Is the tubing overstretched or does it have flat spots, leaks, or discoloration?*  
Install new tubing, stretching it gently beforehand.

*Is the flow of liquid irregular?*

Adjust the tension on the sample and drain tubing, one at a time, using deionized water as follows:

1. Make sure the sample tubing is centered in the channel, then place the clamp around the tubing and swing back the cam lever.
2. Place the sample capillary (SAMPLE IN side) in a container of deionized water. Disconnect the nebulizer capillary from the SAMPLE OUT side and lead the pump tubing to an empty container.
3. Set the pump speed to 1.5 ML/min and start the pump.
4. If droplets are flowing out of the SAMPLE OUT side of the tubing, loosen the adjustment screw (turn counter-clockwise) until no droplets form.
5. Tighten the adjustment screw slowly (turn clockwise) until droplets just start forming at the end of the SAMPLE OUT side of the sample tubing.
6. Tighten the adjustment screw until you see a smooth flow without bubbles.
7. Turn off the pump. The tension is now correctly set. No bubbles should be seen in the sample tubing.
8. Reconnect the SAMPLE OUT side of the tubing to the nebulizer capillary.

##### **Adjusting the Drain Tubing:**

1. Make sure the drain tubing is centered in the channel without disturbing the sample tubing, then place the clamp around the drain tubing and swing back the cam lever.
2. Loosen the adjustment screw (turn counter-clockwise) for the drain tubing channel so that the cam lever can be easily opened and closed with little resistance.

3. Set the pump speed to 1.5 mL/min and start the pump.
4. Tighten the adjustment screw (turn clockwise) up to three turns so that moving bubbles are visible in the drain tubing. It may take up to one minute to see bubbles if the spray chamber is dry.
5. Loosen the adjustment screw until the bubbles stop moving.
6. Tighten the adjustment screw slowly until the bubbles just start moving, again.
7. Tighten the adjustment screw until the bubbles move in a segmented flow.
8. Turn off the pump. The tension is now correctly set.

*Do the pump rollers bind or not roll easily?*

Periodically inspect the pump rollers to make sure they are clean and move freely. Clean the exterior of the pump with a cloth moistened with water. The pump head on the Perkin-Elmer pump can be removed and cleaned if necessary.

**Removing the Pump Head:**

1. Remove the pump tubing.
2. Unscrew the large screw in the center of the pump head.
3. Lift the pump head off. To clean the pump head, use water or a mild solvent and dry thoroughly.
4. Return the pump head to the pump. Orient the pump head by aligning the notch on the bottom of the pump head with the pin on the shaft. The pump head must click in this slot for a proper fit. Replace the washer and tighten the screw.

**B. Capillary Tubing:**

*Are the nebulizer and sample capillary tubing properly connected? Is the tubing discolored or clogged? Is tubing crimped or pinched?*

If the capillary tubing is not in good condition, replace it.

**C. Nebulizer and Spray Chamber:**

*Is there leakage around the end cap? Is there leakage from the spray chamber drain? Is fluid accumulating in the torch?*

Make sure the end cap is on securely. If you cannot get a secure fit, the end cap O-ring may need to be replaced. Check the drain fitting and tubing to be sure the spray chamber is being properly drained. Check that the waste is being pumped in the proper direction.

*Check the nebulizer spray pattern: run deionized water for several minutes, then remove the nebulizer end cap. Is the nebulizer spray sputtering or uneven?*

If the spray pattern is uneven, the nebulizer tips may be clogged or worn. Disassemble the nebulizer and clean or replace the GemTips.

*Are there deposits inside the end cap or spray chamber?*  
Clean out deposits.

**C. Torch Assembly:**

*Are the torch fittings for the gas inlet secure?*  
Finger tighten the fittings. Do not over tighten them.

*Is the quartz torch cloudy or dirty?*  
Check for deposits particularly if running samples with high dissolved solids.

*Is the injector dirty?*  
The injector may be clogged and require cleaning? If running samples with high dissolved solids, check for deposits.

**D. Purge Window:**

*Is the purge window cloudy or dirty?*  
Rinse with deionized water and dry with a soft cloth.

**E. Bonnet:**

*Is the bonnet cloudy or dirty?*  
Remove the bonnet and clean in a 20% nitric acid solution. Rinse well.

**11.2 Plasma Ignition and Stability Problems:**

**A. Plasma Ignition Problems:**

Possible reasons for unsuccessful ignition follow. Correct the problem, if possible, and then try to ignite the plasma again.

- # **System not ready:** If the System Ready indicator on the front of the instrument is not lit, or if you are getting a message indicating that the system is not ready see Section 11.3.
- # **Status icons not ready:** Check the status icon on the System Monitor window. In the **System** menu, click on **System Monitor**. If any icon is red, refer to Section 11.3.
- # **Ignitor:** Open the doors to the sample compartment. Check the ignitor cable is

plugged in. Also, check the ignitor rod.

- # **Exhaust Vent:** Be certain that your exhaust vent is operating properly and is not blocked.
- # **Torch Connections:** Verify that all gas fittings to the torch are finger-tight. Leakage of air into any part of the torch, nebulizer or spray chamber will cause ignition problems. The torch should be clean and in good condition. Note: Do not use tools to tighten the torch fittings as the torch gas inlets may be damaged.
- # **RF Coil:** Check the RF coil for condensation, particularly in labs with high humidity.
- # **Torch Glassware:** Check the condition of the torch glassware. If it is cloudy or dirty, clean it with 5-20% nitric acid solution. See Section 10.4.
- # **Drain:** Check the drain tubing for flat spots and replace the tubing if necessary. Check that the drain fitting on the spray chamber drain is secure. Be sure that the pump is properly draining the spray chamber and that the drain liquid is not backing up into the spray chamber or building up in the torch.
- # **Argon:** Check that the argon supply is on. Check that the pressure is 70-120 psig.
- # **Injector:** Check that the injector is not clogged. You will need to disassemble the torch as described in Section 10.4. In addition, try pumping solutions with the Nebulizer, Auxiliary, and Plasma gas flows on for approximately two minutes.
- # **Nebulizer End Cap:** Check that it is tightly secured to the spray chamber.
- # **Sample Capillary:** Check that it is attached to the nebulizer.

#### **B. Plasma Stability Problems:**

If periodic pulsations of the torch are observed:

- ! Poor sample drainage may be the problem. Make sure that waste drainage is not backing up into the spray chamber and that the waste is draining properly.
- ! Check the condition of the peristaltic pump tubing. If it is flattened, stretched, or damaged by abrasion, replace the tubing.

- ! Air leaks may be causing the pulsation. Finger-tighten the connections on the torch. Make sure the nebulizer/end cap assembly fits tightly to the spray chamber.

### 11.3 Operating Status Problems:

The following table describes problems associated with the instrument status.

<b>Problem</b>	<b>Possible Cause(s)</b>	<b>Corrective Action</b>
The spectrometer icon is red	The spectrometer has been shut down due to a fatal error.	Check the Message Log for error messages that describe the cause.
The water flow icon is red	Cooling water may not be flowing properly.	Check that the chiller is filled and working properly. The shear gas shares the interlock with the cooling water, check that the shear gas is on.
The spectrometer temperature icon is yellow or red	The spectrometer temperature is not within the required range.	If the icon is yellow, monitor the temperature in the Instrument Diagnostic window. If the icon is red, the spectrometer will automatically shut down. Check the water chiller.
The interlock icon is red	It is normal for the Interlock icon to appear in red briefly, but once interlocks are	

checked and found satisfactory, the icon become gray. If it remains red, see below.

The doors to the sample compartment may not be closed securely.

If either the argon or water flow icon is also red, this indicates an interlock problem.

Make sure that the doors are properly closed. Wait for error message to clear.

Check the argon supply.

The Ar (argon) icon is red

Check that the argon supply is on. Check that the pressure is 70-120 psig. The N<sub>2</sub> (nitrogen) icon is red

The System Power Indicator Light does not light up

The System Ready Indicator Light does not light up

The argon pressure is not in the required range.

The nitrogen pressure is not in the required range.

Power to the spectrometer and/or RF generator may be off.

The spectrometer may not have completed the initialization routine.

All interlocks may not be satisfied.

Check that the nitrogen supply is on. Check that the pressure is 30-120 psig.

Check that the Main Instrument Switch and the RF Generator Standby Switch are on.

The software will indicate how long the initialization will take.

One of the status icons in the System Monitor will be red, and/or an error message

indicating the problem will appear in the Message Log.

The computer may be off.

Turn the computer on. The computer must be on for IEEE communication.

The IEEE cables may be disconnected.

Check that the IEEE cable is connected.

The System Ready Indicator Light does not light up

The RF generator or spectrometer may need to be reset.

Turn off the RF Generator Standby switch, wait a few seconds, then turn it back on. It may also be necessary to turn off the Main Instrument switch.

A message is displayed in the Message Log indicating that the system is not ready

If the status of the instrument is OK, this message may indicate a communication problem between system components.

Double-check the chiller, argon, nitrogen supplies.

Open and close the front door to the sample compartment to try resetting the door interlock.

Reset the instrument. If this does not work, try exiting and restarting the software.

If the message indicates that the RF generator is not ready, try turning off the RF Generator Standby switch,, then turning it back on.

#### 11.4 Performance Problems:

Problem	Possible Cause(s)	Corrective Action
RSD (Relative	The sample introduction	Check all components.

Standard Deviation) greater than 1%

system may not be set up correctly or may require maintenance.

Does the sample have a high viscosity or high percentage of dissolved solids?

Try increasing the integration time.

For high dissolved solids, try the cone spray nebulizer.

Try running other samples with normal characteristics to determine if the sample matrix is really the problem.

Look for carryover- check if concentrations of replicates increase from one to the next.

Increase the Read Delay to flush the sample introduction system completely. Increase the rinse time.

RF power and/or nebulizer argon flow may require adjustments.

Adjust as necessary. Check recommended settings.

Cannot obtain suggested BEC value

You may not be comparing the appropriate BEC value for the wavelength you are using.

Recheck the BEC value in the Wavelength Table. In the **Tools** menu, click on **Wavelength Table**.

The torch position may need adjustment.

Refer to Section 10.4 to the torch adjustment procedures.

The plasma viewing position may need to be optimized.

See X/Y Torch view optimization.

Results erroneously high

Standard solutions may have deteriorated or may have been improperly made.

Restandardize with a proven standard.

	Background emission may be present.	Use background correction.
Results erroneously low	Standard solutions may have been improperly made.	Rerun proven standards.
	Blank solution may be contaminated.	Remake the blank solution.
	Is the concentration for the second standard lower than that for the first?	Increase the Read Delay to flush the sample introduction system completely.
	Is background correction being used?	Check the placement of background correction points.
Undetectable emission signal	The sample introduction system may not be set up correctly, or may require maintenance.	Check all components.
	The shutter may be closed.	Check the shutter position set in the software.
Memory effects	Spray chamber may not be drained properly, or may need cleaning.	Check drain fittings and drain tubing. Clean the spray chamber if necessary.
Cannot get suggested detection limits	The sample introduction system may not be set up correctly, or may require maintenance.	
Check all components.	The BEC may be high.	Run the BEC test.

Look for carryover - check if concentrations for the sample replicates increase from one to the next.

Increase the Read Delay and flush the sample into the system completely.

The integration time may be too short.

Increase the minimum and maximum times for integration.

You may not be comparing the appropriate value for the wavelength you are using.

Confirm that you are using the appropriate wavelength and recheck the value.

### 11.5 Pump Problems:

**Problem**

Pump does not start

**Possible Cause(s)**

The pump cable may have become disconnected.

**Corrective Action**

Check that the cable is connected to the back pump.

The software may not be configured for the pump you are using.

In the **System** menu, **Configure Pump**.

The pins on the pump cable connector may be broken.

Check the connector. If pins are broken, order replacement cable.

Pump rollers stick

A spill may have occurred or the pump head may be worn and may need replacement.

Clean the pump head, replace if necessary.

Liquid is not flowing freely

The tension on the pump tubing is too high or low.

Use the adjustment screw to adjust the tension.

Tubing may not be installed correctly in the channel.

Reinstall tubing.

	Tubing may be worn.	Replace tubing.
Pump tubing is stretched on one end and slack on the other end	The tension on the pump tubing is too tight.	Loosen the adjustment screw.
	Rollers may be stuck.	Check the rollers in the pump head to see if they roll freely.
	Tubing may be worn.	Replace tubing.
Pump produces excessive heat	Fan on the pump may be blocked.	Move the pump to give the fan adequate clearance.
Pump makes excessive noise	A mechanical problem may have occurred.	Call a Perkin-Elmer service engineer.

**11.6 Autosampler Problems:**

<b>Problem</b>	<b>Possible Cause(s)</b>	<b>Corrective Action</b>
Autosampler does not respond	There may be a GPIB communication problem.	Use the Instrument Diagnostic window to reset the autosampler.
	The software may not be configured for the autosampler you are using.	In the <b>System</b> menu, click on <b>Configure Autosampler</b> .
	The DIP switches may be set incorrectly.	Check the DIP switch settings.
	One of the cables may have been disconnected or the autosampler may have been	Check the power cable and the cable connecting the autosampler to the

turned off.

computer. Make sure the power is on.

Autosampler does not go to the correct location

The software may not be configured for the correct autosampler tray.

In the **System** menu, click on **Configure Autosampler**. In the dialog, select the appropriate tray.

## 12.0 WASTE DISPOSAL

All waste disposal procedures will follow the Water Quality Laboratory Chemical Hygiene Policy and Procedure Manual.

The following applies for sample disposal procedures:

- A) Discard all remaining analyzed samples in an acid sink.
- B) All croncial containers will be disposed of in trash.

## 13.0 EMPLOYEE TRAINING

- 13.1 When analysts are first introduced to this procedure, a current copy of the SOP for this instrument will be provided. Prior to performing this procedure the analyst will study this SOP in detail to assure complete acquaintance with the procedure and be tested before starting any independent analyses.
- 13.2 The analyst will also be provided with Policy and Procedure Manual and Chemical Hygiene Policy, all safety practices will be reviewed prior to analysis.
- 13.3 The analyst is also encouraged to review and study all reference manuals for procedure. See section 14.0 for a list of manuals available.
- 13.4 The analyst will be under strict supervision by the Supervisor for at least the first six months. During these six months a in-house training and hands on training program will be provided.
- 13.5 After such time a in-house proficiency will be given to analyst in training.
- 13.6 A written test will be given in reference to SOP and other Manual procedures to make certain that the analyst in training has a full understanding of all procedures.
- 13.7 Upon completion of the written exam and in-house proficiency the analyst will be allowed to follow the daily requirements independently with little supervision.
- 13.8 For City of Albuquerque Water Quality Training Program see Attachment 13.1.

## 14.0 REFERENCE MANUALS

- 14.1 Perkin Elmer ICP-Emission Spectrometry Optima 3000 Hardware Guide
- 14.2 Perkin Elmer ICP-Emission Spectrometry Optima 3000 DV Supplement
- 14.3 Perkin Elmer ICP-Emission Spectrometry IPC WinLab Software Guide
- 14.4 Perkin Elmer ICP-Emission Spectrometry Installing ICP WinLab on Windows 95
- 14.5 Perkin Elmer AS-91 Autosampler User=s Guide
- 14.6 City of Albuquerque Water Quality Laboratory Policy and Procedure Manual
- 14.7 City of Albuquerque Water Quality Chemical Hygiene Policy



