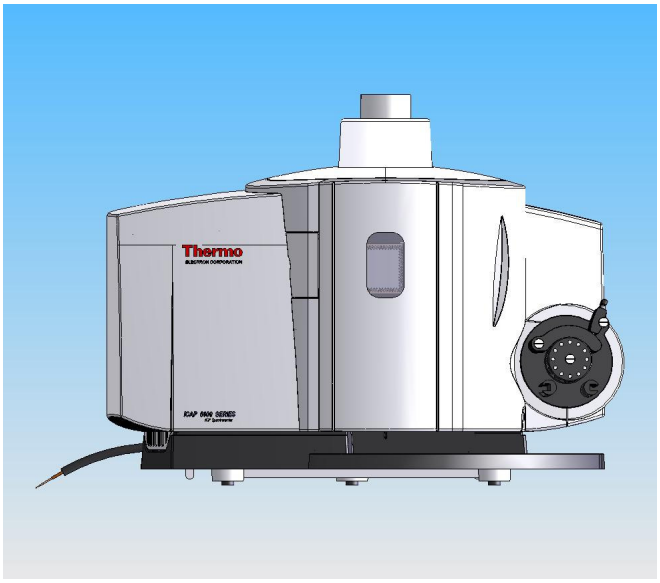
iCAP 6000 Series ICP-OES Spectrometer

Customer Training and Maintenance Manual



Version 1 - March 2009

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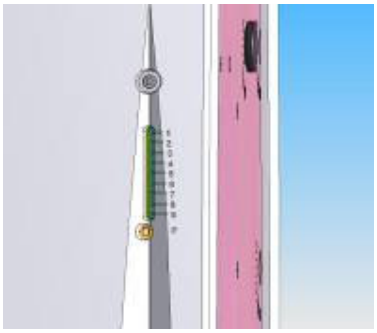
1 Introduction

The iCAP 6000 is designed to be constantly powered up and the optical system continuously purged.
The instrument is powered via an on/off switch at the rear of the left side.

Figure 1 – iCAP 6000 Power On/Off

1.1 Check Led indicators



On the rear right hand side of instrument there are row of Led's which indicate, when power on and the status of the instrument.
Once the chiller has been turned on and reached it set temperature led's 1-7 should be on and led 8 should be flashing
Led 9 is for engineer fast purge for gas to optical tank and should only be on if fast purge is required and turned off when analysing samples.

1.2 Preparing the System for Use

If the gas supplies have been switched off, the optical components should be purged for at least one hour before powering the instrument.

If the instrument is switched off, allow at least two hours after restoring power to thermally stabilise the instrument before the chiller is turned on.

A blank sample should be aspirated for ten minutes to allow the instrument to fully stabilise before analysis.

1.3 Instrument Shut-down

After an analysis is finished a blank sample should be aspirated for five minutes to insure the sample introduction part have been rinsed of sample. To remove the blank sample deionised or distilled water should be aspirated for a further minute.
When organic solvent based samples are being analysed the final rinse should be the pure solvent. Air should be aspirated for two minutes to remove organic vapours.
After completing the above the plasma should be turned off. The optical components will move to a parked position after about thirty seconds.
Allow five minutes after switching off the instrument, or accessories, before disconnecting the electrical power or other supplies.

2 Sample Introduction Glassware Assembly

Warning: Appropriate care and safety procedures should be followed to avoid breaking any glassware and causing injury to the operator. Broken glassware should be handled with appropriate care.

2.1 Torch Assembly

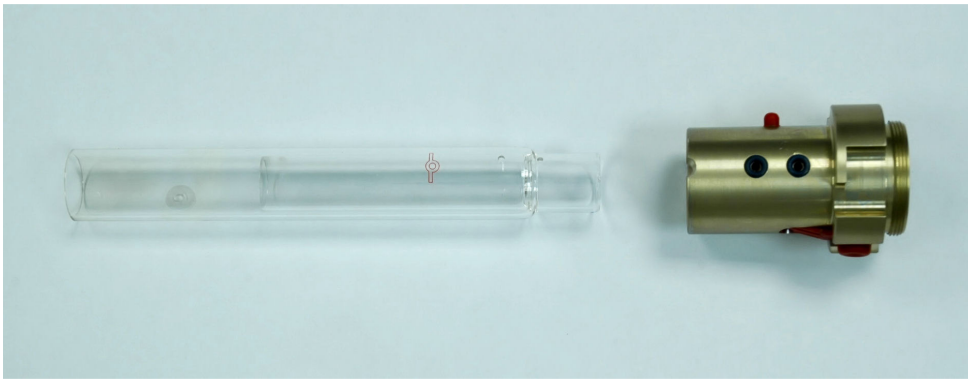


Figure 1 Duo Torch Assembly – Metal Torch Mount

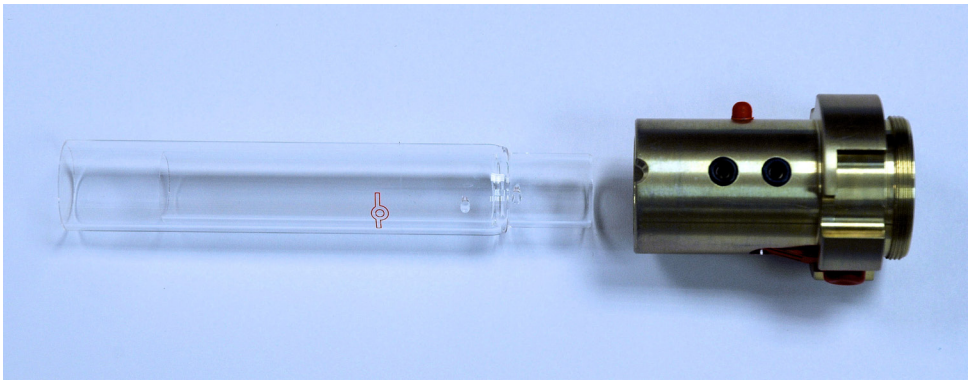


Figure 2 Radial Torch Assembly – Metal Torch Mount

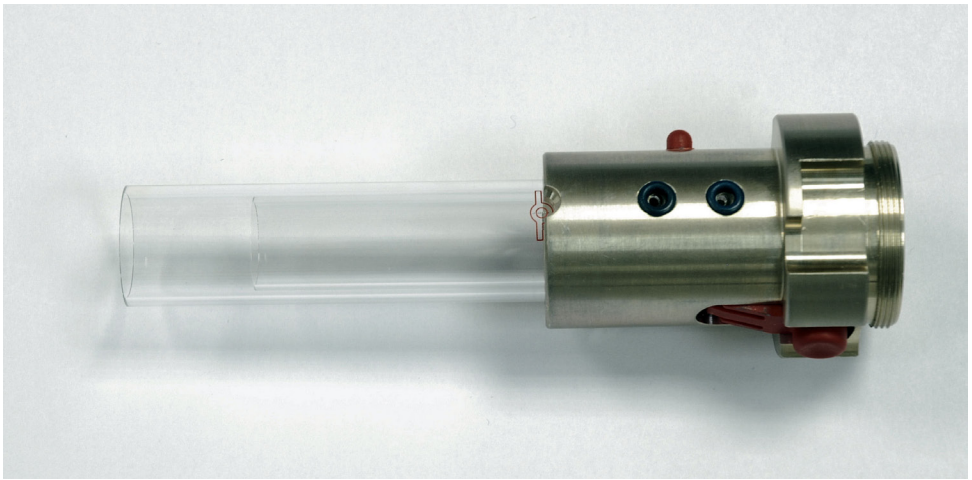
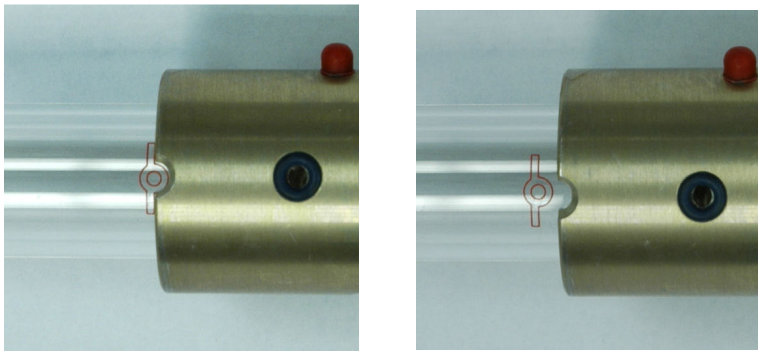


Figure 3 Torch Assembly – torch body

The quartz body of the torch should be pushed fully into the metal torch mount.

The O-rings in the metal torch mount (internally and externally) should be inspected and replaced if any wear, or damage, is visible.
The quartz body of the torch should be pushed into the metal torch mount. Ensure the torch body is pushed **fully** into the metal torch mount. The marked symbol on the quartz torch is used to locate the radial view hole correctly and should touch the metal body.



Correct Incorrect

Figure 4 Torch Assembly – torch body

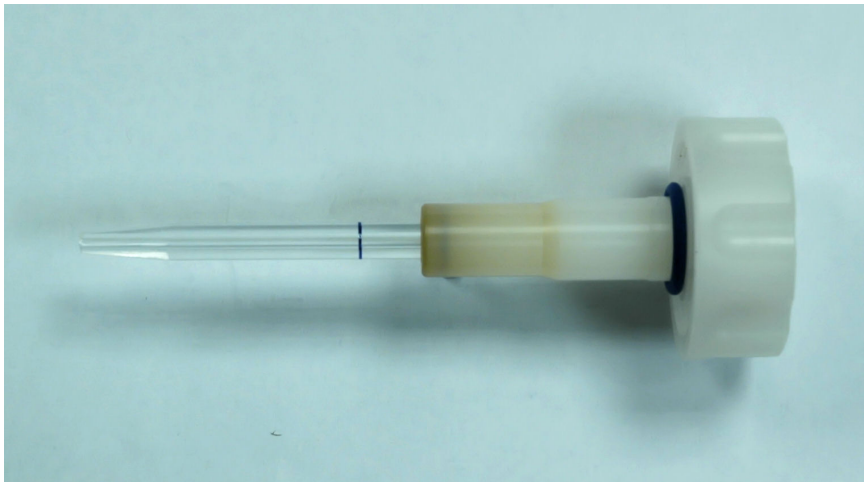


Figure 5 Torch Assembly – centre tube

Insert the centre tube (ground glass joint) **fully** into the centre tube holder.

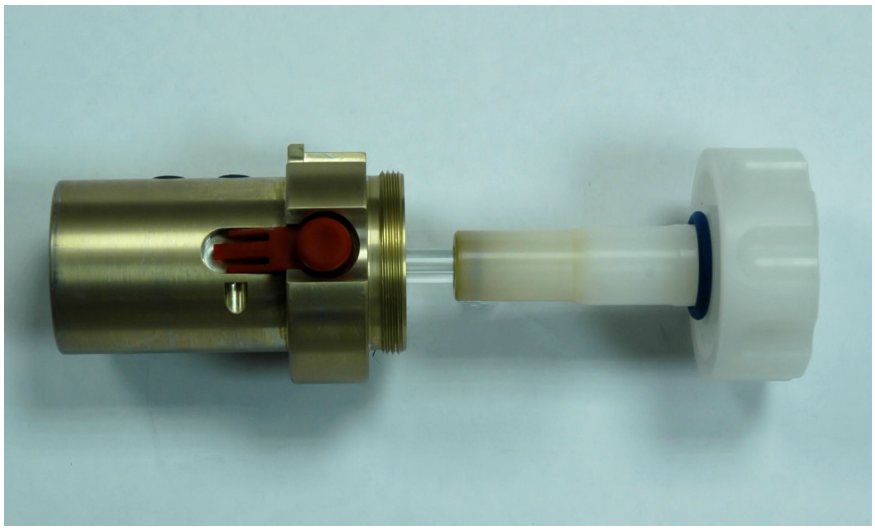


Figure 6 Torch Assembly – centre tube insertion

Insert the centre tube assembly into the metal holder.

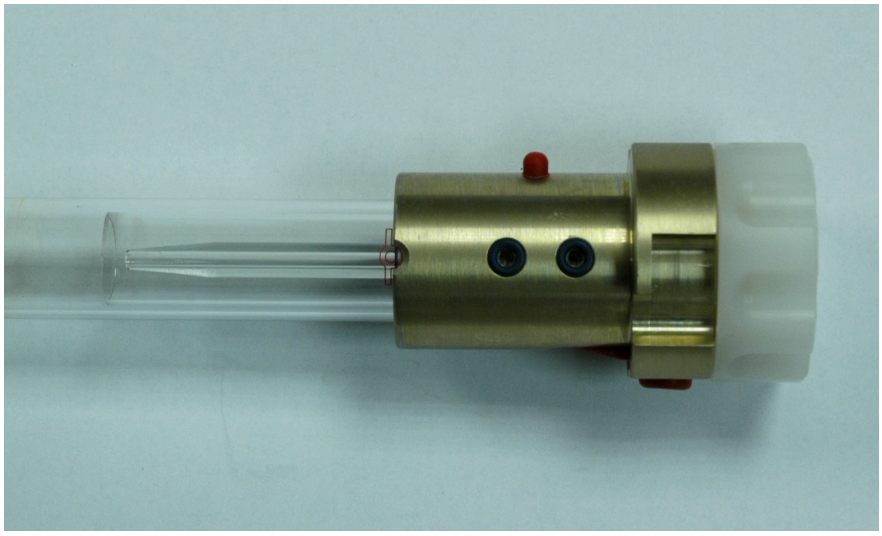


Figure 7 Torch Assembly – centre tube insertion

Screw the centre tube assembly into the metal holder.

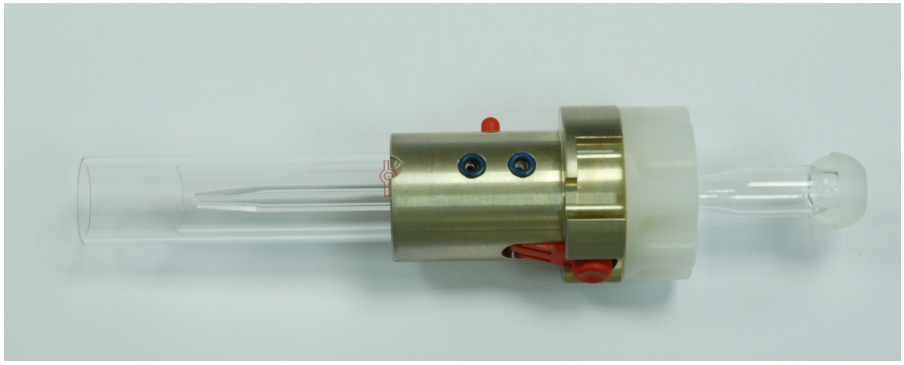


Figure 8 Torch Assembly –spray chamber adaptor

Insert the spray chamber adaptor (ground glass joint) into the rear of the centre tube holder

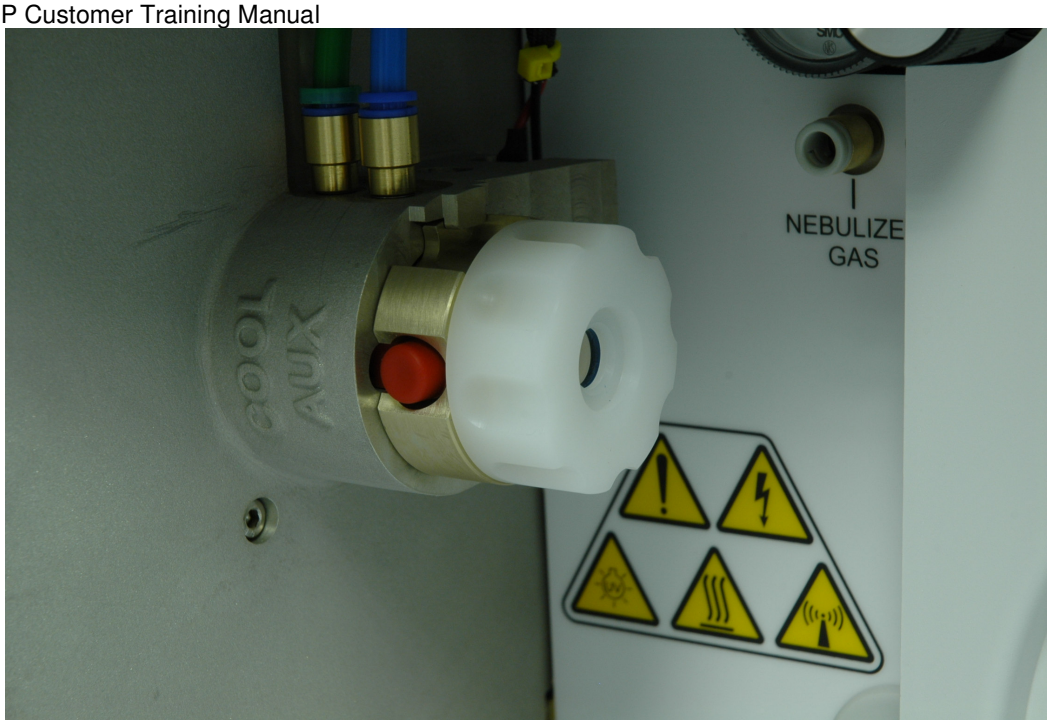


Figure 9 Torch assembly holder

Make sure metal holder is turned and locked in position.

Figure 10 – Torch Holder

Push the torch assembly up into the holder and twist to lock.

Spray Chamber assembly

Start by connecting the drain tubing as figured below

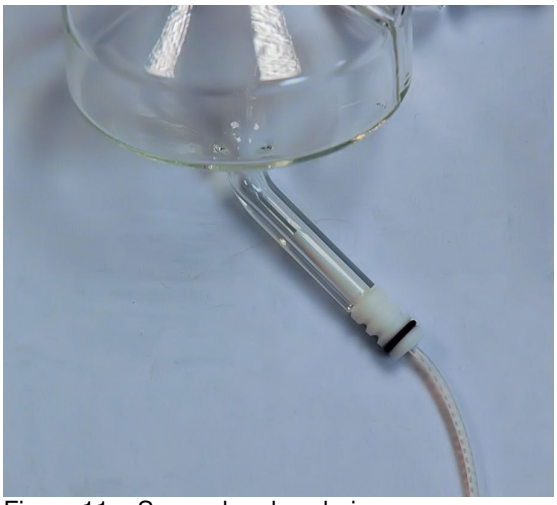


Figure 11 – Spray chamber drain

Insert drain tube connector until it meets the main chamber.

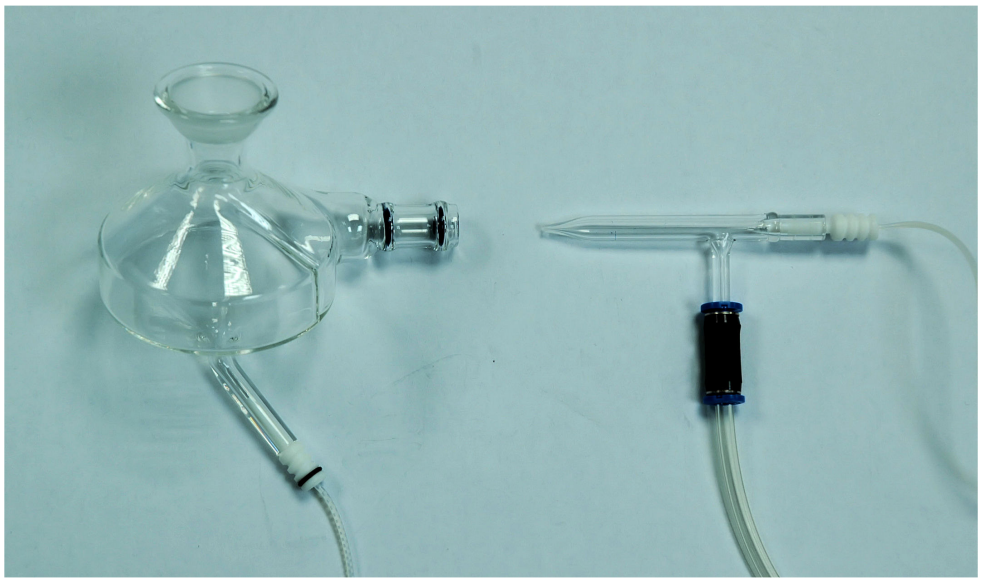


Figure 12 – Nebuliser to Spray Chamber

The O-rings in the spray chamber should be inspected and replaced if any wear, or damage, is visible. Push the Teflon insert with the attached sample tubing (supplied with the nebuliser) into the rear of the nebuliser as far as possible without exerting undue pressure.
Using a twisting motion insert the nebuliser into the spray chamber so that the collar is a tight fit. The collar will set the insertion depth and aid reproducibility of results.

2.2 Final assembly

Duo Spray chamber

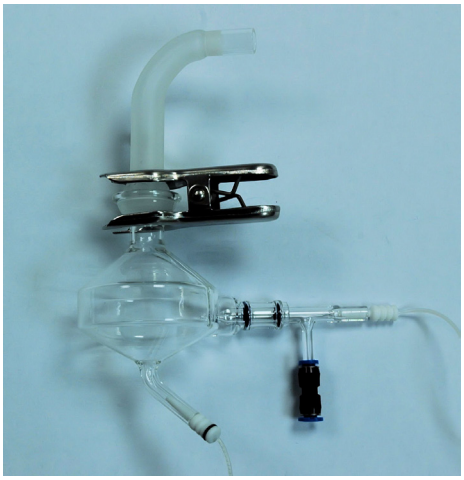


Figure 13 – Duo Spray chamber assembly

Attach the swan neck fitting to the Spray chamber with the fitting clamp provided. The swan neck provided with the instrument is specially designed to prevent UV radiation from escaping from the torch box.

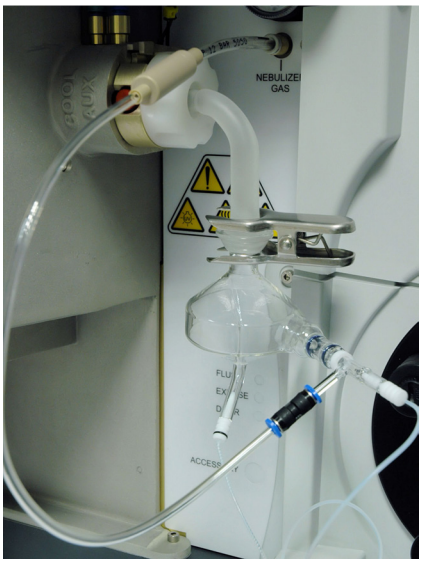


Figure 14 – Duo Spray Chamber fitted

Insert swan neck fitting into torch assembly holder as far as it will go, and connect up the Nebuliser gas supply to the push-fit fitting.

Warning: It is extremely important that the correct Thermo part is used for the swan neck. In addition, systems interlocks on the torch holder and elsewhere are there for safety reasons and must not be bypassed. Operators could be exposed to dangerous UV and radio frequency radiation if alternate parts are used for the swan neck.

For Radial
Connect spray chamber and nebuliser assembly to the torch assembly as figure below

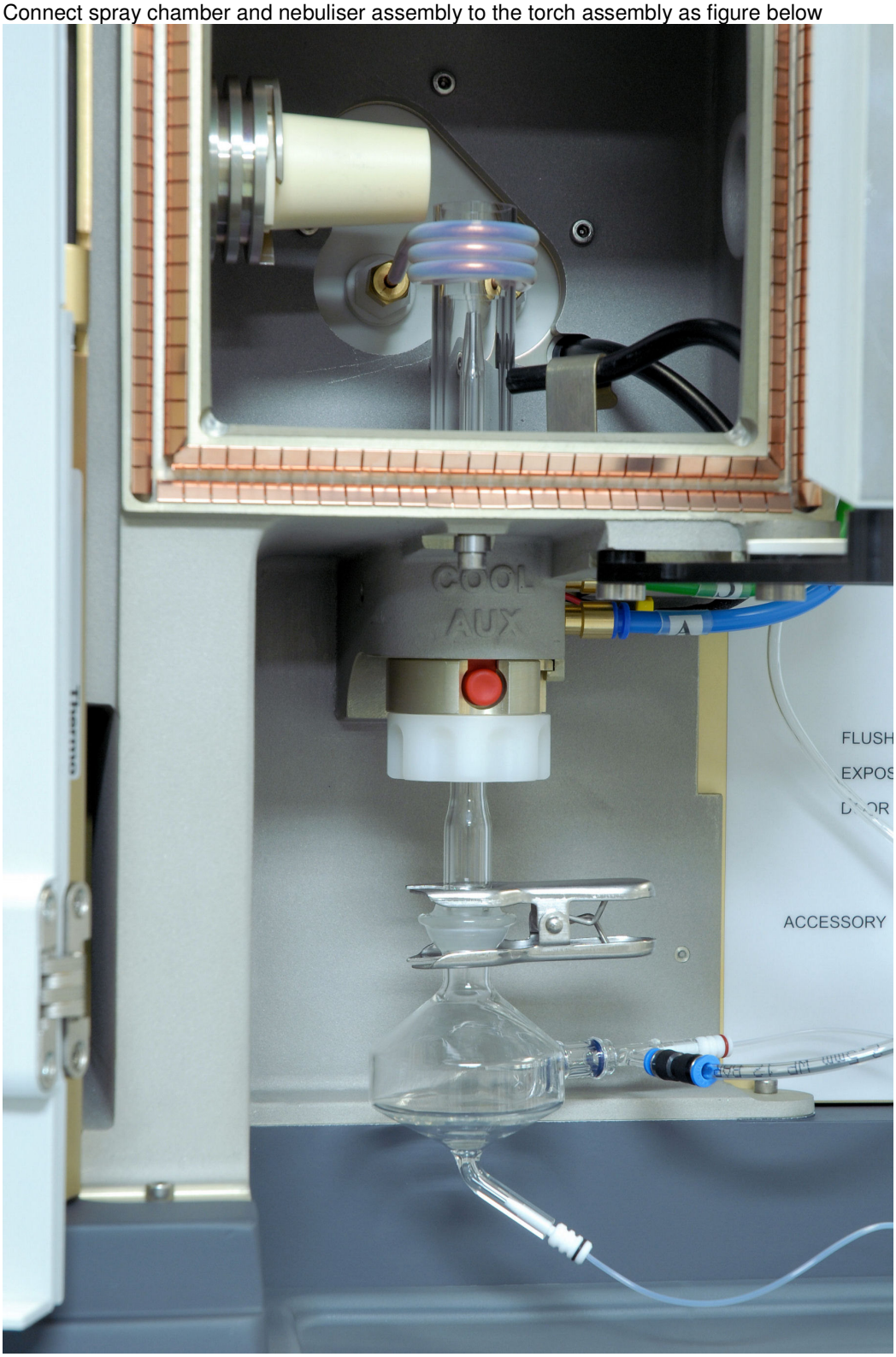


Figure 15 – Radial Sample Introduction setup
The torch compartment door must be closed before use.

After assembly of the sample introduction system and prior to ignition of the plasma a check should be made for correct assembly :-

Make sure the torch is fully rotated and locked in place.
Make sure the centre tube holder is fully rotated and locked into the torch.
Make sure the spray chamber connector is fully pushed into the torch body.
Make sure the spray chamber is tightly connected to ball the joint.
Problems in any of these areas may cause air leaks or disruption of the gas flows making the plasma difficult to ignite and may cause damage to the torch.

2.3 Attaching sample and drain tubing

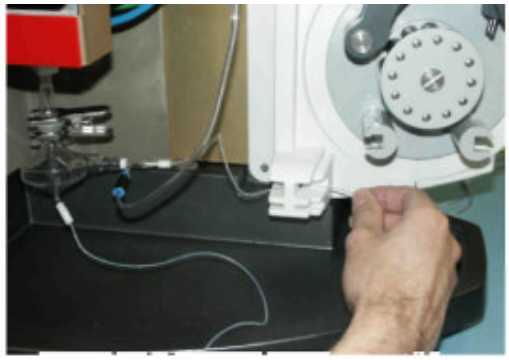


Figure 13 – Sample Capillary Tubing

Feed the sample capillary tubing from the rear of the nebuliser through the upper holder in the cover and towards the pump.
Ensure there are no twists or bends in the nebuliser and drain PTFE tubing that may prevent flow of the sample. Insert the sample and drain PTFE tubing into their respective Tygon pump tubing, making sure that the drain tubing is threaded through the lower hole in the case (see Figure 14 below) for the drain sensor to work correctly.
Please note: the drain tubing should be connected correctly to account for the counter-clockwise flow.

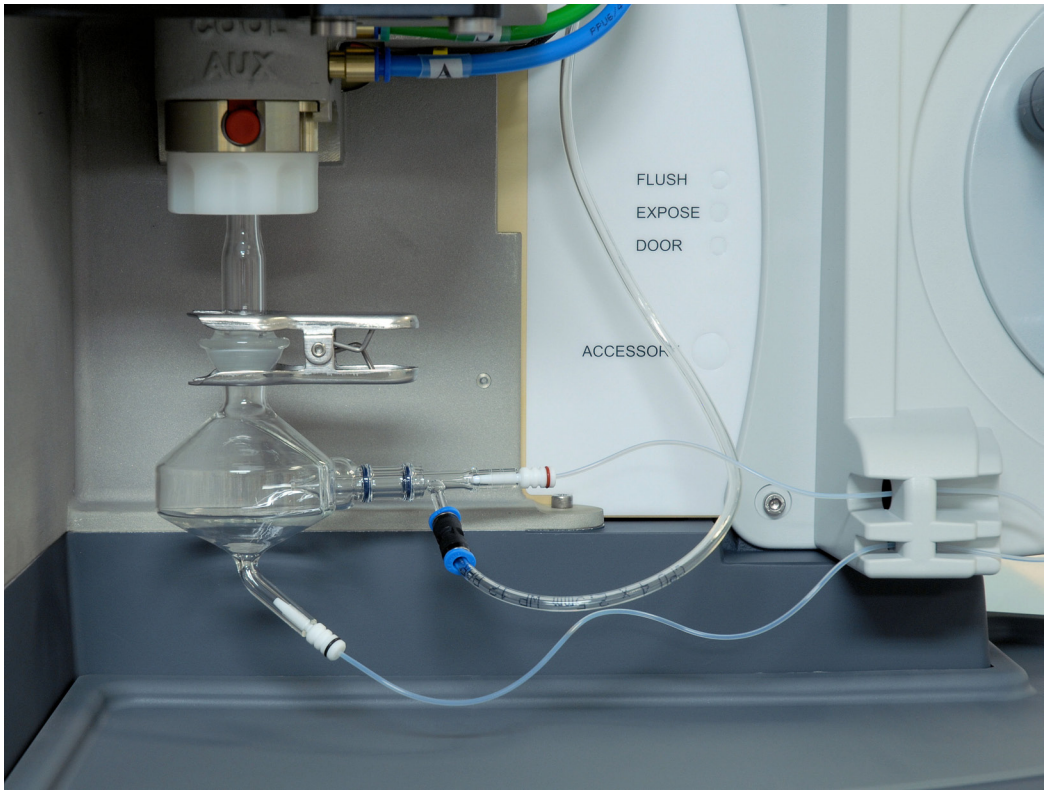


Figure 14 – Sample Capillary Tubing

Pass the drain capillary tubing through the lower holder in the cover and towards the pump. This holder contains a sensor detecting bubbles produced when the spray chamber is draining normally.
The plasma and the pump will be switched off after 2 minutes if no bubbles are detected.

Figure 15 – Pump Tubing

Release the pump tubing clamps and locate the sample and drain pump tubing over the pump rollers, locking the lugs on the pump tubing into the left and right clamps.
Connect the sample pump tubing to the sample capillary tubing and the drain pump tubing to the drain capillary tubing; remember to allow for the direction of flow.
Pump tubing should be inspected before each analysis and should be replaced if there are indications of wear.
Additional lengths of capillary tubing should be used to allow connection to the input of the sample pump tubing to the sample and the output of the drain pump tubing to a waste container.

Figure 16 – Pump Tension Adjustment

The pump tension can be adjusted with the plasma running and the pump stopped. Lock the sample pump tubing and clamp into position. Release the tension adjustment and allow the nebuliser to free aspirate. Tighten the tension adjustment until the flow just stops then tighten by one turn. Turn on the pump and, if necessary, tighten the tension until a smooth flow is produced. Do not over-tighten the pump clamps as it will result in excessive wear and tear of the pump tubing and require replacement tubing at more frequent intervals.

3 Autosampler Use

3.1 Introduction

The autosampler can be configured to suit an application, or several applications.
The volume, number and type of sample will all influence the set-up of the autosampler.

3.2 Autosampler Installation

To comply with safety and warranty requirements the iCAP 6000, accessories and associated equipment must be installed by a Thermo Fisher trained and certified engineer.

3.3 Autosampler Set-up

Figure 37 Autosampler Tubing Set-up

For analysis with an autosampler the capillary tubing attached to the end of the autosampler probe should be attached to the end of the sample pump tubing on the iCAP 6000. To minimise the sample volume required the length of the capillary tubing should be minimised, but should allow free movement over the whole sample area of the autosampler.

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4 iTEVA Familiarisation

4.1 Introduction

The iCAP 6000 will require optimisation that is dependent on the sample being analysed and the method requirements.
It is important that the method development verifies the data produced by the method.
It is also important that a suitable quality control regime is established that verifies the continuing validity of data.
Training courses are available through a local Thermo Fisher Sales Office; contact details are available on http://www.Thermo.com.

4.2 Method Optimisation

The following parameters can all affect the data obtained and should be optimised. Usually a default setting will give data that is satisfactory, but may not be optimal for the analysis requirements:
Nebuliser Gas flow
Plasma viewing height
RF Power
Pump speed
Auxiliary gas flow
Coolant gas
All these instrument parameters are separate to the development of the chemical requirements of the method, for example variation in sample ionisation and solvent effects.

4.3 Example Standard Operating Procedures

This method setup procedure, which by no means covers all the possible parameters used in iTEVA, should be enough for setting up a basic Analysis. It is recommended that the user reads the iCAP software manual and/or Help files for more advanced use of the system.

4.3.1 Preparing the system

Turn Argon Gas on at Cylinder and set for 90psi or 6 Bar pressure on gauge near instrument **Note: for normal use gas should be left purging constantly**
Switch on power to iCAP Spectrometer. **Note: for normal use power should be left on constantly or you must wait until the optics temperature reaches 38 degrees C and Stabilizes which can take 2 hours.**
Switch on Water Chiller
Push Platen on to rollers of pump by way of the 4 (3) pressure screws
Make sure the drain tube is placed in an open neck vessel
Place sample tube in a blank solution
Switch on computer

Click on the **iTEVA** Icon on desktop	

Type **admin** in user name field and click on OK or press enter

4.3.2 Striking the Plasma

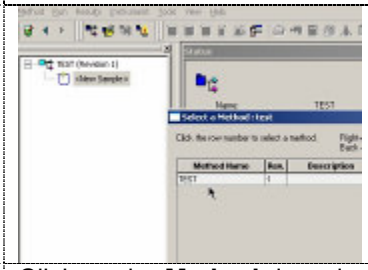
Click on the Plasma Icon and then make sure the interlock indicators all show green, click on **Plasma On**. After the plasma strikes, wait until the Spectrometer Optimization is completed and then click on the close box

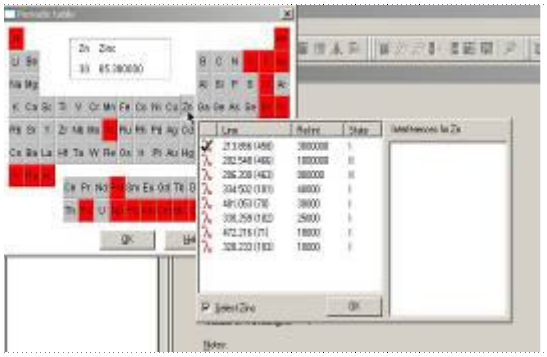
Note: To allow the plasma to stabilise leave Plasma on with blank solution running for about 10 minutes before carrying out an analysis.

4.3.3 Setting up Analyses

Click on the Analyst icon

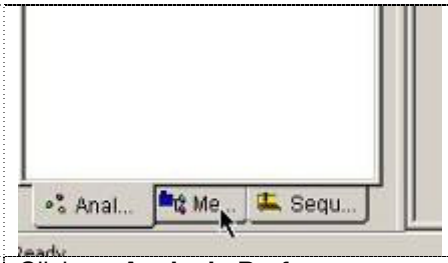
Either select one of the stored methods or create a New method. To create a new method click on Cancel

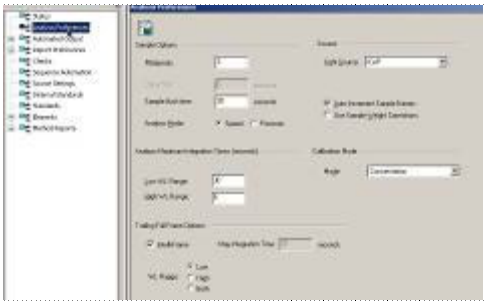




Click on the **Method** drop down menu
Click on **New** and select the Elements required from the Periodic table.
To select suitable lines from the list displayed click **OK** and **OK** again

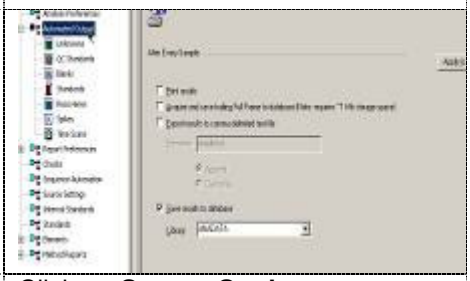
Click on **Method** tab as shown





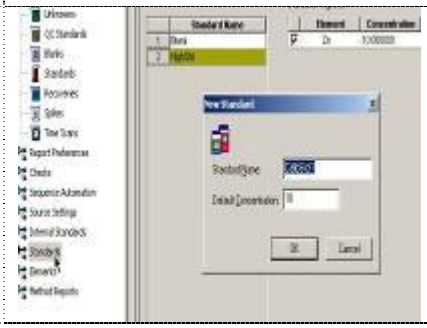
Click on **Analysis Preferences**
Set Parameters as required (see iTEVA software manual for fuller explanation of the various parameters that can be selected)
Note: The parameters shown will be a good starting point for an Analysis

Click on Automated Output and fill in the relevant boxes
Note: Make sure you tick **save results to database** if you require a record of your results



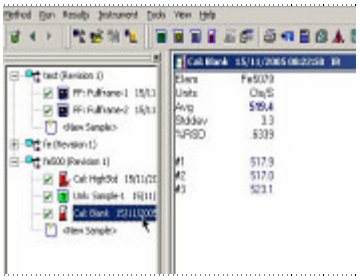
Click on **Source Settings**
Note: the source settings will depend on the particular analysis that is to be performed but for test purposes the defaults should give reasonable results for an aqueous solution.

Clicking on Standards allow you to edit the concentration of the standards in the box.
Note: by clicking on **Add** you can add more standards if required.



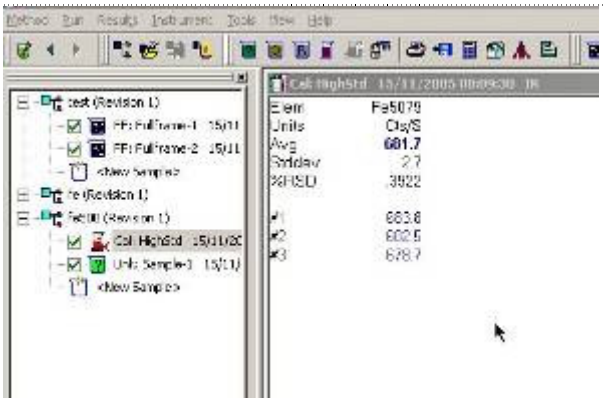
4.3.4 Running the Analysis

Put the sample uptake tube in the blank solution. Click on the Analysis tab and then click on the Calibration Icon, click on **Run** in the Calibration box this will then analyze the blank solution



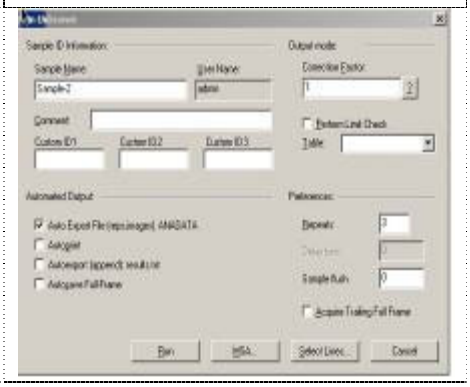
Display of Blank solution results showing the Elements selected in the method setup.

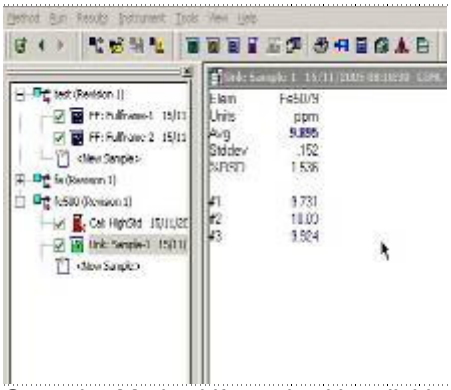
Put the sample uptake tube into the High standard solution
The **HighStd** is now highlighted so click on **Run**



Display of High Std results showing elements selected in the method setup.

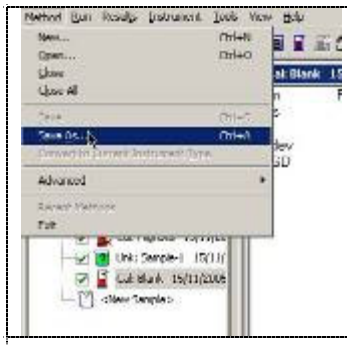
Click on **Unknown** sample Icon then fill in **Run Unknown** box as required Place the sample tube in unknown and Click on **Run**





Display of the Results page

Save the Method if required by clicking on **Method** drop down menu and **Save As,** filling in the appropriate name.



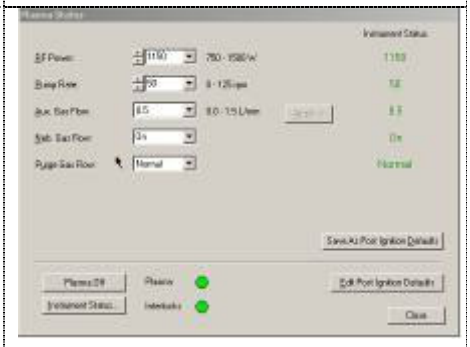
4.3.5 Shutting down the system

Place the sample tube in Deionised water and let it pump through the system for 3 to 5 minutes.

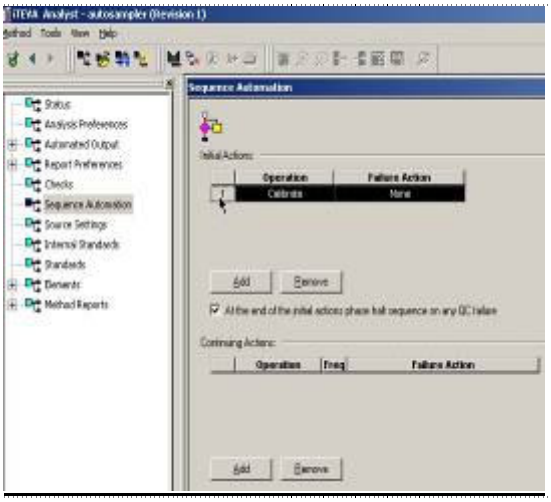
Click on the Plasma Icon  and then click on **Plasma Off.**

Release the tension on the sample pump Platen.

Switch off water circulator.

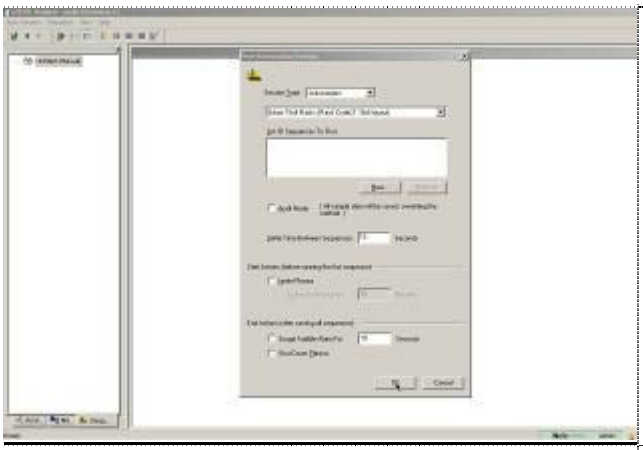


4.3.6 Autosampler Operation



First edit the Sequence Automation page of the method you plan to use. Then save the method.

Click on Sequence tab at bottom of page



Select **New Autosampler** on the **Auto-Session** Menu. This will pop up the dialog box shown.

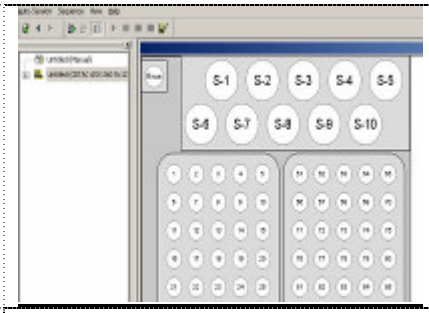
Select the Autosampler rack type from the drop down box. Click on OK

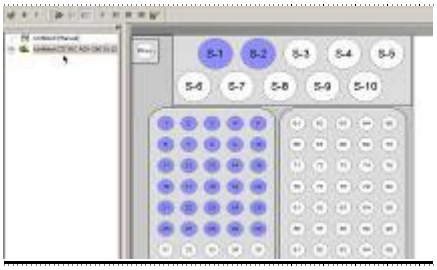
Select **New** this will enable you to select an already saved method to run the Autosampler.

Input the number of samples, and a sample name as required. Click on OK and OK again

Note: The sequence Automation page must be edited and method saved before running samples with the autosampler.

The autosampler position tray will be displayed.





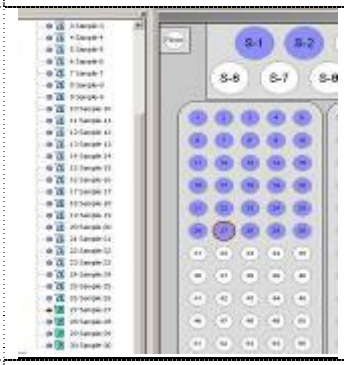
Right click on the session name and select **Auto-Locate** this will then highlight the sample and standard positions.

Note: Make sure the Plasma is ignited

Click on the **Autosampler connect** icon as shown.

Click on **Run icon**

Image of Autosampler showing progress of sample run.



Results as shown on Analysis page

4.3.7 Local Database Creation

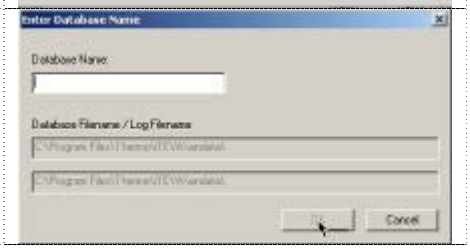
- Click on *TOOLS/OPTIONS* on iTEVA control panel.

- Click on *Application Database* Tab

- Click on, *Run database wizard* box

- Type .\iTEVA in Server Name box
- Type *sa* in User Name box
- Type *teva* in Password box
- Click on *Test connection* box
- This should fill in the Server type and Server version boxes
- Click on *Create* box

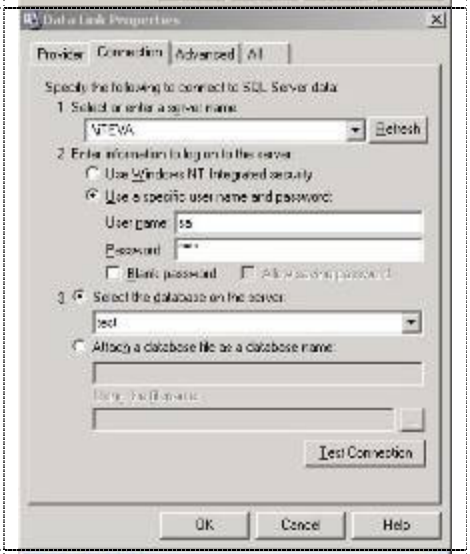
- Enter required name for Database click OK and click on close box



4.3.8 Creating a Database Connection

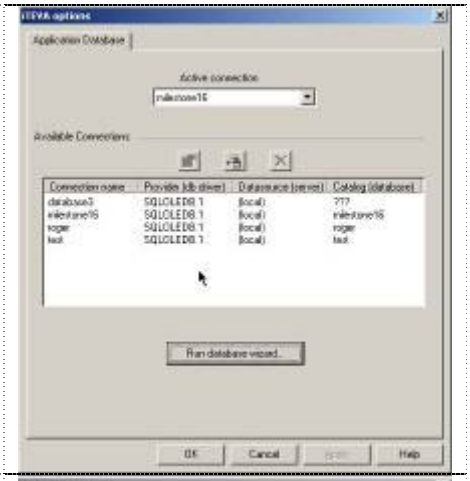
- Click on the *add a connection* box as shown

- Type .\iTEVA in server name box
- Type *sa* in User name box
- Type *teva* in Password box
- From the, *Select the database on the server* box choose the Database required
- Click on *test Connection*
- Click OK



- Follow instructions on box shown, fill in an appropriate name.

- The newly created database will now appear in the list of databases
- Select the created database form the *Active connection drop down box and click on OK*
- This will now make the new database active



4.3.9 Attaching a database

- After running data base wizard click on *Attach*

- Click on *Database Filename/Log Filename*
- *Select database to Attach*
- *Click on OK*
- *Database will now be attached*

4.3.10 Detaching a database

- Carry out the same procedure as Attaching a Database but click on Detach in the Database Wizard

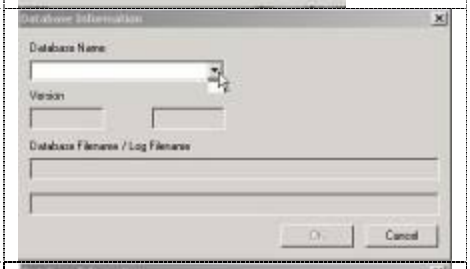
Note: The maximum database size is 4Gb. A new database must be created before this limit is reached.

4.3.11 Backing up databases

- Click on, *Run database wizard* box

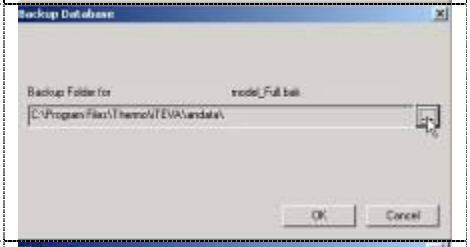
- Type .\iTEVA in server name box
- Type *sa* in User name box
- Type *teva* in Password box
- Click on *Backup*

- Select database name from drop down list



- Click OK

- Choose path for Backup and click OK



- Click Yes
- Backup is now complete

4.3.12 Restoring a database

- From Data base wizard select *Restore* and carry out the same procedure as *Backup* database above

5 Maintenance

5.1 Introduction

The iCAP 6000 has been designed for minimum maintenance; however the sample introduction components should be checked regularly for contamination and wear.

5.2 User Maintenance

Routine operator maintenance of the iCAP 6000 is mainly concerned with keeping the instrument clean.

5.2.1 Instrument Cleaning

'Before using any cleaning or decontamination methods except those specified by the manufacturer, USERS should check with the manufacturer that the proposed method will not damage the equipment.'

Warning: The iCAP 6000 instrument covers are made of ABS plastic materials, which can be damaged by strong solvents and concentrated acids.

Any spillage on the external covers or within the sample introduction areas should be cleaned up immediately, using appropriate safety precautions.
Stains and marks on the covers should be removed with a soft cloth moistened with a dilute detergent solution. Do not use any solvent based cleaners.

5.2.2 Sample Introduction System Cleaning and Decontamination

Failure to maintain the sample introduction system can result in erroneous results, poor precision, poor detection limits and blockages.
After use, the instrument shut down procedures should be followed.
Components contaminated with sample residues should be cleaned.
It is recommended that several spares for each part are available as blockages, sample contamination and breakages often happen at critical moments during analysis.
Suitable protective clothing, glasses and gloves should be worn.

5.2.2.1 Torch Cleaning

Reverse the torch assembly process detailed previously.

Warning: Allow at least 10 minutes for any hot components to cool before removing them from the torch compartment. Care should be taken to remove any broken glass from the Duo radial POP window, if a breakage occurs in the torch box.

To remove salt deposits wash the torch in an ultrasonic bath for five minutes in a dilute surfactant.
To remove metallic deposits from the tip, separate the torch quartz section, immerse the tip of the torch in hot acid (a mixture of nitric and hydrochloric similar to Aqua Regia is suitable)
After cleaning, rinse the torch with de-ionised water and place in a drying oven at 95º until it is dry. Rinsing with a volatile zero residue organic solvent (propanol is suitable) will aid drying.
To clean the torch of carbon deposits, place the torch in a muffle furnace and heat to 750º. Open the door for a few seconds to allow air to enter, close and allow the oven to reach 750º again. Repeat this several times to remove all the carbon. Allow the furnace to cool over several hours, as this will prevent stress building up in the quartz.

5.2.2.2 Spray Chamber Cleaning

Reverse the spray chamber assembly process detailed previously.
Soak the spray chamber in cold acid for two hours (a mixture of nitric and hydrochloric similar to Aqua Regia is suitable). After cleaning rinse the spray chamber in deionised water.

5.2.2.3 Nebuliser Cleaning

Reverse the nebuliser assembly process detailed previously.
Introduce a rinsing agent into the gas input and fill all areas previously exposed to sample solutions, a squeeze bottle is suitable for this. Attach a low pressure clean gas line to the nebuliser gas inlet to expel the liquid. Repeat several times.

Warning: On no account put the concentric nebuliser in an ultrasonic bath or heated in an oven.

5.3 Purged Optical Path Window Cleaning

Before attempting to clean the Purged Optical Path (POP) window (note: there are two on a Duo configuration instrument) turn off the plasma and allow thirty minutes for any hot areas to cool down.
Open the small access door next to the sample compartment door and withdraw the POP window assembly.

Warning: do not open this access door when the plasma is running, there is a potential UV radiation hazard.
All mirrors in the optical system are coated do not to touch the mirror below the radial view POP window in the Duo configuration.
Remove the quartz window from the POP and soak in cold acid for two hours (a mixture of nitric and hydrochloric similar to Aqua Regia is suitable). After acid soaking rinse in de-ionised water, then with a volatile zero residue organic solvent (propanol is suitable) to aid drying.

5.4 Preventative Maintenance

The iCAP 6000 has been designed for minimum maintenance; however periodic checking of performance is required by many laboratories. This is particularly important for customers subject to external standards and regulations (for example ISO9000, EPA or NAMAS). Details of these options are available from a local Thermo Fisher Office.
All electrical supplies, gas supplies and extraction must be checked to insure local health and safety guidelines and regulations are complied with. The gas and cooling water should be checked for leaks at regular intervals.

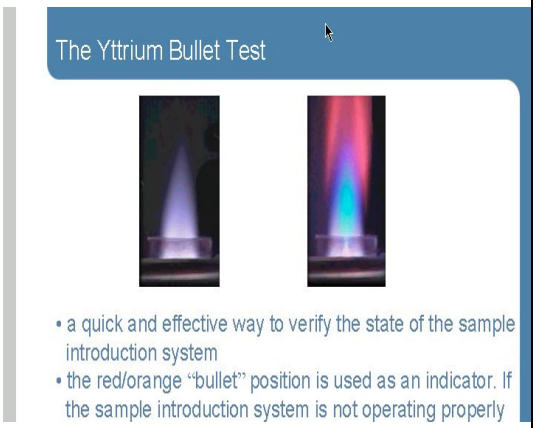
6 Analytical Problems Hints and Tips

6.1 Poor Precision

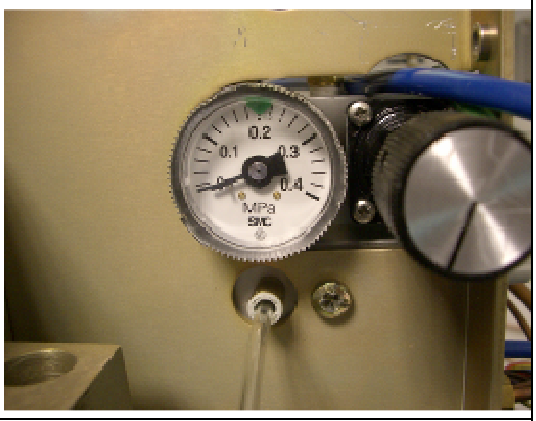
- Before jumping to any conclusions, a quick test should be run to determine if the poor analytical results are matrix related.
- A 10 ppm solution of a few common elements such as Cu, Mn, Ba, Cd, Zn and Fe should be put together and a method made using the primary lines.
- After standardisation, the standard should read back, on the iCAP for example, with a precision of typically 0.2 to 0.5 percent (with a 10 second integration time). If the precision obtained is substantially greater than this try Torch alignment then go on to trouble-shoot further if the problem remains.
- If the instrument meets these specifications then the sample matrix itself is suspect.
- Possibly modifying plasma parameters such as power will help the situation.

Poor precision generally relates to problems in the sample introduction system.

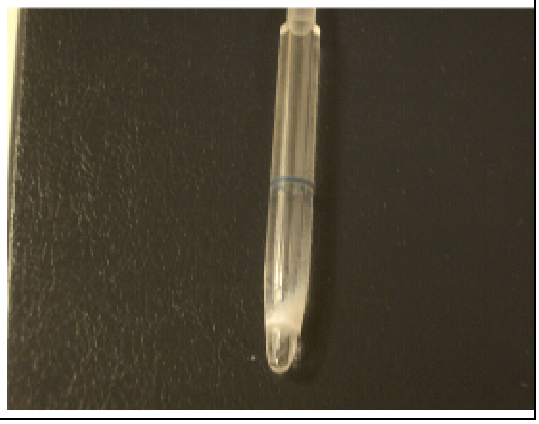
- First check to ensure that the nebuliser pressure or flow is set correctly by aspirating a 1000 ppm Yttrium solution (Sodium will also work if no Yttrium is available).
- Check to ensure that the centre orange "bullet" is even with or slightly above the top of the Radial torch. If not, adjust the nebuliser pressure up or down until the "bullet" looks correct.



- At this point the pressure should be approximately 0.15 mPa for aqueous solutions. If the pressure is substantially higher, the nebuliser orifice is generally to blame and should be cleaned.



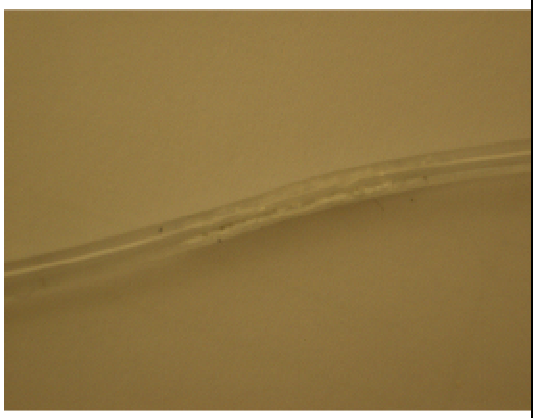
- Pooling and dripping in the spray chamber can also cause many precision problems. You may be able to see this visually using the Y test described above.
- If the Yttrium "bullet" is bouncing up and down inside the plasma, it is usually indicative of dripping. In a

<table>
<tr>
<td>glass or Teflon chamber the chamber should be wetted properly; that is there should be no water droplets building up on the walls of the spray chamber.
❑ This is usually caused by an oily film and can be easily cured by aspirating a 0.1% HF acid solution for about 20 seconds.
❑ HF however, will cause a problem with Si analysis for a short time afterwards. If Si is being analysed try using a Triton X-100 solution. This solution is also good for the polypropylene spray chamber.</td>
<td> 
 
WARNING: Under no circumstances use HF unless you have been trained on its use and have taken appropriate safety precautions</td>
</tr>
<tr>
<td>❑ Other causes of poor precision can be in the expendable parts such as nebuliser and torch/centre tube. Spares should always be available and these should be substituted one by one to observe the result. If the nebuliser is the culprit, check the inside of the orifice, by removing the gas fitting, then with a magnifying glass look for any small obstruction. Also check the capillary for obstructions.</td>
<td>  </td>
</tr>
</table>

	
❑ Less prone to clogging are the high solids nebuliser designs, such as the Babington or V-Groove but orientation is critical as the sample is gravity fed across the argon orifice for atomisation. Note: Two spacing rings to be used at all times	

6.1.1.1 Teflon capillary tubing:

Should be free of kinks and scissor or pinch cut ends. For best results, razor cut at 45° for the drain. Also check the sample peristaltic pump winding condition and platen pressure for proper adjustment. Replace the winding if it is used or collapsed. Pump windings typically last only a couple of days. Introduce an air bubble into the sample uptake tube and watch its migration through the tubing, it should be smooth and consistent.



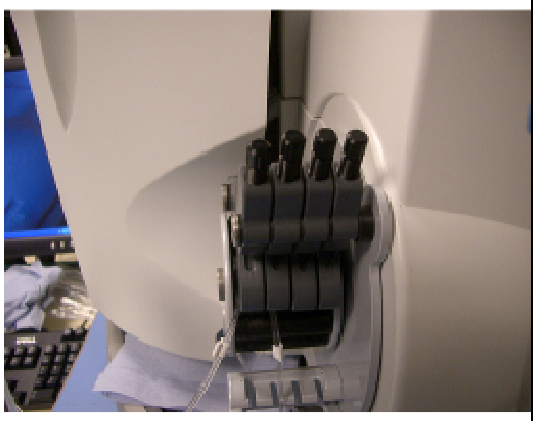
Worn rollers, bent pump head shaft or bad roller bearings can cause inconsistent pump action and any such damaged pumps should be replaced.

- ❑ The peristaltic pump may also be used for the pumped drain spray chamber systems and the internal standards mixing kits.

6.1.1.2 Peristaltic Sample Pump

Note: When using a concentric or crossflow free aspirating type nebuliser, the pump platen pressure should be adjusted with the plasma torch ignited and the pump stopped.

- ❑ To adjust the Platen Pressure
- ❑ Dip the uptake capillary (which is normally connected to a sipper probe) into deionised water.
- ❑ With the nebuliser gas switched off gradually reduce the platen pressure by pushing in the tension adjustment lever until the water freely aspirates through the pump tubing. You can briefly remove the capillary or sipper probe from the rinse for a short period to introduce a small air bubble.
- ❑ With pump turned on adjust the drain pressure to allow small bubbles to flow in the drain tube



- ❑ Finally, argon/air leaks can cause many problems including poor precision.
- ❑ Check the gas fittings on the lines coming from the bulkhead to the torch and nebuliser with a suitable soapy liquid such as Snoop. Leaks at these joints are usually caused by rough tubing and can be stopped by cutting off about 1/2 inch of tubing and reinserting. Replace the tubing with ¼ inch Teflon if the tubing becomes too short

6.2 Poor accuracy/feedback

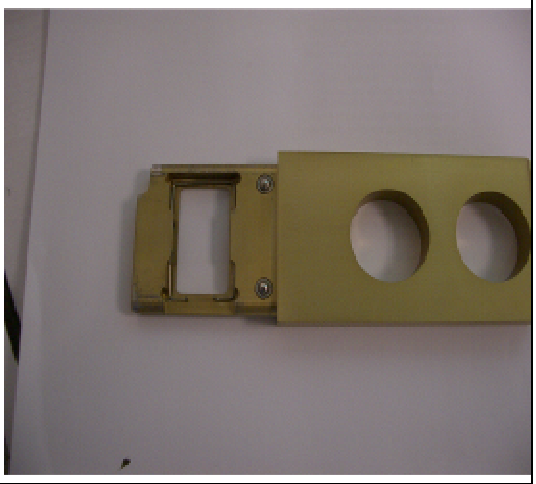
First we should define accuracy as reproducing the standard value once standardised Proceed by making a standard as in the previous section (10 ppm Cu, Mn, Cd, Zn, Fe) and using this as the test solution. Remember we are not defining accuracy for this test as the ability to read a 1 or 100 ppm standard after standardising on the 10 ppm. Most of the time that problem is operator-related. As far as we are concerned at this point we have only one standard to test with. If this simple standard will not repeat back for all elements, check the pump winding first. Replace it if it is used or collapsed.

Note: Pump windings typically last only a couple of days.

- Check the method to see if a fast pump rate is used for the flush period. If it is, then make the flush pump rate the same as the analysis rate and try it again. Inaccuracy can sometimes be traced to the inability of the pump tubing to recover its shape after being stretched.
- Check the flush time for adequate rinse time. A 30-second rinse time is adequate in most cases but not if a slow pump rate is being used or if a very long piece of tubing is used (as with the autosampler probe).

7 Poor detection limits

- This problem can be also related to the poor precision problem discussed earlier and is usually solved by approaching it as such. However, if the loss of intensities is especially pronounced at lower wavelengths it may be due to a dirty window mounted in the Purged Optical Path. UV burn or a dirty mirror is characterised by a long term decline (six months or longer) of intensities.



8 Suggested maintenance in the case of poor precision and detection limits

8.1 Introduction

Maintenance refers to a series of periodic activities that should be performed on a periodic basis to optimise the short term and long term performance of the system. In this chapter we describe activities that should be performed by the typical user of the instrument.

8.2 Typical Maintenance Schedule

- Replace Pump Tubing Weekly
- Clean the Nebuliser Weekly
- Clean the Plasma Torch Monthly

8.3 Replacing pump windings

Type	Solvent Types
Tygon	Aqueous solutions, strong acids and highly polar organic solvents (e.g. methanol and ethanol)
Viton	Solvents of low polarity (e.g. alkanes, aromatics and halogenated hydrocarbons such as gasoline, kerosene, Toluene, xylene, chloroform and carbon tetrachloride).

- A pump tubing in poor condition is characterised by either being flattened hard or discoloured. Squashed tubing is usually caused by leaving the platen pressure on the tubing when it is not being used
- To minimise squashing of the tubing, release platen pressure when the pump is not being used, even for short periods. Hardened and discoloured tubing is caused by chemical reactions with the sample. While these phenomena cannot be avoided, they can be minimised by frequently flushing it with deionised water.

8.4 Preventing blocking of the nebuliser

- The most common problem with the nebuliser is the blockage of the tip by the deposition of particulate matter. In this section we provide a series of suggestions to minimise blockage.
- In most instances blockage in the nebuliser is caused by either particulate matter (from the sample) or chemical deposits. It normally occurs in the nozzle where the flow passages are extremely small and constriction is greatest in the annular gas channel between the tip of the capillary and the taper of the nozzle.

- **Tip:** Filter the carrier gas. Low-impedance in-line gas filters should be installed to prevent particles from being carried into the nebuliser and lodging in the gas annulus. This is especially important when Teflon® tape has been used in the gas line plumbing. Shreds of this tape have been found wedged in the gas annulus and have resulted in drastic reductions in performance. For the same reason, we suggest that you avoid using Teflon or other friable sealants at the gas connection to the nebuliser.
- **Tip:** Filter the sample. The sample capillary is more tolerant of particulate matter than the gas annulus. For high sample uptake nebulisers, the capillary will frequently transport visibly turbid suspensions. We suggest that you filter or centrifuge the sample if the solids are not of analytical importance. Particulates and colloids of a polar nature such as silica, peptides, polyvalent metal hydroxides and others tend to build up on the (polar) glass and impede the fluid flow. In some instances you can prevent deposition by adjusting the pH of the suspension away from its isoelectric point.

- **Tip:** Rinse the nebuliser. It is very important to rinse the nebuliser before turning the gas off. It is advisable to rinse the nebuliser periodically throughout the sequence (depending on the chemistry of your samples).
- Solids may be deposited in the nozzle as sample solvent evaporates, further constricting the flow passages and reducing the signal. Rinsing will minimise or eliminate these deposits.
- Gas flow through most nebuliser models creates a venturi suction at the capillary tip which can be used to draw rinse liquid through the capillary.
- After the testing of any salt solution, promptly rinse the system with a chemically compatible rinse consisting only of volatiles (this is especially necessary in flow injection analysis systems).
- A low-pH (acidic) sample should be followed by a low-pH rinse, a high-pH sample by a high-pH rinse and an organic sample by an appropriate solvent. The final rinse should use deionised water and/or isopropyl alcohol.

Note: Allow the nebuliser to dry before turning off the gas and make sure that the liquid feed is disconnected or arranged so that siphoning into the nebuliser while the gas is off cannot occur.

DO NOT use ultrasonic cleaning to remove particulate matter as sympathetic vibrations may be set up in the capillary causing it to bounce against the inside of the nozzle and chip, Nebuliser performance can decline as a result.

8.5 Removing the concentric glass nebuliser

- Remove the sample capillary by pulling the small white Teflon stopper that attaches it to the back of the nebuliser.
- Remove the nebuliser carefully out of the spray chamber using a rotation motion at the same time as pulling it out.

Note: Be very careful not to dislodge the O-rings as you remove the nebuliser. The O-rings are difficult to replace.

8.6 Removing solids from the nebuliser

- If solids inside the nebuliser are interfering with performance of the system, the steps described below will generally remove them and provide normal operation.

8.6.1 To rinse the nebuliser

- Introduce a rinsing agent into the shell, either from the gas input or the nozzle (a squeeze bottle works well in both cases). Fill all areas previously exposed to corrosive solutions.
- Attach pressurised gas to the side-arm to expel the liquid.
- Inject more rinse solution into the liquid input while the gas is flowing and allow venture suction to draw it through the capillary.
- The final rinse should use isopropyl alcohol to speed the drying process.
- Repeat the treatment if you think it is necessary.
- After the rinse is complete, dry the nebuliser completely.

8.6.2 Particles

These operations are ranked in order of increasing aggressiveness. We recommend that you start with the gentlest procedure and continue with more aggressive procedures as required.

- Tap the liquid input line of the nebuliser gently against a wooden surface (or a surface of comparable hardness) to shake the particle loose. This helps the particle to move in the direction of increasing inner diameter. Repeat the tapping as necessary to work the particle toward the appropriate exit orifice. Avoid extremely harsh tapping.
- Apply compressed gas (15-30 psi) to the nozzle, forcing the gas backwards through the annulus and the capillary (back flushing).

Note: Make sure you hold the nebuliser securely during this operation.

- Gently tap or flick the shell soundly with your fingernail a few times. If this fails to dislodge the particle, close off the liquid and gas input tubes with your fingertips. When the pressure builds up, move your fingertip quickly off the appropriate orifice (if something is wedged in the gas annulus, "pop" your finger off the gas input; if in the capillary). The sudden expansion of gas should help jar the particle loose in the direction of increasing inner diameter. Try to orient the nebuliser so that gravity assists you.

- ❑ Force isopropyl alcohol backwards through the nozzle in an attempt to float the particle out through the larger gas and liquid input tubes. Use a squeeze bottle or plastic dropper with a tip that will form a good seal over the nebuliser nozzle. After the particle has been removed, remove the alcohol through the input tubes using compressed gas, or drain onto lint-free tissue. A variation of this procedure involves the use of a solvent that is known to dissolve the particle (this variation works best if you know which passage the particle is in and your nebuliser is a type C or K with a recessed capillary. In this procedure, inject a slug of 1/4" to 1/2" of solvent into the shell through the nozzle or the gas input tube and close off the nozzle with a fingertip. Apply pressurised gas to the passage that does not contain the particle. Pressurised solvent will force its way out the other channel in the direction of increasing diameter, hopefully carrying the particle along with it).
- ❑ If the particle still remains and you believe that it might be a shred of PTFE tape from the gas line, immerse the nebuliser nozzle in hot water and apply gentle gas pressure to the side arm. The hot water "relaxes" the polymer and allows it to be forced out of the nozzle.

8.6.3 Solid deposit in sample capillary

Note: This step assumes that a passage still exists through the contaminating material (i.e. the tip is not entirely clogged).

- ❑ Try to deduce the chemical nature of the deposit from the type of samples that are being analysed and select the solvent most likely to dissolve it. Inject the solvent into the nozzle with a plastic dropper or squeeze bottle until the affected area is filled. Expel the solvent with compressed gas. Refill and expel the solvent repeatedly. Examine the nebuliser under magnification. If the material is gone, rinse the nebuliser with isopropyl alcohol and dry thoroughly.
- ❑ Immerse the nozzle in a rinse solution. Warm the solution for stubborn deposits. Follow with a rinse of pure solvent, then isopropyl alcohol and dry thoroughly.
- ❑ If the deposit remains after prolonged soaking, apply pressurised gas at the appropriate input(s) to help force the deposit away.

8.6.4 Organic matter

- ❑ Immerse the nozzle of your nebuliser in a hot cleaning solution of chromic and sulphuric acids at 100ºC.

Caution: This solution is corrosive, use suitable precautions

- ❑ Allow the solution to rinse into the passages of the nebuliser until the affected area is filled. Expel and replace the solution at intervals until the deposit is gone or until the chromium reduction (green colour) ceases. Rinse the nebuliser thoroughly with water, then with isopropyl alcohol and dry completely.

8.6.5 Plugged capillary (fusible solids e.g. waxes)

Note: This procedure should be used when no passage remains through the deposit.

- Carefully heat the nebuliser in the region of the capillary obstruction. Simultaneously (or intermittently) apply gentle gas pressure at the sample input tube.

Caution: Avoid overheating residues that may produce insoluble pyrolysis products

- Stop treatment when you have opened a passage through the blockage.

8.6.6 Firmly lodged particle in the capillary

Warning: This procedure places the nebuliser at substantial risk.
Resort to this only when all other methods have failed.

- Insert a fine fishing line, or (7-8 mil) piano wire into the capillary from the nozzle end of the nebuliser. Gently push against the particle, in the direction of increasing diameter, until it is dislodged. Avoid pushing hard enough to buckle the wire as this can break the capillary. Such damage will be permanent and could have a drastic negative effect on the nebuliser' performance. Remove the wire and back flush with compressed gas. Do not attempt to insert the wire (or any other object) into the gas annulus.

8.7 Cleaning the glass mixing chamber

- The glass mixing chambers need cleaning if an oily film is deposited on the chamber walls as these deposits can cause the sample to bead and form large droplets on the chamber walls. When these large droplets finally drip to the drain, a change in back pressure causes a spike to the sample injection and elevates the RSDs.
- The sample should drain smoothly from the sides of the chamber.
- To clean the mixing chamber, wash it with soapy water. The mixing chamber does not need drying before re-installation in the spectrometer. The easiest way to remove any oily film in the glass mixing chamber is to aspirate a 0.1 % HF acid solution for 20 seconds.
- Keep in mind that this causes a high blank for a while afterwards

WARNING: Under no circumstances use HF unless you have been trained on its use

- To clean the torch of carbon deposits, place the torch in a muffle furnace and heat to 750ºC. Open the door to admit air for a few seconds, then close the door and allow the temperature to return to 750ºC. Repeat two or three times until the carbon is burned off. Switch off the muffle furnace and allow it to cool down without opening the door This will take several hours
- The furnace will cool sufficiently slowly to prevent stress in the quartz. It is recommended that at least 2 torches be rotated, so that you do not have to stop work while waiting for the torch to be cleaned.

9 Notes