The IonSABRE APCI User’s Guide

This User’s Guide describes the IonSABRE APCI Probe and its associated Ion Sabre Control Unit; it should be read in conjunction with the appropriate User’s Guide supplied with the instrument.

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Safety Information

The IonSABRE APCI Probe and Ion Sabre Control Unit are designed solely for use with the mass spectrometers listed below; any attempt to use them for any other purpose is liable to damage the Probe, Control Unit, or mass spectrometer, and will invalidate their respective warranties.

- Quattro Ultima™.
- Quattro micro™.
- Q-Tof Ultima™.
- Q-Tof micro™.
- Q-Tof Ultima™ Global.
- LCT™.
- ZQ™.

This equipment conforms to European standard EN61010-1:2001, Safety Requirements for electrical equipment for measurement, control and laboratory use – Part 1: General requirements.

The equipment has been designed and tested in accordance with recognized safety standards. If the equipment is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired.

Whenever the safety protection of the equipment has been compromised, disconnect the equipment from all power sources and secure the equipment against unintended operation.

The Ion Sabre Control Unit must be installed in such a manner that the user can easily access and isolate the power source.

Safety Symbols

Warnings in this User’s Guide, or on the instrument, must be observed during all phases of service, repair, installation and operation of the instrument. Failure to comply with these precautions violates the safety standards of the design and intended use of the instrument.

Waters Corporation assumes no liability for the user’s failure to comply with these requirements.

The following safety symbols may be used in the User’s Guide, or on the instrument. A Warning is an instruction that draws the user’s attention to the risk of injury or death; a Caution is an instruction that draws attention to the risk of damage to the instrument.
**Warning:** This is a general warning symbol, indicating that there is a potential health or safety hazard; the user should refer to this User's Guide for instructions.

**Warning:** This symbol indicates that hazardous voltages may be present.

**Warning:** This symbol indicates that hot surfaces may be present.

**Warning:** This symbol indicates that there is danger from corrosive substances.

**Warning:** This symbol indicates that there is danger from toxic substances.

**Warning:** This symbol indicates that there is danger from flammable substances.

**Warning:** This symbol indicates that there is danger from laser radiation.

**Caution:** This is a general caution symbol, indicating that care must be taken to avoid the possibility of damaging the instrument, or affecting its operation.
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Hardware Specifications

Ion Sabre Control Unit Dimensions

Figure 1  Ion Sabre Control Unit Dimensions

Note:

75 mm clearance is required at the back of the Control Unit for ventilation and the mains cable.

Weight

Ion Sabre Control Unit: 6.5 kg.
Power

**Warning:** A correctly-rated safety earth must be provided.

Mains voltage: 110 to 230 V a.c., 3.7 to 1.8 A, 50 to 60 Hz.

Maximum power: 400 W.

Environment

Ambient temperature: 15 to 28 °C (59 to 82 °F).
Short term variance (1.5 hours): ≤2 °C (≤4 °F).

Humidity: Relative humidity ≤70%.

Installation Category: II.

Pollution Degree: 2.

Services

All services, apart from the mains supply, are provided via the instrument and are described in the instrument’s User’s Guide.
Instrument Description

Introduction

Atmospheric Pressure chemical Ionization (APcI) is a HPLC-MS interface that allows the analysis of many samples that do not run well with ElectroSpray ionization.

In a standard APcI interface, the eluant from a HPLC is fed into the APcI probe in which it undergoes nebulization to form an aerosol spray of fine droplets. On formation, the nebulizer plume is rapidly heated in a stream of nitrogen gas before emerging from the probe as a stream of desolvated sample droplets. These are then directed between the corona discharge needle and sample cone.

Reagent ions are formed in the region of the corona discharge needle. These react with analyte molecules to form singly-charged protonated, or deprotonated analyte ions. The resulting sample and reagent ions then pass through the sample cone into the ion block prior to being extracted in to the ion transfer optics.

Since the sample ionization step is dependent on ion-molecule reactions, signal responses can vary with sample.

The IonSABRE APCI Probe

The IonSABRE APCI probe uses a higher power heater design than that used in the standard APcI probe. The nebulizer plume is expanded into a directly heated region with an increased internal diameter; this results in a larger aerosol expansion and more efficient droplet evaporation. The nebulizer support gas allows the droplet residence times to be controlled and the sample to be swept from the probe in a positive manner, resulting in optimized probe performance.

The IonSABRE APCI probe fits in the probe adjuster assembly in a similar way to the standard APcI probe. The electrical connections are made via the Ion Sabre Control Unit, while the gas supplies remain unchanged.
The IonSABRE APCI probe has an additional Support Gas control valve fitted on the probe body. This controls the nebulizer support gas, which helps to control the residence time of the sample droplets in the heater region. It is important that a nebulizer support gas flow is maintained at all times, however, the flow can be controlled to optimize sample signal.

For APCI operation, the desolvation gas is not heated in the desolvation nozzle, however, a low desolvation gas flow should be used at all times; see Table 1, on page 17, for typical values.

The IonSABRE APCI Probe can be used with the standard type source (glass enclosure with in/out and sideways adjustment, see Figure 4) and the micro type source (aluminium enclosure with sideways adjustment, see Figure 5).
The Ion Sabre Control Unit

The Ion Sabre Control Unit allows the instrument to control and regulate the higher power APcI heater assembly required to drive the IonSABRE APCI probe.
The front panel has two multi-way electrical connections PROBE CONTROL and PROBE OUTPUT.

The PROBE CONTROL (10-way connector) is connected directly to the instrument front panel ESI/APCI socket via cable 4087013CCI. This cable carries the temperature control and interlock signals between the instrument and the Ion Sabre Control Unit.

**Note:**

1. *On LCT instruments, cable number 4087015CCI is used instead of cable number 4087013CCI.*

2. *On Q-Tof Global instruments, cable number 4087016CCI is used instead of cable number 4087013CCI.*

The PROBE OUTPUT (13-way connector) is connected directly to the IonSABRE APCI probe.

![Figure 7 Control Unit Connections](image-url)
Atmospheric Pressure Chemical Ionization

Introduction

Atmospheric Pressure Chemical Ionization (APcI) is an easy to use LC-MS interface that produces singly-charged protonated or deprotonated molecules for a broad range of involatile analytes.

The ability to operate with 100% organic, or 100% aqueous mobile phases, at flow rates between 0.2 and 2.0 mL/min, makes APcI an ideal technique for standard analytical column (2.1 to 4.6 mm i.d.), normal phase and reverse phase LC-MS.

The APcI interface consists of the standard Z-spray source fitted with a corona discharge pin and a heated nebuliser probe. Mobile phase from the LC column enters the probe where it is pneumatically converted into an aerosol and is rapidly heated and converted to a vapor/gas at the probe tip. Hot gas from the probe passes between the sample cone and the corona discharge pin; this is typically operated with a discharge current of 5 µA. Mobile phase molecules rapidly react with ions generated by the corona discharge to produce stable reagents ions. Analyte molecules introduced into the mobile phase react with the reagent ions at atmospheric pressure and typically become protonated (in positive ion mode) or deprotonated (in the negative ion mode). The sample and reagent ions pass through the sample cone into the ion block prior to entering the analyzer.

Changeover between ElectroSpray and APcI operation is simply accomplished by changing the probe and installing the corona discharge pin within the source enclosure; see the instrument’s User’s Guide for details.

For APcI operation, the desolvation gas is not heated in the desolvation nozzle. However, it is important that desolvation gas is used throughout, see Table 1, on page 17, for typical flow rates.

Preparation

General

Ensure that the source is assembled as described in the Maintenance section of the instrument’s User’s Guide. (For a standard type source, ensure that the outer plastic tube is fitted.) Also, ensure that the instrument is pumped-down and prepared for APcI operation as described in the instrument’s User’s Guide.

Checking the Probe Prior to Use

Ensure that the probe heater is switched off.

Unplug the probe from the instrument’s front panel and remove the probe from the source.
Connect the PTFE tube to the **Nebuliser** outlet on the front panel.

Remove the probe heater assembly by carefully loosening the two grub screws.

Disconnect the heater from the probe body by pulling parallel to the axis of the probe.

Ensure that 0.5 to 1 mm of fused silica is protruding from the stainless steel nebuliser tube.

Connect the LC pump to the probe with a flow of 50:50 acetonitrile:water at 1 mL/min.

Check that the liquid jet flows freely from the end of the capillary and that the LC pump backpressure reads 250 to 400 psi.

Check that the nitrogen supply pressure is 6 to 7 bar (90 to 100 psi).

Check that the support gas is set to 1.0.

Select the Tool Bar button, or select the Menu Bar **Gas, Gas** command to turn on the nitrogen gas.

Check that the liquid jet converts to a fine uniform aerosol.

Switch off the liquid flow.

Select the Tool Bar button, or select the Menu Bar **Gas, Gas** command to turn off the nitrogen gas.

Reconnect the probe tip assembly.

Insert the APcI probe into the source and secure it by tightening the two thumbscrews.

Disconnect the plug labeled **ESI** (from the probe adjustment flange) from the front panel.

Connect the probe cable to the front panel **APcI/ESI** socket.

---

**Obtaining an Ion Beam**

**Warning:** For instruments with glass source enclosures, it is normal for the source enclosure, the glass tube and parts of the probe adjustment flange to reach temperatures of up to 80 °C during prolonged APcI operation. The protective cover provided with the APcI probe must be fitted over the glass enclosure to prevent contact with the hot glass surfaces. Care should be exercised when handling source components immediately after operation.
**Warning:** For instruments with aluminium source enclosures, it is normal for the source enclosures, and parts of the probe adjuster assembly, to reach high temperatures when in use.

**Warning:** Switch off the liquid flow, and allow the probe to cool before removing it from the source.

**Caution:** Failure to employ a desolvation gas and support gas flow during APcI operation may lead to heat damage to the source and APcI heater.

Ensure that the corona discharge pin is fitted and connected as described in the instrument’s User's Guide.

Ensure that the APcI probe is fitted as described above, that the desolvation gas tube is connected to the front panel, and that the purge gas outlet is plugged (if present).

If necessary, change the ionization mode using the Tune Page Menu Bar **Ion Mode** menu. The top line of the tune page indicates the current ionization mode.

Set **Source Temp (°C)** to 130 °C.

Set **APcI Probe Temp (°C)** to 20 °C with no liquid flow and the nitrogen off. Set **Corona (µA)** to 5 µA.

**Note:**

The corona discharge may have either current or voltage regulation, depending on the model of instrument.

Set **Cone** to 50 V.

When **Source Temp (°C)** reaches 130 °C:

Select the Tool Bar button, or select the Menu Bar **Gas, Gas** command to turn on the nitrogen gas.

Adjust **Desolvation Gas** to 150 L/h and set **Nebuliser Gas** to its maximum setting (where applicable; the nebulizer gas flow cannot be adjusted on certain types of instrument).

To monitor the flow rate, select the Tune Page **Source** tab and observe the **Gas Flows** read-back window.

Set the support gas to 1.0.

Select one of the peak display boxes and set **Mass** to 50 and **Span** to 90.
Select the button.

Increase Gain until peaks become clearly visible on the Tune Page.

Set APci Probe Temp (°C) to 500 °C.

When APci Probe Temp (°C) reaches 500 °C:

Start the LC pump at a flow of 1 mL/min.

Adjust the probe’s in/out position so that it is fully retracted (Quattro Ultima type sources only).

Adjust the probe’s position so that the spray is directed approximately at the midpoint between the corona pin and the sample cone.

Check that a stable beam of solvent ions is now apparent on the Tune Page.

Refer to the Hints for Sample Analysis section for further information on source tuning.

Calibration

Having obtained a stable APci beam, refer to the Mass Calibration section in the instrument’s User’s Guide.

Hints for Sample Analysis

Tuning for General Qualitative Analysis

Refer to the Obtaining an Ion Beam section and tune on solvent ions.

Adjust the in/out position of the probe so that it is fully retracted from the source.

Using the sideways adjuster ensure that the spray is directed approximately at the mid-point between the corona pin and the sample cone.
For general qualitative analysis of mixtures, the following parameters are typical:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tr>
<td>Corona Discharge</td>
<td>5 µA*</td>
</tr>
<tr>
<td>Source Temp</td>
<td>130 °C</td>
</tr>
<tr>
<td>APcI Probe Temp</td>
<td>500 °C*</td>
</tr>
<tr>
<td>Desolvation Gas</td>
<td>150 L/h*</td>
</tr>
<tr>
<td>Cone Gas (if present)</td>
<td>100 L/h</td>
</tr>
<tr>
<td>Support Gas</td>
<td>1.0</td>
</tr>
</tbody>
</table>

* See the following section for specific tuning details.

**Table 1  Parameter Values**

### Specific Tuning for Maximum Sensitivity

- For quantitative Multiple Reaction Monitoring (MRM) analysis, optimum APcI conditions should be obtained for each analyte using standard solutions.

- Tuning may be performed using a tee to introduce a standard solution (typically 100 pg/µL) at 10 µL/min into the mobile phase stream.

- Alternatively, to optimize the APcI, repeat direct loop injections of a standard solution (typically 10 pg/µL) into the mobile phase stream may be used, while acquiring in the MRM acquisition mode. The source parameters may be adjusted, and the effects observed, during an acquisition.

### Corona Discharge Current

Corona discharge current can have a significant effect on sensitivity. The corona discharge current required can depend upon the polarity of the compound and the polarity of the analytical mobile phase. As recommended above, optimization should be done in the presence of the analytical mobile phase being delivered at the chosen flow rate.

To find the optimum value:

Start at 5 µA and increase **Corona (uA)** in 2 µA steps until the optimum is found, allowing the current to stabilize for each step before taking a reading.

**Note:**

*The corona discharge may have either current or voltage regulation, depending on the model of instrument.*
Probe Position

The position of the APcI probe has a significant effect in the sensitivity obtained, with the position of the probe tip relative to the corona needle tip being important.

Use the probe adjuster mechanism to ensure that the spray is directed approximately at the mid-point between the corona pin and the sample cone. Adjust the probe position around this point, one turn at a time, to optimize the signal.

Probe Temperature

Caution: (LCT only) The APcI Probe Temp (°C) must not be set to more than 650 °C.

It is important to optimize APcI Probe Temp (°C) for maximum sensitivity, as follows:

Ensure that the analytical mobile phase is used during optimization.

Starting at 650 °C, (the maximum set temperature), reduce the temperature in 50 °C steps, allowing time for the temperature to stabilize for each step before taking a reading.

Note:

It is possible to set APcI Probe Temp (°C) too low for the mobile phase. This often results in reduced chromatographic peak resolution.

Support Gas

This helps to support the nebulisation processes and aids sample flow through the probe. Adjustment of the support gas flow can influence the signal obtained; typically, a dial setting of 1.0 is used.

Desolvation Gas

In most circumstances, the desolvation gas flow has little effect on signal intensity. However, in some situations, it has been observed to have an effect on chemical background noise levels. Adjusting Desolvation Gas while acquiring in the MRM mode can be used as a check for this.

Removing the Probe

After a session of APcI operation:

Turn off the LC flow.

On the Tune Page, set APcI Probe Temp (°C) to 20 °C.

Select the Press for Standby button.
When the probe temperature falls below 100 °C:

Select the Tool Bar button, or select the Menu Bar Gas, Gas command to turn off the nitrogen gas.

**Warning:** Take care when removing the APcI probe; there is a risk of burns to the operator.

**Caution:** Removal of the APcI probe when hot shortens the life of the probe heater.

Undo the two thumbscrews and remove the probe from the source.
Maintenance and Fault Finding

Introduction

**Warning:** Many of the procedures described in this chapter involve the removal of possibly toxic contaminating deposits using flammable or caustic agents. Personnel performing these operations should be aware of the inherent risks, and should take the necessary precautions.

**Warning:** Never disconnect the electrical source assemblies while the instrument is in Operate mode. Always wait for 30 seconds after going into the Standby mode before disconnecting assemblies.

Refer to the instrument’s User’s Guide, which describes the correct maintenance and fault finding procedures for the instrument.

Cleanliness and care are of the utmost importance whenever internal assemblies are removed from the instrument.

- Always prepare a clear clean area in which to work.
- Make sure that any tools or spare parts that may be required are nearby.
- Obtain some small containers in which screws, washers, spacers, etc. can be stored.
- Use tweezers and pliers whenever possible.
- If nylon or cotton gloves are used, take care not to leave fibers in sensitive areas.
- Avoid touching sensitive parts with fingers.
- Do not use rubber gloves.
- Before reassembling and replacing dismantled components, inspect O-rings and other vacuum seals for damage. Replace with new if in doubt.
- Always ensure that none of the cooling fans are obstructed. It is essential that the fan filter is checked at regular intervals, and renewed if there is any doubt about its effectiveness.
- Ensure that good vacuum procedures are followed at all times. The correct procedures for pumping and venting are described in the instrument’s User’s Guide.

Should a fault occur soon after a particular part of the system has been repaired, or otherwise disturbed, it is advisable to ensure that this part has been correctly refitted and/or adjusted. Also check that adjacent components have not been inadvertently disturbed.
The Source

Overview

**Warning:** Cleaning the various parts of the source requires the use of solvents and chemicals that may be flammable and hazardous to health. The user should take all necessary precautions.

The Z-spray source is a robust assembly requiring little maintenance; it is described in depth in the instrument’s User’s Guide. The source consists of three basic parts:

- The probe adjustment flange.
- The source enclosure.
- The source flange assembly.

The probe adjuster assembly and source enclosure are integral items on instruments with aluminium source enclosures, for example ZQ and Quattro micro, whereas they are separate on instruments with glass source enclosures, for example Quattro Ultima.

On the Quattro Ultima the probe adjustment flange and the glass tube can be readily removed, without venting the instrument, to gain access to the ion block and sample cone. On the Quattro micro the ion block and sample cone can be accessed by opening the source enclosure front door.

This allows the following operations to be performed:

- Removing the cone gas nozzle and sample cone.
- Fitting or removing the APcI discharge pin.
- Fitting or removing the exhaust liner and cleanable baffle.

Cleaning of the sample cone and cone gas nozzle may be achieved by removing them from the source. This may also be done without venting the instrument, by closing the isolation valve located on the ion block.

**Cleaning the Z-Spray Source and Sample Cone**

**Warning:** Removal of the APcI probe or desolvation nozzle when hot may cause burns.

**Caution:** Removal of the APcI probe when hot shortens the probe heater’s life.
**Warning:** The ion block is exposed when the source enclosure has been removed. Ensure that the ion block heater has cooled before proceeding.

**Caution:** The sample cone is a delicate and expensive component; it should be handled with extreme care. Do not attempt to remove any obstruction by poking. This may result in damage to the sample cone.

Cleaning may be necessary due to lack of sensitivity or fluctuating peak intensity, or if deposited material is visible on the outside of the nozzle or sample cone. Proceed as described in the instrument’s User’s Guide.

If material build-up is observed on the exhaust liner and cleanable baffle, they should be cleaned as described in the instrument’s User’s Guide.

The removal and cleaning of the Ion Block is described fully in the instrument’s User’s Guide.

### The Corona Discharge Pin

If the corona discharge pin becomes dirty, or blunt:

- Remove it from the source.
- Wipe it clean with methanol and sharpen it using 600-grade emery paper.

The needle should be renewed if it becomes bent or otherwise damaged.

### The IonSABRE APCI Probe

Signs that IonSABRE APCI Probe maintenance is required include:

- The probe tip assembly becomes contaminated, for example by involatile samples, if the probe temperature is too low during operation (<300 °C).
- The appearance of chromatogram peak broadening or tailing.

Samples that give rise to a good chromatogram peak shape in APCI (for example reserpine and common pesticides) should display peak widths at half height of the order 0.1 min for 10 µL loop injections at a flow rate of 1 mL/min. The appearance of significant peak broadening or tailing with these compounds is most likely to be due to a broken fused silica capillary or probe tip heater assembly.

- Low LC pump backpressure.

For 50:50 acetonitrile:water at a flow rate of 1 mL/min, a LC pump back pressure less than 14 bar (200 psi) is indicative of a broken fused silica capillary or a leaking connector.
• High LC pump backpressure.

For 50:50 acetonitrile:water at a flow rate of 1 ml/min, a LC pump back pressure above 35 bar (500 psi) is indicative of a blockage or partial blockage in the fused silica capillary, in a LC connector or in the filter. It is advisable to change the inner filter pad (see the Renewing the Fused Silica Capillary section) on a regular basis.

• Gas flow problems.

Check all gas connections for leaks using a suitable leak-searching agent such as Snoop.

Cleaning the Probe Tip

Remove the probe from the source, see the Removing the Probe section, on page 18.

Remove any visible deposits on the inner wall of the probe heater with a nylon tube brush (supplied in the spares kit) soaked in methanol:water.

Before starting an analysis:

With the probe out of the instrument, connect the nebulising gas supply line.

Select the Tool Bar button, or select the Menu Bar Gas, Gas command to turn on the nitrogen gas.

Allow the gas to flow for several seconds to clear any debris from the heater.

Select the Tool Bar button, or select the Menu Bar Gas, Gas command to turn off the nitrogen gas.

Insert the probe into the source.

Select the Tool Bar button, or select the Menu Bar Gas, Gas command to turn on the nitrogen gas.

Caution: (LCT only) The APCI Probe Temp (°C) must not be set to more than 650 °C.

Caution: Do not set APCI Probe Temp (°C) to 650 °C immediately, as this may damage the probe heater.
Raise **APCI Probe Temp** (°C) gradually, starting at 100 °C and increasing in 50 °C intervals to 650 °C over a period of 10 minutes. This procedure should remove any chemical contamination from the probe tip.

**Renewing the Probe Tip Heater**

![Renewing the Probe Tip Heater Diagram](image)

Remove the probe tip assembly after carefully loosening the two grub screws.

Disconnect the heater from the probe body by pulling parallel to the axis of the probe.

Fit a new heater assembly.

Reconnect the probe tip assembly.
Renewing the Fused Silica Capillary

Remove the probe from the source, see the *Removing the Probe* section, on page 18.

Remove the probe tip assembly and the heater, as described in the preceding section.

Remove the probe end cover by removing the two screws and the grub screw that retains the LC filter.

Loosen the filter from the adapter nut.

Unscrew the adapter nut from the probe.

Remove and discard the fused silica capillary.

Using a ceramic capillary cutter, cut a new length of 300 µm o.d. × 100 µm i.d. fused silica capillary, about 10 mm excess in length.

Using a GVF/004 ferrule and the adapter nut, connect the sample capillary to the filter ensuring that the liner tube is fully butted into the filter.
Using a ceramic capillary cutter, cut the capillary at the nebulizer gas tube so that between 0.5 and 1.0 mm of capillary is protruding from the nebulizer gas tube.

**Note:**

*It is important to cut the capillary square. This should be examined using a suitable magnifying glass.*

Undo the adapter nut from the probe.

Withdraw the capillary from the probe.

Use a flame to remove 20 mm of polyamide coating from the end of the capillary, and clean with a tissue saturated with methanol.

Carefully re-feed the sample capillary through the probe.

Using a Rheodyne spanner, gently tighten the adapter nut to the probe.

Refit the probe end cover and retaining screws.

Using a 1.5 mm Allen key, tighten the grub screw in the probe end cover to clamp the filter.

Carefully refit the heater and probe tip assembly.

**Electronics**

**Warning:** There are high voltages present throughout the mass spectrometer. Extreme caution should be taken when taking measurements with a meter or an oscilloscope. In the Standby mode the high voltages are switched off in the source and analyzer assemblies, but high d.c. voltages and mains voltages remain in the power supply units.

**Caution:** The instrument’s electronic systems contain complex and extremely sensitive components. Only Waters engineers should carry out any fault finding procedures for the electronic systems.

**User-Replaceable Fuses**

In the following list, the designation “T” indicates a time lag fuse.

**Analog PCB**

<table>
<thead>
<tr>
<th>Fuse</th>
<th>Fuse Type</th>
<th>Ref. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mains</td>
<td>4 A (T) 20 mm anti-surge ceramic</td>
<td>1340164</td>
</tr>
</tbody>
</table>
Fault Finding Check List

*Warning:* There are high voltages present throughout the mass spectrometer. Extreme caution should be taken when taking measurements with a meter or an oscilloscope. In the standby mode *(Operate* not selected) the high voltages are switched off in the source and analyzer assemblies, but high d.c. voltages and mains voltages remain in the power supply units.

*Caution:* Any investigation in the RF generator must be made only by a Waters engineer.

The fault finding procedures are detailed in the instrument’s User’s Guide.

**No Beam**

Refer to the relevant chapters of the instrument’s User’s Guide and check the following:

- Normal tuning parameters are set and, where appropriate, read-back values are acceptable.
- All necessary cables have been correctly attached to the source and probe.
- All necessary cables have been correctly attached to the Ion Sabre Control Unit.
- The Ion Sabre Control Unit mains power has been switched on. (Check the Ion Sabre Control Unit’s red LED.)
- The support gas is set correctly.
- The probe temperature is set correctly.
- *Operate* is on (check the LED on the instrument’s front panel).
- The source has been assembled correctly and is clean.
- The source isolation valve is open.
- There are no error messages reported by the electronics (see the viewing window at the rear of the instrument).

**Unsteady or Low Intensity Beam**

If the preceding checks fail to reveal the cause of the problem, check that:

- Gas and liquid flows are normal.
- The support gas is set correctly.
- The probe temperature is set correctly.
• The analyzer pressure is less than $1 \times 10^{-4}$ mbar.

• The source and corona current read-backs vary with Tune Page settings.

### High Noise Level in MRM Analyses

**Note:**

*High noise level is not applicable to many instruments.*

The background noise in MRM analyses can be either electronic or chemical. To distinguish between chemical noise and electronic noise, an acquisition should be performed with and without ions being transmitted to the detector. Ions are best prevented from reaching the detector by setting the Tune Page **Analyser tab Ion Energy 1** (MS1) and **Ion Energy 2** (MS2) fully negative. If there is a significant decrease in the background noise with the ion energies set negative then the major contribution to the overall noise is chemical noise. Any residual noise is electronic noise which is described in more detail in the instrument’s User’s Guide.

If the dominant source of noise is chemical, a reduction in electronic noise does not yield significant improvements in overall signal to noise ratio.

### Chemical Noise

The most common source of noise is chemical noise.

• If the auto injector, probe or connecting tubing have been exposed to a high concentration of the compound to be analyzed then this may be giving a high background due to “carry over”. This can occur if concentrations of a few ng/µL are used for tuning prior to attempting sub pg/µL detection levels. If the injector is contaminated, the signal level normally changes with subsequent injections of mobile phase.

**Caution:** Remove the LC column before injecting 10% formic acid and/or isopropanol into the system.

Repetitive injections of 10% formic acid and/or isopropanol may help reduce the noise. If the probe or connecting tubing is contaminated, infusing 10% formic acid and/or isopropanol with a syringe pump may help.

• Check that the LC system is not adding contaminants into the mobile phase.

Using a syringe pump, infuse a syringe of mobile phase taken from the solvent reservoir. Compare this with the background when the LC system is delivering the solvent.

• Try a different ion or MRM transition.

This may reduce the noise level if the compound(s) contributing to the chemical noise do not yield the same set of daughter ions as the compound being analyzed.
• Check the purity of solvents and additives.

Try a different type of solvent or the same type of solvent from a different manufacturer. Ensure all solvents and additives are HPLC grade. Check the cleanliness of any glassware used.

• Check source tuning.

Try re-optimizing the probe position, gas flows and source tuning. Tune the corona discharge current.

**High Backpressure**

For APcI, a higher than normal backpressure readout on the HPLC pump can imply that there is a blockage in the capillary transfer line or injection loop, due to particulate matter from the sample.

Often, injections of neat formic acid help to redissolve any solute that has precipitated out of solution.

If the blockage cannot be cleared in this fashion:

Remove the finger-tight nut and tubing from the back of the probe.

If the backpressure remains high, the blockage is not due to the probe.

If the backpressure is due to a restriction in the probe, renew the fused silica capillary inside the probe.

If the backpressure is still high, renew the probe inline filter.

The solvent flow can be readjusted and the probe refitted into the source.

**Cleaning Materials**

*Warning:* Many of the procedures described in this chapter involve the removal of possibly toxic contaminating deposits using flammable or caustic agents. Personnel performing these operations should be aware of the inherent risks, and should take the necessary precautions.

When cleaning internal components, it is important to maintain the quality of the surface finish. Deep scratches or pits can cause loss of performance. Where no specific cleaning procedure is given, fine abrasives should be used to remove dirt from metal components. Recommended abrasives are:

• 600- and 1200-grade emery paper.
After cleaning with abrasives, it is necessary to wash all metal components in suitable solvents to remove all traces of grease and oil. The recommended procedure is to sonicate the components in a clean beaker of solvent and subsequently to blot them dry with lint-free tissue. Recommended solvents are:

- Isopropyl Alcohol (IPA).
- Methanol.
- Acetone.

Following re-assembly, components should be blown with oil-free nitrogen to remove dust particles.

Further investigations, requiring the services of a qualified service engineer, should be left to Waters personnel.

**Customer Consumables**

**Fuses**

Refer to the *Electronics, User-Replaceable Fuses, Analog PCB* section.

**General Consumables**

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<th>Item</th>
<th>Reference Number</th>
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<td>M955189BC1</td>
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<tr>
<td>Probe Assembly</td>
<td>M956079DC1</td>
</tr>
<tr>
<td>Probe Heater Assembly</td>
<td>M956076BC1</td>
</tr>
<tr>
<td>Cable</td>
<td>4087013CC1</td>
</tr>
<tr>
<td>Cable (LCT only)</td>
<td>4087015CC1</td>
</tr>
<tr>
<td>Cable (Q-Tof <em>Global</em> only)</td>
<td>4087016CC1</td>
</tr>
<tr>
<td>Probe Consumables Kit</td>
<td>M956079DC3</td>
</tr>
<tr>
<td>Ion Sabre Control Unit</td>
<td>4087001DC1</td>
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