# Waters Micromass ZQ Detector

**Operator's Guide** 





34 Maple Street Milford, MA 01757 71500044702, Revision B



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**Note:** When you use the instrument, follow generally accepted procedures for quality control and methods development.

If you observe a change in the retention of a particular compound, in the resolution between two compounds, or in peak shape, immediately determine the reason for the changes. Until you determine the cause of a change, do not rely on the separation results.

**Note:** The Installation Category (Overvoltage Category) for this instrument is Level II. The Level II Category pertains to equipment that receives its electrical power from a local level, such as an electrical wall outlet.



**Atención:** Changes or modifications to this unit not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment.

*Important :* Toute modification sur cette unité n'ayant pas été expressément approuvée par l'autorité responsable de la conformité à la réglementation peut annuler le droit de l'utilisateur à exploiter l'équipement.

**Achtung:** Jedwede Änderungen oder Modifikationen an dem Gerät ohne die ausdrückliche Genehmigung der für die ordnungsgemäße Funktionstüchtigkeit verantwortlichen Personen kann zum Entzug der Bedienungsbefugnis des Systems führen.

**Avvertenza:** eventuali modifiche o alterazioni apportate a questa unità e non espressamente approvate da un ente responsabile per la conformità annulleranno l'autorità dell'utente ad operare l'apparecchiatura.

**Atención:** cualquier cambio o modificación efectuado en esta unidad que no haya sido expresamente aprobado por la parte responsable del cumplimiento puede anular la autorización del usuario para utilizar el equipo.

**注意**:未經有關法規認證部門允許對本設備進行的改變或修改,可能會使使用者喪失操作 該設備的權利。

**注意**:未经有关法规认证部门明确允许对本设备进行的改变或改装,可能会使使用者丧失 操作该设备的合法性。

**주의 :** 기기 검교정 담당자의 승인 없이 무단으로 기기를 변경 또는 수정하는 경우에 는, 그 기기 운영에 대한 허가가 취소될 수 있습니다.

**注意**:規制機関から明確な承認を受けずに本装置の変更や改造を行うと、本装置のユーザ としての承認が無効になる可能性があります。



*Caution:* Use caution when working with any polymer tubing under pressure:

- Always wear eye protection when near pressurized polymer tubing.
- Extinguish all nearby flames.
- Do not use Tefzel tubing that has been severely stressed or kinked.
- Do not use Tefzel tubing with tetrahydrofuran (THF) or concentrated nitric or sulfuric acids.
- Be aware that methylene chloride and dimethyl sulfoxide cause Tefzel tubing to swell, which greatly reduces the rupture pressure of the tubing.

**Attention :** Soyez très prudent en travaillant avec des tuyaux de polymères sous pression :

- Portez toujours des lunettes de protection quand vous vous trouvez à proximité de tuyaux de polymères.
- Eteignez toutes les flammes se trouvant à proximité.
- N'utilisez pas de tuyau de Tefzel fortement abîmé ou déformé.
- N'utilisez pas de tuyau de Tefzel avec de l'acide sulfurique ou nitrique, ou du tétrahydrofurane (THF).
- Sachez que le chlorure de méthylène et le sulfoxyde de diméthyle peuvent provoquer le gonflement des tuyaux de Tefzel, diminuant ainsi fortement leur pression de rupture.

*Vorsicht:* Bei der Arbeit mit Polymerschläuchen unter Druck ist besondere Vorsicht angebracht:

- In der Nähe von unter Druck stehenden Polymerschläuchen stets Schutzbrille tragen.
- Alle offenen Flammen in der Nähe löschen.
- Keine Tefzel-Schläuche verwenden, die stark geknickt oder überbeansprucht sind.
- Tefzel-Schläuche nicht für Tetrahydrofuran (THF) oder konzentrierte Salpeteroder Schwefelsäure verwenden.
- Durch Methylenchlorid und Dimethylsulfoxid können Tefzel-Schläuche quellen; dadurch wird der Berstdruck des Schlauches erheblich reduziert.

**Precauzione:** prestare attenzione durante le operazioni con i tubi di polimero sotto pressione:

- Indossare sempre occhiali da lavoro protettivi nei pressi di tubi di polimero pressurizzati.
- Estinguere ogni fonte di ignizione circostante.
- Non utilizzare tubi Tefzel soggetti a sollecitazioni eccessive o incurvati.
- Non utilizzare tubi Tefzel contenenti tetraidrofurano (THF) o acido solforico o nitrico concentrato.
- Tenere presente che il cloruro di metilene e il dimetilsolfossido provocano rigonfiamento nei tubi Tefzel, che riducono notevolmente il limite di pressione di rottura dei tubi stessi.

Advertencia: manipular con precaución los tubos de polímero bajo presión:

- Protegerse siempre los ojos en las proximidades de tubos de polímero bajo presión.
- Apagar todas las llamas que estén a proximidad.
- No utilizar tubos Tefzel que hayan sufrido tensiones extremas o hayan sido doblados.
- No utilizar tubos Tefzel con tetrahidrofurano (THF) o ácidos nítrico o sulfúrico concentrados.
- No olvidar que el cloruro de metileno y el óxido de azufre dimetilo dilatan los tubos Tefzel, lo que reduce en gran medida la presión de ruptura de los tubos.
- 警告:當在有壓力的情況下使用聚合物管線時,小心注意以下几點:
  - 當接近有壓力的聚合物管線時一定要戴防護眼鏡。
  - 熄滅附近所有的火焰。
  - 不要使用已經被壓癟或嚴重彎曲的特氟隆(Tefzel)管線。
  - 不要在特氟隆(Tefzel)管線中使用四氫呋喃(THF)或濃硝酸或濃硫酸。
  - 要了解使用二氯甲烷及二甲基亞楓會導致特氟隆(Tefzel)管線膨脹,大大降低管線的耐壓能力。



警告:当在有压力的情况下使用聚合物管线时,小心注意以下几点

- 当接近有压力的聚合物管线时一定要戴防护眼镜
- · 熄灭附近所有的火焰。
- 不要使用已经被压瘪或严重弯曲的特氟隆(Tefzel)管线。
- 不要在特氟隆(Tefzel)管线中使用四氢呋喃(THF)或浓硝酸或浓硫酸。
- 要了解使用二氯甲烷及二甲基亚砜会导致 特氟隆 (Tefzel) 管线膨胀, 大大降低管线 的耐压能力。

경고: 폴리머재질의 튜빙을 압력하에서 사용할 때는 다음 사항에 유의하십시오.

- 압력을 받은 폴리머 튜빙 부근에서는 반드시 보호안경을 착용할 것
- 모든 화기의 접근을 금함
- 눌리거나 뒤틀린 Tefzel 튜빙은 사용하지 말 것
- Tefzel 튜빙을 테트라히드로퓨란 (THF)이나 염산 및 황산과 함께 사용하지 말 것
- 디클로로메탄(methylene chloride)와 디메틸설폭시드(dimethyl sulfoxide)는 Tefzel 튜빙을 팽창시켜 쉽게 파열되므로 주의할 것
- 警告:ポリマーチューブに圧力をかけて取り扱う場合は、次のように注意してください。
  - 加圧したポリマーチューブの付近では、常に保護めがねを着用してください。
  - 付近の火はすべて消してください。
  - 激しい応力やねじれを受けたTefzelチューブは使用しないでください。
  - テトラヒドロフラン (THF)、濃硝酸、あるいは濃硫酸には、Tefzelチューブを使用 しないでください。
  - メチレン-クロライドやジメチルスルホキシドはTefzelチューブを膨張させ、 チューブの破断圧力を大幅に低下させますので、注意してください。



**Caution:** The user shall be made aware that if the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

**Attention :** L'utilisateur doit être informé que si le matériel est utilisé d'une façon non spécifiée par le fabricant, la protection assurée par le matériel risque d'être défectueuses.

**Vorsicht:** Der Benutzer wird darauf aufmerksam gemacht, dass bei unsachgemäßer Verwenddung des Gerätes unter Umständen nicht ordnungsgemäß funktionieren.

**Precauzione:** l'utente deve essere al corrente del fatto che, se l'apparecchiatura viene usta in un modo specificato dal produttore, la protezione fornita dall'apparecchiatura potrà essere invalidata.

**Advertencia:** el usuario deberá saber que si el equipo se utiliza de forma distinta a la especificada por el fabricante, las medidas de protección del equipo podrían ser insuficientes.

**警告**:使用者必須非常清楚如果設備不是按照制造廠商指定的方式使用,那么該設備所提供的保護將被消弱。

**警告**:使用者必须非常清楚如果设备不是按照制造厂商指定的方式使用,那么该设备所提供 的保护将被消弱

**경고 :** 제조사가 지정한 것 이외의 방법으로 기기를 사용하는 경우에는, 사용자가 위 험으로부터 보호될 수 없는 경우가 발생할 수 있음에 유념하십시오.

警告:ユーザは製造業者が指定していない方法で装置を使用した場合は装置が提供する 保護が損なわれることがあるということを承知しているものとします。



**Attention :** Remplacez toujours les fusibles par d'autres du même type et de la même puissance afin d'éviter tout risque d'incendie.

*Vorsicht:* Zum Schutz gegen Feuergefahr die Sicherungen nur mit Sicherungen des gleichen Typs und Nennwertes ersetzen.

**Precauzione:** per una buona protezione contro i rischi di incendio, sostituire i fusibili con altri dello stesso tipo e amperaggio.

Advertencia: sustituya los fusibles por otros del mismo tipo y características para evitar el riesgo de incendio.

警告:為了避免火災的危險,應更換同种類型及規格的保險絲。

**警告**:为了避免火灾的危险,应更换同种类型及规格的保险丝。

**경고 :** 화재를 방지하기 위해서는 퓨즈 교체 시 같은 종류, 같은 등급의 것을 사용하십 시오.

警告:火災の危険防止のために、ヒューズの交換は同一タイプおよび定格のもので行なってください。



*Caution:* To avoid possible electrical shock, disconnect the power cord before servicing the instrument.

**Attention :** Afin d'éviter toute possibilité de commotion électrique, débranchez le cordon d'alimentation de la prise avant d'effectuer la maintenance de l'instrument.

*Vorsicht:* Zur Vermeidung von Stromschlägen sollte das Gerät vor der Wartung vom Netz getrennt werden.

**Precauzione:** per evitare il rischio di scossa elettrica, scollegare il cavo di alimentazione prima di svolgere la manutenzione dello strumento.

**Precaución:** para evitar descargas eléctricas, desenchufe el cable de alimentación del instrumento antes de realizar cualquier reparación.

警告:要避免觸電,請在修理或保養器材前把電源線拔出。

警告: 为避免可能引起得触电危险, 在修理前请切断电源连接。

경고: 전기 충격의 가능성을 피하기 위해서는, 기기를 수리하기 이전에 전원 코드를 차단하십시오.

警告:感電の危険性を避けるために、装置の保守を行う前には装置の電源コードを引き抜いてください。

	Direct current Courant continu Gleichstrom Corrente continua Corriente continua 直流電 直流电 직류 直流
$\sim$	Alternating current Courant alternatif Wechselstrom Corrente alternata Corriente alterna 交流電 交流电 교류 交流
	Protective conductor terminal Borne du conducteur de protection Schutzleiteranschluss Terminale di conduttore con protezione Borne del conductor de tierra 保護的導線端子 保护性的接地端 보호 도체 단자 接地

### **Commonly Used Symbols**

### Commonly Used Symbols (Continued)

,	Frame or chassis terminal Borne du cadre ou du châssis Rahmen- oder Chassisanschluss Terminale di struttura o telaio Borne de la estructura o del chasis 結構或底盤端子 机架或底盘接地端 프레임 또는 틀 단자 フレームまたはシャーシアース
	Caution or refer to manual Attention ou reportez-vous au guide Vorsicht, oder lesen Sie das Handbuch Prestare attenzione o fare riferimento alla guida Actúe con precaución o consulte la guía 小心或査閱手冊 小心或査閱手册 경고 또는 사용설명서 참조 警告またはマニュアルを参照
	Caution, hot surface or high temperature Attention, surface chaude ou température élevée Vorsicht, heiße Oberfläche oder hohe Temperatur Precauzione, superficie calda o elevata temperatura Precaución, superficie caliente o temperatura elevada 警告,熱表面或高溫 警告,熱表面或高温 경고, 뜨거운 표면 또는 고온 警告、熱くなっている面、あるいは高温

### Commonly Used Symbols (Continued)

Â	Caution, risk of electric shock (high voltage) Attention, risque de commotion électrique (haute tension) Vorsicht, Elektroschockgefahr (Hochspannung) Precauzione, rischio di scossa elettrica (alta tensione) Precaución, peligro de descarga eléctrica (alta tensión) 警告,小心触電(高壓電) 警告,小心触电(高压电) 경고, 전기충격의 위험 (고압) 警告、電気ショックの危険性(高電圧)
	Caution, risk of needle-stick puncture Attention, risques de perforation de la taille d'une aiguille Vorsicht, Gefahr einer Spritzenpunktierung Precauzione, rischio di puntura con ago Precaución, riesgo de punción con aguja 警告,小心尖狀物刺傷 警告,小心尖狀物刺伤 경고, 뾰족한 것으로부터의 상해 위험 警告、ニードルで穴をあける危険性
	Caution, ultraviolet light Attention, rayonnement ultrviolet Vorsicht, Ultraviolettes Licht Precauzione, luce ultravioletta Precaución, emisiones de luz ultravioleta 警告,紫外光 결고, 자외선 警告、紫外線

### Commonly Used Symbols (Continued)

	Fuse Fusible Sicherung Fusibile Fusible 保険絲 保险丝 开즈 ヒューズ
1	Electrical power on Sous tension Netzschalter ein Alimentazione elettrica attivata Alimentación eléctrica conectada 開啓電源 接通电源 전원 켜기 <b>電源オン</b>
0	Electrical power off Hors tension Netzschalter aus Alimentazione elettrica disattivata Alimentación eléctrica desconectada 關閉電源 切断电源 전원 끄기 <b>電源オフ</b>

### **Intended Use**

Waters designed the Waters<sup>®</sup> Micromass<sup>®</sup>  $ZQ^{TM}$  Detector to with an HPLC system to determine mass-to-charge ratio (m/z) for a wide range of analytes.

### **Biological Hazard**

When you analyze physiological fluids, take all necessary precautions and treat all specimens as potentially infectious. Precautions are outlined in "CDC Guidelines on Specimen Handling," CDC - NIH Manual, 1984.

### Calibration

Follow the calibration methods set forth in this guide, using pure standards. The concentration range should cover the entire range of quality-control samples, typical and atypical specimens.

### **Quality Control**

Routinely run three quality-control samples. Quality-control samples should represent subnormal, normal, and above-normal levels of a compound. Ensure that quality-control sample results are within an acceptable range, and evaluate precision from day to day and run to run. Data collected when quality-control samples are out of range may not be valid.

# **Table of Contents**

Prefac	e	23
Chapte	er 1	
Overvi	ew	27
1.1	About the Micromass ZQ Detector	27
1.2	Theory and Principles of Operation	28
1.3	MassLynx 4.0 Software	29
Chapte	er 2	
Installi	ng	30
2.1	Site Selection and Power Requirements	31
2.2	Unpacking and Inspecting	32
2.3	Installing the Detector	33
	2.3.1 Installing the Rotary Pump	33
	2.3.2 Installing the Oil Return Connection Kit	34
	2.3.3 Connecting the Nitrogen Supply and Exhaust	35
	2.3.4 Connecting the Rheodyne Injector (for Manual	
	Injections)	37
	2.3.5 Installing the ESI Probe	38
	2.3.6 Connecting the Workstation	39
	2.3.7 Preparing the Syringe and Syringe Pump	40

Chapter 3

 Tuning		41
3.1 Opening	MassLynx and Starting the Instrument	41
3.2 Tuning ir	ESI Mode	43
3.2.1 S S	pecifying Parameter Settings on the ES+ ource Page	44
3.2.2 S A	pecifying Parameter Settings on the nalyser Page	46
3.3 Tuning ir	APCI Mode	47
3.3.1 P	reparing the Source for APCI Operation	48
3.3.2 Ir	stalling the Tee Fitting	49
3.3.3 S F	pecifying Parameters on the APCI+ Source age	49
3.4 Readbac	ks	52
Chapter 4 Calibrating		54
4.1 Setting l	Jp the Calibration File	54

### 

4.2.5 Mass Measure Dialog Box 62
4.2.6 Automatic Calibration Dialog Box
4.2.7 Calibration Acquisition Dialog Box

### Chapter 5

Maintaining73	'3
5.1 Considerations73	'3
5.2 Routine Maintenance	'5
5.2.1 Checking the Rotary Pump Oil	'5
5.2.2 Replacing the Pump Oil	'5
5.2.3 Gas-Ballasting the Rotary Pump	'8
5.2.4 Replacing the Oil Mist Filter	'9
5.2.5 Cleaning the Source Assembly	30
5.2.6 Cleaning the APCI Probe Tip	)1
5.2.7 Cleaning and Replacing the Corona Discharge	
Needle	)1
5.3 Replacing Parts92	)2
5.3.1 Replacing the Ion Block Cartridge Heater	)2
5.3.2 Replacing the Stainless Steel Capillary	)3
5.3.3 Replacing the ESI Probe Tip	)5
5.3.4 Replacing the APCI Fused Silica Capillary and	
Filter Pad	)6
5.3.5 Replacing the APCI Probe Heater	8
Chapter 6	
Troubleshooting	9

IOUDIE	eshooting	. 99
6.1	Safety and Handling	. 99
6.2	Component Hardware Troubleshooting	101
6.3	Inspecting the APCI Probe	107

Appendix A	
Using the ESCi Multi-Mode Ionization Source	80
A.1 Preparing for Operation10	80
A.1.1 Installing the Corona Discharge Needle	80
A.1.2 Setting Up MassLynx1	10
A.2 Daidzein Test 1	17
A.2.1 Test Conditions1	18
A.2.2 Signal-to-Noise Ratio1	18
Appendix B	
Specifications	19
B.1 ZQ Detector Specifications1	19
B.2 ESCi Multi-Mode Ionization Source Specifications 12	21
Appendix C	
Accessories and Spare Parts 12	22
Index12	29

## List of Figures

Installing the Detector	30
Pump Oil Level	33
Fitting the Gas Ballast and Hose adaptors to the Rotary Pump	35
ZQ Detector Rear Panel	36
Nitrogen Stud	36
Drying Gas Exhaust Bottle	37
Rheodyne Injector	38
ESI Probe in Situ	39
Syringe Pump	40
MassLynx Login Dialog Box	41
MassLynx Main Window	42
Tune Window Displaying the ES+ Source Page	43
Syringe Selection Dialog Box	45
Tune Window Displaying the Analyser Page	47
Combined Flow into the Tee	49
Tune Window Displaying the APCI+ Source Page	50
Readbacks Dialog Box	52
Diagnostics Page Readbacks	53
Calibration Window	54
Open Dialog Box	55
Tune Window Displaying the ES+ Source Page	57
Tune Window Displaying Analyser Page Parameters	58
Instrument Threshold Settings Dialog Box	59
Automatic Calibration Check Dialog Box	59
Calibration Parameters Dialog Box	60
Mass Measure Dialog Box	62
Automatic Calibration Dialog Box	63
Calibration Acquisition Setup Dialog Box	65
	Installing the Detector

4-11	Display Calibration Graphs Dialog Box	67
4-12	Calibrate Window Showing ZQ-4000 Calibration Graphs	69
5-1	Rotary Pump Assembly Fitted with Oil Mist Filter	76
5-2	Rotary Pump Oil Filler Plug, Drain Plug, and Sight Glass	77
5-3	Isolation Valve	79
5-4	Oil Mist Filter Assembly	80
5-5	Pumping Block Assembly	82
5-6	Ion Block Assembly	83
5-7	Probe Assembly in Position on the Source	84
5-8	ZQ Detector Front View	85
5-9	Source Showing the Corona Discharge Needle	86
5-10	Ion Block	86
5-11	Sample Cone and Cone Gas Nozzle	87
5-12	Ion Block Rear View	88
5-13	Hexapole Assembly	89
5-14	Ion Block Cartridge Heater	92
5-15	Replacing the Heater Cartridge	93
5-16	ESI Probe Tip with Capillary Protruding 0.5 mm	95
A-1	ZQ Mass Detector, Front View	109
A-2	Installing the Corona Needle	110
A-3	Tune Window	110
A-4	Tune Window Showing Options List with ESCi Mode Selected	. 111
A-5	Tune Window, Diagnostics Page	112
A-6	Selecting the ESCi+ or ESCi- Ionization Mode	112
A-7	Selecting the Ion Mode in the MassLynx Peak Editor	113
A-8	Tune Window as it Appears During ESCi Operation	114
A-9	Function List Editor Window (Blank)	115
A-10	Function: <i>n</i> MS Scan Dialog Box	115
A-11	Function List Editor Window Showing Specified Functions	116
A-12	ESCi Mode Disabled	117
A-13	Daidzein (m/z = 255.2 [M + H] and 253.2 [M – H])	117

## List of Tables

2-1	Installation Site Requirements
3-1	ES+ Source Page Parameters43
3-2	Source and Desolvation Temperature Settings 44
3-3	Analyser Page Parameters 46
3-4	APCI+ Source Page Parameters 49
4-1	Recommended Calibration Acquisitions Setup Parameters65
4-2	Calibration Failure Troubleshooting 69
5-1	Maintenance Schedule74
6-1	Hardware Troubleshooting101
A-1	HPLC Conditions118
A-2	MS Conditions 118
B-1	ZQ Detector Operational Specifications119
B-2	ZQ Detector Environmental Specifications 120
B-3	ZQ Detector Dimensions 120
B-4	ZQ Detector Electrical Specifications 120
B-5	ESCi Environmental Specifications 121
B-6	ESCi Electrical Specifications 121
C-1	Fuses
C-2	Probe Components 122
C-3	Vacuum Components 123
C-4	Valves and Flow Meters 124
C-5	Kits 125
C-6	Power Supplies 125

C-7	Source and Analyser Components	126
C-8	Washers, Screws, and Nuts	127
C-9	Miscellaneous Components	128

## Preface

The Waters Micromass ZQ Detector Operator's Guide describes procedures for unpacking, installing, using, maintaining, and troubleshooting the Waters<sup>®</sup> Micromass<sup>®</sup> ZQ<sup>™</sup> Detector. Its appendixes explain how to use the optional ESCi<sup>™</sup> Multi-Mode Ionization Source, and list instrument specifications, accessories, and spare parts.

This guide is intended for individuals who need to install, operate, maintain, and/or troubleshoot the Micromass ZQ Detector.

### **Organization**

This guide contains the following:

<u>Chapter 1</u> describes the instrument, including its features and options.

<u>Chapter 2</u> describes how to unpack and install the instrument and how to make power, liquid line, gas, signal, and other hardware connections.

<u>Chapter 3</u> describes how to configure the instrument, start it operating, and tune and calibrate it.

<u>Chapter 4</u> describes how to set up a calibration file and specify calibration parameters in MassLynx<sup>TM</sup>.

Chapter 5 explains routine maintenance procedures.

Chapter 6 describes troubleshooting procedures.

<u>Appendix A</u> explains how to use the ESCi Multi-Mode Ionization Source option.

Appendix B presents instrument specifications.

Appendix C lists recommended and optional accessories and spare parts.

### **Related Documentation**

*Waters Licenses, Warranties, and Support:* Provides software license and warranty information, describes training and extended support, and tells how Waters handles shipments, damages, claims, and returns.

### **Online Documentation**

**MassLynx Help:** Describes all MassLynx windows, menus, menu selections, and dialog boxes for the base software and software options. Also includes reference information and procedures for performing all tasks required to use MassLynx software. Included as part of the MassLynx software.

#### **Printed Documentation for Base Product**

MassLynx User's Guide

MassLynx Interfacing Guide

MassLynx Security User's Guide

MassLynx 4.0 Guide to Inlet Control

MassLynx 4.0 Guide to ZQ Data Acquisition

Waters Micromass ZQ with MassLynx v4.0 Software and Instrument Verification Procedure

### **Related Adobe Acrobat Reader Documentation**

For detailed information about using Adobe<sup>®</sup> Acrobat<sup>®</sup> Reader, see the *Adobe Acrobat Reader Online Guide*. This guide covers procedures such as viewing, navigating, and printing electronic documentation from Adobe Acrobat Reader.

### **Printing This Electronic Document**

Adobe Acrobat Reader lets you easily print pages, page ranges, or the entire document by selecting **File > Print**. For optimum print quantity, Waters recommends that you specify a PostScript<sup>®</sup> printer driver for your printer. Ideally, use a printer that supports 600 dpi print resolution.

### **Documentation Conventions**

Convention	Usage
Purple	Purple text indicates user action such as keys to press, menu selec- tions, and commands. For example, "Click Next to go to the next page."
Italic	Italic indicates information that you supply such as variables. It also indicates emphasis and document titles. For example, "Replace <i>file_name</i> with the actual name of your file."
Courier	Courier indicates examples of source code and system output. For example, "The SVRMGR> prompt appears."
Courier Bold	Courier bold indicates characters that you type or keys you press in examples of source code. For example, "At the LSNRCTL> prompt, enter set password oracle to access Oracle."

The following conventions can be used in this guide:

Convention	Usage
Underlined Blue	Indicates hypertext cross-references to a specific chapter, section, subsection, or sidehead. Clicking this topic using the hand symbol brings you to this topic within the document. Right-clicking and selecting <b>Go Back</b> from the shortcut menu returns you to the originating topic. For example, "Monitoring Readbacks are described in <u>Section 3.4, Readbacks</u> "
Keys	The word <i>key</i> refers to a computer key on the keypad or keyboard. <i>Screen keys</i> refer to the keys on the instrument located immediately below the screen. For example, "The A/B screen key on the 2414 Detector displays the selected channel."
	Three periods indicate that more of the same type of item can optionally follow. For example, "You can store <i>filename1</i> , <i>filename2</i> , in each folder."
>	A right arrow between menu options indicates you should choose each option in sequence. For example, "Select File > Exit" means you should select File from the menu bar, then select Exit from the File menu.

#### Notes

Notes call out information that is helpful to the operator. For example:

Note: Record your result before you proceed to the next step.

### Attentions

Attentions provide information about preventing damage to the system or equipment. For example:



Attention: To avoid damaging the detector flow cell, do not touch the flow cell window.

### Cautions

Cautions provide information essential to the safety of the operator. For example:



*Caution:* To avoid burns, turn off the lamp at least 30 minutes before removing it for replacement or adjustment.



*Caution:* To avoid electrical shock and injury, unplug the power cord before performing maintenance procedures.



*Caution:* To avoid chemical or electrical hazards, observe safe laboratory practices when operating the system.

# Chapter 1 Overview

This chapter describes the Waters<sup>®</sup> Micromass<sup>®</sup> ZQ<sup>™</sup> Detector, its features and options.

## 1.1 About the Micromass ZQ Detector

The ZQ Detector is a quadrupole mass analyser that can determine the mass-to-charge ratio (m/z) of diverse analytes. An HPLC system, or syringe pump, delivers liquid sample to the instrument's analyser source. There the sample molecules ionize by means of one of two ionization modes: electrospray (ESI) or atmospheric pressure chemical ionization (APCI). In ESI mode, sample molecules ionize in solution before they reach the source. On entering the evacuated source, they begin a desolvation process. In APCI mode, an electrical discharge inside the source ionizes the sample molecules whereupon they undergo desolvation.

The ions ultimately reach the quadrupole, which separates them according to their mass-to-charge ratios. A photomultiplier then detects the mass-separated ions, amplifies their signals, and sends the mass information to the data system.

### **Probes**

An electrospray ionization (ESI) probe or an atmospheric pressure chemical ionization (APCI) probe introduces the sample to the ion source.

### **Sample Inlet**

Either of two methods deliver solvent and sample to the installed probe:

- An HPLC system Delivers the eluent from an HPLC analysis.
- A built-in syringe pump Delivers standard solutions or infusions of unknown samples.

### Vacuum System

An external rotary (roughing) pump and an internal split flow turbomolecular pump combine to create the source vacuum. The turbomolecular pump evacuates the analyser and ion transfer region.

Vacuum leaks and electrical or vacuum pump failures cause vacuum loss, which protective interlocks guard against. The system monitors turbomolecular pump speed and continuously measures vacuum pressure with a built-in Pirani gauge. The gauge also serves as a switch, discontinuing detector operation when it senses vacuum loss.

A vacuum isolation valve isolates the source from the mass analyser, allowing routine source maintenance without venting.

### Mass Analyser (Quadrupole)

The mass analyser separates ions by mass-to-charge ratio (m/z).

### **Data System**

The data system collects information from the mass analyser and includes these components:

- MassLynx<sup>™</sup> 4.0 software
- An external workstation
- An embedded PC

MassLynx software controls the workstation-based data system and mass detector through the detector's embedded PC. Using MassLynx, you tune the instrument, set up and run the HPLC system, and acquire and process data. When they are part of the system, the software also controls the autosampler and the divert and injector valves.

The workstation uses a Windows NT<sup>®</sup>, Windows<sup>®</sup> 2000, or Windows XP color graphical environment and allows full user interaction with the keyboard or mouse. A network link communicates between the workstation and the detector's embedded PC.

MassLynx acquires and stores data from conventional LC detectors simultaneously with data the mass detector acquires. It can also acquire data from selected systems, like Waters 996/2996 Photodiode Array Detectors. Consult the *MassLynx 4.0 Guide to Inlet Control* for details about MassLynx.

## 1.2 Theory and Principles of Operation

### **Electrospray Ionization (ESI)**

In ESI, a high electrical voltage charges the eluent as it emerges from a nebulizer, producing an aerosol of charged droplets. As the solvent evaporates, the droplets shrink, developing a charge dense enough to eject ions from their surfaces (ion evaporation). The mass analyser then sorts the singly or multiply charged ions by mass-to-charge m/z ratio.

The analyser source can accommodate eluent flows of up to 1 mL/min. You can enhance performance by reducing the rate of eluent flow at the ion source.

### **Atmospheric Pressure Chemical Ionization (APCI)**

A heated nebulizer vaporizes the sample. The sample ions then merge with solvent ions in the atmospheric source, enabling proton transfers between the solvent and sample ions.

APCI generally produces both protonated and deprotonated molecular ions from the sample. For positive ions, this ionization occurs by means of a proton transfer mechanism. For negative ions, the mechanism is proton abstraction.

## 1.3 MassLynx 4.0 Software

MassLynx 4.0 software permits these major operations:

- Configuring the instrument
- Creating HPLC inlet and MS methods that define operating parameters for a run
- Tuning and calibrating the mass detector
- · Running samples
- Monitoring the run
- · Acquiring data
- Processing data
- Reviewing data
- · Printing data

See the *MassLynx 4.0 Guide to Inlet Control* and *MassLynx Help* for more information on installing and using MassLynx software.

# Chapter 2 Installing

This chapter describes how to unpack and install your Waters Micromass ZQ Detector. <u>Figure 2-1</u> summarizes these procedures.



Figure 2-1 Installing the Detector

## 2.1 Site Selection and Power Requirements

Install the detector on a stable, level, and appropriately clean surface that meets the specifications in <u>Table 2-1</u>.

Factor	Requirement
Temperature range	15 to 28 °C (59 to 82.4 °F)
Relative humidity range	20 to 80%, noncondensing
Bench space	Width: 15.2 in. (38.1 cm)
	Depth: 26 in. (66.1 cm)
	Height: 23 in. (57.2 cm)
	Weight: 210 lb. (95.3 kg)
Clearance	Rear: 4.75 in. (120 mm)
	Right side: 20 in. (0.5 m) to allow for service access.
	<i>Note:</i> Movable equipment can be located as close as 4.75 in. (120 mm).
	Left side: 0.0 in./mm
	Top: 11 in. (28 cm)
Power requirements	Grounded AC, 230 V, 50/60 Hz
Electromagnetic fields	No nearby source of electromagnetic noise, such as NMR
Static cloctricity	Nogligible
Vibration	Negligible

#### Table 2-1 Installation Site Requirements



**Attention:** To avoid overheating the instrument, allow the prescribed clearances at the top, sides, and rear of the instrument.



**Attention:** To avoid damaging the instrument, measure the voltage at the 230 VAC outlet before installing the instrument. Readings of less than 208 VAC indicate the need for a step-up transformer.

## 2.2 Unpacking and Inspecting

The Waters Micromass ZQ system is shipped in several cartons. Among them, they contain these items:

- Micromass ZQ Detector with Startup Kit
- Rotary pump
- MassLynx workstation
- MassLynx 4.0 documentation set
- Waters Micromass ZQ Detector Operator's Guide

### **Required Material**

Utility knife or scissors

### Procedure

- 1. Note any tipping or shock indicators on the cartons that shipping might have triggered. Also, inspect the carton for damage.
- 2. Cut and remove the straps that secure the large carton.
- 3. Lift the carton off the pallet.
- 4. Remove foam packing material from the top of the instrument.
- 5. Remove the Startup Kit, and set it aside.
- 6. Lift the detector from the foam support on the pallet and carefully set the unit down on a bench.



*Caution:* At least four people must lift the instrument from its shipping pallet and place it on the bench.

**Note:** The instrument should overhang the bench top by several inches to allow the waste tube, which extends from the lower-right corner, a straight descent to the liquid waste container.

- 7. Check the Startup Kit contents against the accompanying parts list to confirm that all items are included.
- 8. Open the carton containing the rotary pump, then remove the packing material and Startup Kit.
- 9. Carefully remove the rotary pump, and set it temporarily down on a level surface.

### **Contacting Waters Technical Service**

Inspect all items for damage. Immediately report any shipping damage to both the carrier and Waters. North American customers who report damage should contact Waters Technical Service at 800 252-4752. All others should call their local Waters subsidiary or Waters corporate headquarters in Milford, Massachusetts (U.S.A.).

## 2.3 Installing the Detector

This section describes how to install the Waters Micromass ZQ 2000 and ZQ 4000 Detectors.

### 2.3.1 Installing the Rotary Pump



Attention: The rotary pump is shipped without oil. You must fill it with oil before starting it.

- 1. Place the rotary pump on the floor, within 5 feet of the instrument. Place the PTFE drip tray beneath the pump.
- 2. Fill the pump with oil:
  - a. Remove the filler plug.
  - b. Pour oil into the pump until the level reaches the MAX mark on the bezel at the oil-level sight glass. If the level exceeds the MAX mark, remove the drain plug, and let the excess oil drain (<u>Figure 2-2</u>).
  - c. After a few minutes, recheck the oil. If the level is now below the MAX mark, add the appropriate amount of oil.
  - d. Refit the oil filler plug. Finger tighten it. Do not overtighten it.



Figure 2-2 Pump Oil Level

**Note:** Use Ultragrade 19 or Inland Q45 oil only. Refer to the manufacturer's manual for more information about filling the pump with oil.

- Attach the NW25 tee, included in the Startup Kit, to the inlet of the rotary pump using the NW25 center ring and clamp (<u>Figure 5-1</u>).
- 4. Attach a length of 1-inch ID vacuum hose to each open port on the NW25 tee. Use the NW25 flanges, center rings, and clamps provided in the Startup Kit.
- 5. Connect the opposite ends of the two lengths of vacuum hose to the two straight NW25 vacuum ports on the detector's rear panel. Use NW25 flanges, center rings, clamps, and elbows, as necessary.
- 6. Remove the nozzle fitting on the pump exhaust port, and replace it with the NW25 flange and O-ring from the Startup Kit.

*Note:* Ensure the NW25 flange is tight. Pump vibration can loosen it, causing oil to leak.

- 7. Connect the oil mist filter assembly to the pump exhaust port. Use an NW25 center ring and clamp.
- 8. Connect an NW25 nozzle fitting to the oil mist filter assembly. Use an NW25 center ring and clamp.
- 9. Connect the 12-mm clear PVC exhaust tubing to the NW25 nozzle fitting. Secure the tubing with a hose clamp.
- 10. Route the open end of the exhaust tubing to a suitable exhaust vent.
- 11. Remove the drain plug and bonded seal from the oil mist filter housing. Save the drain plug for future use.
- 12. Connect the female end of the rotary pump power cord to the connector on the rotary pump relay box. Connect the male end of the power cord to the rotary pump connector on the instrument's rear panel.
- 13. Turn the power switch to On.

*Note:* The pump will not start at this time. It is controlled by the system software.

### 2.3.2 Installing the Oil Return Connection Kit

The oil return connection kit collects excess oil from the oil mist filter housing and returns it to the rotary pump oil reservoir.

 Install the drain adaptor and bonded seal onto the oil mist filter drain port (Figure 2-3).



- 1. E1/E2M Pump
- 2. Filters
- 3. Wire Mesh
- 4. Circlip
- 5. O-Ring
- 6. Gas Ballast Adaptor
- 7. Bonded Seal
- 8. Hose Adaptor
- 9. Bonded Seal
- 10. Banjo Bolt

Figure 2-3 Fitting the Gas Ballast and Hose adaptors to the Rotary Pump

- 2. Remove the circlip, wire mesh, and filters from the gas ballast inlet on the pump.
- 3. Insert the gas ballast adaptor and O-ring into the gas ballast inlet port on the pump.
- 4. Connect the hose adaptor to the gas ballast adaptor. Use the banjo bolt and the two bonded seals.
- Estimate how much flexible oil return tubing you need to loosely connect the oil mist filter housing's drain port to the pump's inlet port. When fitted, the tube must be free of kinks or tight bends.
- 6. Cut the tubing, ensuring the cut ends are burr-free and square.
- 7. Lubricate the restrictor with oil, then insert it into one end of the tube.
- 8. Connect one end to the drain adaptor on the oil mist filter housing, and the other end to the hose adaptor.
- 9. Secure the ends of the tube with hose clips.

### 2.3.3 Connecting the Nitrogen Supply and Exhaust

 Connect one end of a length of the 6-mm PTFE tubing to the N<sub>2</sub> In port on the rear of the instrument (<u>Figure 2-4</u>).



Figure 2-4 ZQ Detector Rear Panel

 Attach a nitrogen regulator to the nitrogen supply, and install the 6-mm stud (Figure 2-5) into the regulator outlet.



Figure 2-5 Nitrogen Stud

- 3. Connect the free end of the 6-mm PTFE tubing to the 6-mm stud.
- 4. Locate the drying gas exhaust bottle (Figure 2-6) in an accessible area.
- 5. Cut a length of 10-mm tubing long enough to connect the instrument to the drying gas exhaust bottle. Connect one end of the tubing to the exhaust port on the rear panel. Connect the other end to one of two ports on the drying gas exhaust bottle.
- 6. Cut a second length of 10-mm tubing long enough to connect the drying gas exhaust bottle to the exhaust vent. Insert one end of the tubing into the remaining port on the drying gas exhaust bottle. Route the other end to the exhaust vent.


Figure 2-6 Drying Gas Exhaust Bottle

**Attention:** The instrument requires two separate exhaust systems, one for nitrogen, the other for the rotary pump. Vent them to atmosphere through separate exhaust lines. Oil mist can seriously damage the instrument when the nitrogen exhaust line connects with the rotary pump exhaust line. Your warranty does not cover damage caused by routing exhaust lines incorrectly.

7. Route the PTFE waste tubing from the detector's lower-right corner to a suitable liquid waste container.

# 2.3.4 Connecting the Rheodyne Injector (for Manual Injections)

Refer to Figure 2-7 for this procedure.

- 1. Connect the PTFE waste tube to injector port 5.
- 2. Install the needle port fitting onto injector port 6.
- 3. Connect the 10-µL injection loop between injector ports 1 and 4.
- 4. Connect the LC system tubing to injector port 2.



Figure 2-7 Rheodyne Injector

### 2.3.5 Installing the ESI Probe

Refer to Figure 2-8 for this procedure.

- 1. Connect the PTFE tubing from the probe adjustment flange to the desolvation gas port on the front panel.
- 2. Remove the protective sleeve, if fitted, from the electrospray probe tip.
- 3. Slide the probe into the hole in the probe adjustor plate until the probe body rests on the probe adjustment flange. The probe identification contacts must touch the screws on the probe adjustment flange.
- 4. Secure the probe with the two knurled thumbscrews.
- 5. Connect the 4-mm PTFE tubing from the probe to the nebulizer gas port.
- 6. Connect the electrical lead from the probe to the capillary connector on the front panel.



Figure 2-8 ESI Probe in Situ

# 2.3.6 Connecting the Workstation

Waters ships the workstation with preinstalled MassLynx software. Before connecting the workstation to the instrument, set it up according to its accompanying instructions. You should locate the workstation within 16 feet (5 meters) of the instrument.

- 1. Connect one end of the network cable to the appropriate port on the rear panel of the detector.
- 2. Connect the other end of the network cable to the port labeled ZQ on the workstation rear panel.



**Caution:** Do not connect the instrument's power supply cord until you complete the installation procedures in the previous sections.

To connect the instrument to the power source:

- 1. Select the correct power cord for your location.
- 2. Connect the female end of the power cord to the power port on the rear panel of the instrument.

# 2.3.7 Preparing the Syringe and Syringe Pump

This section refers to the 250-µL Hamilton syringe, various syringe fittings, and the API Setup Solution (polypropylene glycol/reserpine/cyclodextrin) found in the Startup Kit.

- 1. Flush the syringe three times with methanol or a volume-to-volume mixture of 70% methanol : 30% water.
- 2. Load the syringe with the setup solution, and connect the Rheodyne 9013 needle port fitting to the PEEK union, finger tightening it.
- 3. Clip the ground cable (with the plug-in clip), located on the lower-right side of the front panel, into the syringe needle.



**Caution:** To avoid electrical shock, always ground the needle.

 Fit the syringe into the syringe pump, and set the syringe stop accordingly (<u>Figure 2-9</u>).



*Caution:* The syringe pump includes a positive syringe stop to prevent certain syringe types from breaking. Nevertheless, as added protection against syringe breakage, you should set the syringe stop adjustor. This prevents the syringe plunger from traveling its full stroke inside the syringe barrel, reducing the potential for breakage.



Figure 2-9 Syringe Pump

# Chapter 3 Tuning

Tuning involves adjusting source settings, analyser settings, and gas flows to produce optimal peak intensities.

After you tune, calibrate the instrument in electrospray (ESI) mode, even if you intend to operate it in APCI mode. See <u>Section 3.3.3</u> for details about tuning in APCI mode.

# 3.1 Opening MassLynx and Starting the Instrument

 Double-click the MassLynx V4.0 desktop icon to open the application. The MassLynx Login dialog box appears (<u>Figure 3-1</u>).

MassLynx	Login		×
	MassLynx <sup>T</sup> Glol Mass-INFO	A.0 Dal RMATICS	
<b>10</b>	Type a logon nai	me and password to log in.	OK Cancel
	<u>L</u> ogon Name:	DG02	
	<u>P</u> assword:	****	
	<u>D</u> omain	M-1	

Figure 3-1 MassLynx Login Dialog Box

2. Complete the Logon Name, Password, and Domain fields.

**Note:** The Login dialog box appears only when you enable MassLynx security. Otherwise, the MassLynx Main window (*Figure 3-2*) appears after you click the MassLynx V4.0 desktop icon. See the MassLynx Security User's Guide (version 4.0) for details about enabling MassLynx security.



3. Click **OK**. The MassLynx Main window appears (Figure 3-2).

Figure 3-2 MassLynx Main Window

**Note:** After initiating, the Main window displays "Instrument Present" in the status bar.

- 4. The shortcut bar should appear in the MassLynx Main window, and "Instrument" should appear at its top. If it fails to appear, click Shortcut, in the toolbar, to open it. Then click Instrument, at the left edge of the shortcut bar.
- Select MS Tune from the Main window (<u>Figure 3-2</u>) shortcut bar to open the Tune window (<u>Figure 3-3</u>).

ZQ - d:\empower\zq\zq.pro\acq File _lon Mode _Calibration _Gas Ramps _Options	udb\default.ipr Elep				
	🔼 🛅 II 🕘				
ES+Source Analyser Diagnostics		Mass Sp	an Gain		
Vokages		175.1 5	10		
Capillary (kV) 3.55 3.50		603.9 5	10		
Cone (V) 66 60 -		2034.6 5	10		
Extractor (V) 3 3		,			
EF Lens (V) 0.0		175.1 📓	609.3 X	1080.8	2034.6
Temperatures					
Source <u>I</u> emp (°C) 110 80					
Desolvation Temp (*C) 149 150					
Gas Flow Desolvation (L/hr) Cone (L/hr)					
311 E					
	174	.0 176.0	608.0 610.0	1080.0 1082.5	032.5 2035.0
					Press for Standby
Ready		Vacuum DK		Operate	1.

Figure 3-3 Tune Window Displaying the ES+ Source Page

# 3.2 Tuning in ESI Mode

<u>Table 3-1</u> describes the Tune window's ES+ Source page parameters.

**Note:** The voltage parameters shown in this table optimize sensitivity and stability. The temperature and flow rate parameters control the extent of solvent evaporation and adduct formation.

Table 3-1 ES+ Source Page Paramete
------------------------------------

Parameter	Description
Capillary voltage	Enhances or suppresses ion density by supplying excess charge to droplets. Optimal voltages: 2 to 4 kV for positive ions and 2 to 3 kV for negative ions.
Cone voltage	Helps draw ions into the first vacuum region (20 to 70 V optimal).
Extractor voltage	Focuses ions toward the hexapole RF lens (3 to 10 V optimal). Increasing voltage can induce fragmentation.
RF Lens voltage	Focuses ions toward the center of the quadrupole (~0.5 V).

Table 3-1 ES+ Source Page Parameters (Continued)

Parameter	Description
Source temperature	See <u>Table 3-2</u> .
Desolvation temperature	See <u>Table 3-2</u> .
Desolvation gas flow	Optimizes gas flow depends on mobile phase composition and flow rate (>100 L/hr).
Cone gas flow	Helps reduce adduct ions and keep the sample cone clean (50 to150 L/hr).

#### **Source and Desolvation Temperatures**

The Source and Desolvation temperature settings control desolvation for a specified flow rate. <u>Table 3-2</u> gives temperature ranges for specific flow rates.

HPLC Flow Rate (µL/hr)	Source Temperature °C	Desolvation Temperature °C
<100	80 to 100	100 to 350
100 to 250	100 to 130	350 to 400
250 to 1000	130 to 150	400 to 450

Table 3-2 Source and Desolvation Temperature Settings

### 3.2.1 Specifying Parameter Settings on the ES+ Source Page

1. Click [1] (API Gas) in the Tune window to toggle the nitrogen flow to On.



Attention: You must toggle API gas to Off before reopening the nitrogen supply once it has been shut off. Otherwise, the sudden inrush of gas can damage the flow meter.

 Make sure the instrument is operating, noting whether peak activity appears in the Tune window's Peak Display area (Figure 3-3). If the instrument is not operating (indicated by no peaks), click Press for Operate. 3. Click the ES+ Source tab, and specify these suggested starting parameters in the corresponding fields of the ES+ Source page.

Parameter	Suggested Value
Capillary (kV)	3.5
Cone (V)	60
Extractor (V)	3
RF Lens (V)	0.5
Source Temp (°C)	80
Desolvation Gas Flow (L/hr)	300
Desolvation Temp (°C)	150
Cone Gas Flow (L/hr)	50

STOP

**Attention:** Monitor readbacks, letting the source temperature, desolvation gas flow, desolvation temperature, and cone gas flow reach their setpoints before proceeding. See <u>Section 3.4</u> for details about monitoring readbacks.

4. Select all four Peak Editor fields, and enter these mass assignment values.

Mass	Span	Gain
175.1	5	10
609.3	5	10
1080.8	5	10
2034.6	5	10

 Select Options > Syringe Type. The Syringe Selection dialog box appears (Figure 3-4).

Syringe Selection		
Hamilton 250ul	<b>.</b>	
Tranicon 23002		<u>E</u> dit

Figure 3-4 Syringe Selection Dialog Box

- 6. Ensure the drop-down list displays Hamilton 250uL, then click OK.
- Set the syringe pump to deliver 10 μL/min by entering that rate in the Pump Flow field of the Analyser page (<u>Figure 3-5</u>).
- 8. Click **[1]** (Syringe pump) to infuse the startup solution into the source.
- 9. Click the Analyser tab, and ensure the syringe pump flow rate is 10 µL/min.
- 10. Turn the probe adjustor (see <u>Figure 2-8</u>) in one direction or the other to optimize peak intensities.
- 11. Adjust the capillary, cone, extractor, and RF lens voltages to optimize peak intensities.

# 3.2.2 Specifying Parameter Settings on the Analyser Page

Analyser settings optimize mass peak resolution. With span settings at 5, the bases of mass peaks as they appear on the Tune window Analyser page should measure 1 da.

Note: The parameters LM Resolution, HM Resolution, and Ion Energy optimize resolution.

Table 3-3 describes the Tune window's Analyser page parameters.

Parameter	Description
LM Resolution HM Resolution	Affect mass peak concentration. Usually, 15, an arbitrary unit, yields adequate mass resolution. Increasing the value lowers sensitivity; decreasing it enhances sensitivity.
Ion Energy (V)	Decreases resolution. Set between –1 and 3 V, as low as possible without reducing peak intensity.
Multiplier (V)	Modifies gain (attenuation). Settings between 400 and 450 yield a suitable signal-to-noise balance.

Table 3-3 Analyser Page Parameters

1. Click the Analyser tab. The Tune window Analyser page appears (Figure 3-5).

ZQ - c:\MassLynx\default.pro\acqudb\defau	ult.ipr				
	· Helt				
ES+ Source Analyzer Diagnostics Analyzer LM Resolution 150 HM Resolution 150		Mass         Sp           175.13         5           609.27         5           1080.8         5           2034.63         5	Gain 8 20 40 39		
Ion Energy 0.3	9.35e6	175.1 X	609.3 X 3.18e6 ×20	1080.8 X 1.41e6 ×40	2034.6 X 8.78e5 X39
Syringe Pump Flow (uL/min) 10.0 Syringe Status					
	174.	0 176.0	608.0 610.0	1080.0 1082.5	032.5 2035.0
					Press for Standby
Ready		Vacuum OK		Operate	14

Figure 3-5 Tune Window Displaying the Analyser Page

2. Specify these suggested starting parameters in the Analyser page fields.

Parameter	Suggested Value
LM Resolution	15
HM Resolution	15
Ion Energy	0.3
Multiplier	650

**Note:** If you change HM and/or LM Resolution after calibrating the instrument, you should recalibrate. Otherwise, any data the instrument acquires could fall outside the calibration mass range.

# 3.3 Tuning in APCI Mode

After you tune and calibrate (see <u>Chapter 4</u> for calibration information) in ESI mode, you may tune for APCI operation. This entails preparing the source, installing a tee fitting upstream of the probe, and specifying APCI parameters.

# 3.3.1 Preparing the Source for APCI Operation

To prepare the source for APCI tuning when the instrument is in ESI mode:

- 1. Prepare the syringe according to the procedure in <u>Section 2.3.7</u>, loading it with API Setup Solution from the Startup Kit.
- 2. Click Press for Standby on the lower right of the Tune window.
- 3. Disconnect the nebulizer gas line and both electrical connections from the front panel.
- 4. Remove the ESI probe.
- 5. Insert the APCI probe into the source, tightening the two thumbscrews.
- 6. Connect the APCI nebulizer and desolvation gas lines at the front panel.
- 7. Open the source enclosure cover.



*Caution:* The ion source block is hot. It can reach 150 °C, maintaining its set temperature, even with the source enclosure removed.

- Remove the blanking plug from the corona pin mounting contact, and fit the corona discharge pin (<u>Figure 5-9</u>). Align the tip of the corona discharge pin with the tip of the sample cone.
- 9. Close the source enclosure cover.
- 10. Connect the electrical lead to the Source/Probe receptacle on the front panel.
- 11. Click Press for Operate.

The source is now ready for APCI operation.



Attention: Do not start the liquid flow until the gas flow and probe heater are switched on and the probe inserted.

# 3.3.2 Installing the Tee Fitting

To optimize APCI peaks, temporarily install a tee fitting to merge the sample flow from the syringe pump with the solvent flow from an HPLC pump. The combined sample/solvent stream flows into the probe (Figure 3-6).



Figure 3-6 Combined Flow into the Tee

- 1. Connect the HPLC pump tubing to injector port 2 (see Figure 2-7 for port positions).
- 2. Connect the capillary from the syringe pump to one of the tee's three ports.
- Connect a 1/16-in. OD x 0.007 in. ID x 1 M tube between injector port 3 and a second tee port.
- 4. Connect another 1/16-in. OD × 0.007 in. ID × 1 M tube from APCI probe to the tee's third port.

#### 3.3.3 Specifying Parameters on the APCI+ Source Page

<u>Table 3-4</u> describes the APCI+ Source page parameters.

Parameter	Description
Corona	Affects sensitivity. The amount of current required depends on the polarity of both the compound and mobile phase. Optimize when mobile phase is present.
APcl Probe Temp	Affects sensitivity. Start at 650 °C and reduce in 50° steps, allowing time for stabilization to take place before reading. Optimize while mobile phase is flowing.
Desolvation	Gas flow rate usually affects signal intensity only marginally. Nevertheless, adjusting it can suppress chemical background noise.
Cone Gas	Gas flow rate can minimize formation of solvent adducts.

Table 3-4	APCI+	Source	Page	Parameters
-----------	-------	--------	------	------------

 Select Ion Mode > APCI+ from the Tune window (Figure 3-3). The APCI+ Source page appears (Figure 3-7).



Figure 3-7 Tune Window Displaying the APCI+ Source Page

2. Click [1] (API Gas) in the Tune window to toggle the nitrogen flow to On.

**Stop** Attention: You must toggle API gas to Off before reopening the nitrogen supply once it has been shut off. Otherwise, the sudden inrush of gas can damage the flow meter.

**Note:** If you do not observe any peak activity, the instrument may not be in operational mode. To remedy this, click **Press for Operate**.

3. Specify these suggested parameters in the APCI+ Source page fields.

Parameter	Suggested Value
Corona (µA)	0.1
Cone (V)	48
Extractor (V)	3
RF Lens (V)	0.0
Source Temp (°C)	120
Desolvation Gas Flow (L/hr)	300
APcl Probe Temp (°C)	500
Cone Gas Flow (L/hr)	50



Attention: Before proceeding, let the source temperature, desolvation gas flow, desolvation temperature, and cone gas flow reach their setpoints. See <u>Section 3.4</u> for information about monitoring readbacks.

4. Select all four Peak Editor fields, and enter these mass assignment values.

Mass	Span	Gain
175.1	5	10
609.3	5	10
1080.8	5	10
2034.6	5	10

- 5. Start the LC pump, specifying a 1 mL/min flow rate.
- 6. Click **[** (Syringe pump) to infuse the startup solution into the source.
- Click the Analyser tab, and ensure the syringe pump flow rate is 10 μL/min. Turn the probe adjustor (see Figure 2-8) in either direction to optimize peak intensities.

Readbacks report current instrument performance in most of the parameters whose values you specify on the Tune window's pages. They appear as red numerals in read-only fields. These fields are adjacent to those that contain the parameters' set values. Monitor readbacks to determine whether the instrument performs to your parameter settings.

MassLynx provides readbacks on all Tune window pages. You can, however, prevent them from appearing on the Source and Analyser pages or limit their display to those that are out of range.

1. Select Options > Readbacks to open the Readbacks dialog box (Figure 3-8).

Figure 3-8 Readbacks Dialog Box

 Evaluate readbacks, including those on the Diagnostics page (<u>Figure 3-9</u>). Most Source and Analyser page readbacks should match the parameter values you specify.

**Note:** Some readbacks serve a broad diagnostic purpose and therefore do not necessarily mirror their set values. For example, a voltage readback can vary from its set value when the instrument's proper operation does not depend on that voltage. In such a case, whether voltage is at all present is the more critical measure.



ZQ - c:\masslynx\default.pro\acqudb\r File IonMode Calibration Gas Ramps Op	naisci.ipr htions Help				_0×
🗅 🥔 🖬 🎒 🥂 🚍 🚺 丫 🗠	2 🗖 🖆 🗉 🕘 🖓				
ES+Source Analyser Diagnostics Analyser UM Resolution HM Resolution 15.0 HM Resolution 15		Mass ▼ 1 172.9 ▼ 2 772.5 ▼ 3 1521.9 ▼ 4 1971.6	Span         Gain           5         1           5         10           5         40           5         20		
Multiplier		172.9 X 100.0% ×1	772.5 X	1521.9 X 2.2% ×40	1971.6 X 3.8% ×20
Syringe Pump Flow (uL/min) 5.0 Syringe Statue			0.0 772.5 775	520.0 1522.0 1524	1970.0 1972.0 197
Acquire				Press	for Standby
Ready		Vacuum OK		Operate	

Figure 3-9 Diagnostics Page Readbacks

- 3. Let the ion beam stabilize for 3 to 5 minutes.
- 4. Monitor for mass peaks, which should appear at approximately the mass values you specified on the ES+ Source or APCI+ Source page.

#### Setting Up the Calibration File 54

# Chapter 4 Calibrating

Calibrating the mass scale entails setting up a calibration file and specifying calibration parameters in MassLynx.

# 4.1 Setting Up the Calibration File

Before calibrating, you must remove the current calibration file and select a reference file:

 Select Calibration > Calibrate Instrument from the Tune window (Figure 3-3). The Calibration window appears (Figure 4-1).

Reference File List	Calibration: Uncal.cal File Edit Calibrate Process View	Help       Use air refs
	Last Calibrated:	03 Aug 99 09:25
	Data Directory:	C:\MassLynx\Diverse.PR0\data\
	Static:	No calibration
	Scanning:	No calibration
	Scan Speed Compensation:	No calibration
	, Ready	NUM

Figure 4-1 Calibration Window

 Select NaICs2 from the reference file list to choose the reference file, if your ZQ Mass Detector is a 2000 model. Select NaICs4 if it is a 4000 model. 3. Select File > Open. The Open dialog box appears (Figure 4-2).

Open			? ×
Look jn:	🔁 acqudb	- 🗈 🖸	* 🔳
Uncal.cal			
File <u>n</u> ame:			<u>O</u> pen
Files of type:	Calibration File (*.cal)	•	Cancel

Figure 4-2 Open Dialog Box

- 4. Select Uncal.cal, and click Open. The Calibration window reappears.
- 5. Make sure the phrase "No calibration" follows the three calibration types: Static, Scanning, and Scan Speed Compensation.

# 4.2 Setting Calibration Parameters

The rest of this chapter describes how to specify calibration parameters in these windows and dialog boxes:

- Tune window
- Instrument Threshold Settings dialog box
- Automatic Calibration Check dialog box
- Calibration Parameters dialog box
- Mass Measure dialog box
- Automatic Calibration dialog box
- Calibration Acquisition Setup dialog box

#### 4.2.1 Tune Window Settings

- 1. Follow the syringe preparation procedure in <u>Section 2.3.7</u>, this time loading the syringe with sodium cesium iodide solution (from the API Test Kit).
- 2. Click **[1]** (Syringe pump) in the Tune window to infuse the solution into the source.

 Enter these suggested initial reference solution values in Tune window's Peak Editor.

Row	Mass (ZQ 2000)	Mass (ZQ 4000)	Span	Gain
1	172.9	172.9	5	8
2	772.5	1521.9	5	20
3	1521.9	2271.4	5	40
4	1971.6	3470.5	5	39

**Note:** These settings are offered as reference points only and, once adopted, might require adjusting.

- Select rows 1 to 4 in the Peak Editor. This specifies four mass peaks in the Tune window's Peak Display area (see <u>Figure 3-3</u>).
- Click the ES+ Source tab, and enter these suggested parameters in the corresponding fields of the ES+ Source page (Figure 4-3).

Parameter	Suggested Value
Capillary (kV)	3.5
Cone (V)	50
Extractor (V)	3
RF Lens (V)	0.5
Source Temperature (°C)	80
Desolvation Gas Flow Rate (L/hr)	250
Desolvation Temperature (°C)	120
Cone Gas Flow (L/hr)	50



*Caution:* Failure to flow desolvation gas during ESI operation can damage the source.



Figure 4-3 Tune Window Displaying the ES+ Source Page

 Click the Analyser tab to open the Analyser page (Figure 4-4), and enter these suggested parameters in the corresponding fields.

Parameter	Suggested Value
LM resolution	15
HM resolution	15
Ion energy (V)	0.5
Multiplier (V)	600
Cone Gas Flow Rate (L/hr)	50
Syringe Pump Flow Rate (µL/min)	5

ZQ - c:\masslynx\default.pro\acqudb\r Ele Jop Mode Calibration Gas Ramos Or	naisci.ipr				_ <b>_</b> ×
	2 🗖 🖆 🗉 🕘 🛛 😵				
ES+Source Analyser Diagnostics Analyser LM Resolution HM Resolution IS0 Ion Energy 0.5 Multicler 319 590		Mass ✓ 1 1729 ✓ 2 7725 ✓ 3 15219 ✓ 4 1971.6 172.9 \$ 100.0% ×1	Span         Gain           5         1           5         10           5         40           5         20           772.5         8           6.5%         ×10	1521.9 💥 2.2% ×40	1971.6 St 3.8% ×20
Syringe Pump Flow (uL/min) 5.0 Syringe Status					
		172.5 175.	0.0 772.5 77	£ 520.0 1522.0 1524.	1970.0 1972.0 197
Acquire				Press	for Standby
Ready		Vacuum OK		Operate	1.

Figure 4-4 Tune Window Displaying Analyser Page Parameters

- 7. Maximize the signal intensity of the four mass peaks in the Tune window Peak Display:
  - a. Turn the probe adjustor knob (<u>Figure 2-8</u>) to adjust the orientation of the probe relative to the sample cone orifice.
  - b. Adjust the source parameters from the Tune window's ES+ Source page. These include Capillary, Extractor, RF Lens, and Cone voltages, as well as desolvation and cone gas flows.
- Adjust the slide adjustors for LM (low mass) Resolution, HM (high mass) Resolution, and Ion Energy on the Tune window's Analyser page (Figure 3-5) to obtain a full-width-at-half-height measurement of 0.4 to 0.6 da.

Note: Make sure you can see all ions and that none are saturated on a gain of 1X.

### 4.2.2 Instrument Threshold Settings Dialog Box

This dialog box contains parameters that control how the system preprocesses data before sending the data to a host computer.

For most low mass-range calibrations, the instrument acquires calibration data in continuum mode. The continuum-data parameter settings in the Instrument Threshold Settings dialog box ensure appropriate scanning speeds.

- Select Options > Set Instrument Threshold from the Tune window. The Instrument Threshold Settings dialog box appears (<u>Figure 4-5</u>).
- 2. Accept the default settings by clicking OK.

Instrument Threshold Settings		X
Profile Data Baseline Level: 0 Points per Dalton: 8 Centroid Data Minimum centroid height: 1000 Minimum points per peak: 12	Profile Data - Spike Removal         Use Spike Removal         Minimum Spike Intensity:         5         Spike Percentage Ratio:         20         Analog Data         Analog samples/sec:         4	OK Cancel
SIR Data SIR Baseline Level: 0 Ion Counting Threshold: 25		

Figure 4-5 Instrument Threshold Settings Dialog Box

# 4.2.3 Automatic Calibration Check Dialog Box

Select Edit > AutoCal Check Parameters from the Calibration window (Figure 4-1). The Automatic Calibration Check dialog box appears (Figure 4-6).



Figure 4-6 Automatic Calibration Check Dialog Box

#### **Parameters**

**Missed Reference Peaks** – Specifies the maximum number of unmatched consecutive peaks you allow between the reference spectrum and the acquired calibration spectrum.

The calibration fails when the number of unmatched peaks exceeds the maximum you specify. The default value, 2, in most cases suffices.

**Maximum Std Deviation** – For each pair of matched peaks, MassLynx calculates the difference between the measured mass in the acquired calibration file and the true mass in the reference file. The calibration fails when the standard deviation for a set of mass differences exceeds the maximum standard deviation you specify. Decreasing the value imposes a more stringent limit. Increasing it makes the requirement easier to meet, though you should normally avoid setting values greater than 0.20, the default value.

**Apply Span Correction** – Enabling this option ensures correct mass assignment, even when the mass scale differs from the one the instrument was calibrated with.

**Note:** You should not enable this option when the mass range of interest is less than 1000 da and includes the subrange 0 to 150 da.

**Check Acquisition Calibration Ranges** – Waters recommends you enable this option, which displays messages alerting you when the instrument attempts to acquire data outside the calibrated ranges for mass and scan speed.

# 4.2.4 Calibration Parameters Dialog Box

Select Edit > Calibration Parameters from the Calibration window (Figure 4-1) to open the Calibration Parameters dialog box (Figure 4-7).

Calibration Parameters	
Peak Match         ✓       Perform auto peak matching         Peak window (Da)       +/-         Initial error (Da)       2.0         Intensity threshold       0.01	Cancel
Curve Fit Polynomial order 4	
Display Calibrate display	

Figure 4-7 Calibration Parameters Dialog Box

#### **Parameters**

**Perform auto peak matching** – When enabled, matches peaks in the reference file to those in the acquired file.

**Peak window** – Specifies the maximum mass difference between the reference peaks and the *expected* position of corresponding peaks in the acquired spectrum. Normal operating range is 0.3 to 1.5 da.

**Initial error** – Specifies the maximum mass difference you will allow between the first reference peak the software chooses (for its position at or near the center of the calibration range) and the peak it corresponds to in the acquired spectrum.

**Note:** Increasing Peak window and Initial Error values may result in incorrect peak matching.

**Intensity threshold** – Specifies the lower intensity limit of peaks that form the calibration curve. The threshold is expressed as a percentage of the most intense peak of the acquired spectrum. Normal operating range is 0 to 5%.

**Note:** MassLynx does not use any peaks in the acquired spectrum that fall below the Intensity threshold parameter.

**Polynomial order** – Once MassLynx matches each peak in the reference spectrum to one in the acquired spectrum, it calculates the mass difference (the acquired mass less the reference mass) for each peak pair. It then plots these differences as points on a graph and fits a smooth curve through the points. This parameter, set to values 0 to 5, determines the type of curve MassLynx draws:

- Polynomial order = 0 a horizontal baseline
- Polynomial order = 1 a linear curve
- Polynomial order = 2 a quadratic curve
- Polynomial order = 3 a cubic curve
- Polynomial order = 4 a fourth-order curve
- Polynomial order = 5 a fifth-order curve

Waters suggests a polynomial order of 2 for calibrations that use sodium cesium iodide as the reference solution and where the calibrated mass range starts below 100 da and extends through 650 da. Use polynomial order 4 for the wide mass ranges at the high end of the mass scale (600 to 1000+ da) and for calibrating with widely spaced reference peaks.

**Intensity weighting** – Weights the curve toward points that represent the more intense acquired peaks. Thus each point's weight equals the square root of the acquired peak's intensity.

**Calibrate display** – Lets you calibrate the raw data peaks in the upper graph of the Calibration report. As you select each peak, the display recalibrates, bringing the other spectral masses into line.

### 4.2.5 Mass Measure Dialog Box

Mass measure parameters control conversion of raw continuum data to centroid data, which the calibration process requires. You must therefore specify them before calibrating in ESI mode.

**Note:** If you use centroid data for calibration, you need not specify mass measure parameters.

Select Edit > Calibrate Quad Mass Measure Parameters from the Calibration window (Figure 4-1). The Mass Measure dialog box appears (Figure 4-8).

lass Measure		>
Background subtra	đ	OK
<u>Polynomial order</u>	2	Cancel
Below curve (%)	33	
✓ Smooth		]
Peak <u>w</u> idth (Da)	0.5	
<u>N</u> umber of smooths	2	
⊖ Me <u>a</u> n		
Savitzky <u>G</u> olay		
<u>M</u> in peak width at half height (channels)	4	]
• Iop		

Figure 4-8 Mass Measure Dialog Box

# 4.2.6 Automatic Calibration Dialog Box

You should perform all three types of calibration: static, scanning, and scan speed compensation. This lets you subsequently use any data acquisition mode. It also lets you change mass ranges and scan speeds while maintaining correct mass assignment.

- Select Calibration > Calibrate Instrument from the Tune window. The Calibration window appears (Figure 4-1).
- Select Calibrate > Start Acquisition from the Calibration window. The Automatic Calibration dialog box appears (Figure 4-9).

Automatic Calibration
Types
✓ Static Calibration
☑ Scanning Calibration
Scan Speed Compensation
Acquisition Parameters
Process
🗾 🗹 Acquire & Calibrate
Acquire & Verify
Print <u>B</u> eport
OK Cancel

Figure 4-9 Automatic Calibration Dialog Box

3. Specify the type or types of calibration you intend to perform: Static, Scanning, and/or Scan Speed.

**Note:** For a complete calibration, select **Static Calibration**, **Scanning Calibration**, and **Scan Speed Compensation** in the Types area. Then, in the Process area, select **Acquire & Calibrate** and **Print Report**.

#### **Considerations**

Though Waters suggests you perform all three types of calibration, you can nevertheless specify one, or any combination, of calibration types. Beware, however, that doing so invokes the following limitations:

• Specify Static to calibrate only for acquisitions where the quadrupole "parks" at a single mass (for example, SIR acquisitions).

- Specify **Scanning** to calibrate only for scanning acquisitions. This limits acquisitions to the same mass range and scan speed you specified in the calibration.
- Specify Scan Speed Compensation to calibrate only for scanning acquisitions over the same mass range and at the same scan speed you specified in the calibration. Thus to correctly calibrate for scan speed compensation, you must also perform a scanning calibration.
- Specify both Static and Scan Speed Compensation to calibrate for acquisitions
  where the quadrupole "parks" at a single mass. Also, provided you specify the same
  scan speed, this calibrates for scanning acquisitions whose mass ranges lie within
  the scanning calibration's mass ranges. Accordingly, when you specify a 2-second
  scan (400 amu/sec) calibration from 100 to 900 m/z, the instrument can acquire data
  from 100 to 500 amu for a 1-second scan (also 400 amu/sec) and maintain correct
  mass assignment. In this case the static calibration would determine the acquisition's
  start mass and the scanning calibration the mass assignment and scan range.
- Specify both Scanning Calibration and Scan Speed Compensation to calibrate for scanning acquisitions over the mass range you specify for the calibration. You can, however, change the scan speed, provided it remains within the scan speeds you specified in the two calibrations. In this case do not change the mass range, because no static calibration exists to locate the start mass.
- Specify all three types of calibration, Static, Scanning, and Scan Speed Compensation, to allow all types of acquisitions, provided the mass range and scan speed fall between the lower and upper limits for scanning calibration and scan speed compensation.

### 4.2.7 Calibration Acquisition Dialog Box

Specify mass ranges, scan speeds and acquisition mode in this dialog box, which, when you first open it, contains default parameter values for the specified reference file. These values represent the scan range and speed limits of the parameters and instrument.

**Note:** For improved calibration performance, Waters suggests you adopt the parameter values in <u>Table 4-1</u>.

Click Acquisition Parameters in the Automatic Calibration dialog box (Figure 4-9) to open the Calibration Acquisition Setup dialog box (Figure 4-10).

Calibration	Acquisition	n Setup			×
Acquisit	ion Paramete	18		OK	
Scan F	rom	22.98	amu	Cancel	-
Scan T	0	1971.61	amu	Default	-
Run Du	uration	0.5	mins	Derault	-
Data T	ype	Continuur	n 💌		
Scan P	arameters				
Static	Span ±	4	amu		
Static	Dwell	0.1	sec		
Slow S	can Time	6	sec		
Fast S	can Time	1	sec		
Inter S	can Delay	0.1	sec		



Parameter	ZQ 2000	ZQ 4000
Scan From (amu)	50	50
Scan To (amu)	2040	4080
Run Duration (mins)	1.0	2.0
Data Type	Continuum	Continuum
Static Span ± (amu)	4	4
Static Dwell (sec)	0.1	0.1
Slow Scan Time (sec)	10	20
Fast Scan Time (sec)	0.40	0.90
Inter Scan Delay (sec)	0.1	0.1

 Table 4-1
 Recommended Calibration Acquisitions Setup Parameters

4

#### **Parameters**

Scan From, Scan To – Specifies the scan range for each calibration type.

**Run Duration** – Specifies how much time the instrument takes to acquire each calibration data file.

**Data Type** – Specifies data type as centroid, continuum, or MCA. Waters suggests using continuum or MCA acquisitions for electrospray calibrations.

Note: Calibrating in MCA mode limits maximum acquisition speed to 400 da/sec.

**Static Span** – Specifies how much of the mass scale on either side of a reference peak the instrument scans.

Static Dwell – Specifies the time the instrument takes to scan the static span.

**Slow Scan Time** – Specifies how much time it takes the instrument to scan the mass scale over the selected range when acquiring data for *scanning* calibrations (see next section for details).

**Fast Scan Time** – Specifies how much time it takes the instrument to scan the mass scale over the selected range when acquiring data for *scan speed* calibrations (see next section for details).

**Inter Scan Delay** – Specifies how much time elapses between the end of one scan and the beginning of another.

Main - Resets all parameters to their default values.

#### **About Establishing Scan Speeds**

<u>Table 4-1</u> shows Waters' recommended parameter settings for using sodium cesium iodide as the calibrant. However, if you do not calibrate with sodium cesium iodide, you might need to derive your own scan speed parameter values.

To determine Slow Scan Speed and Fast Scan Speed parameter values, first decide what scan speed *range* to acquire your sample data within. Do this by applying the following equation twice, once to establish the range's lower limit and once to establish its upper limit:

Scan speed = Scanning range / Scan time + Interscan delay

Note: These calculations use the ZQ 2000 parameter values in Table 4-1.

Use Slow Scan Time to calculate the slowest scan speed a calibration can accommodate (the range's lower limit). Thus, where Slow Scan Time = 10 seconds:

Scan speed = (2040 - 50)/(10 + 0.1) = 197 amu/sec

Use Fast Scan Time to calculate the fastest scan speed the calibration can accommodate (the range's upper limit). Thus, where Fast Scan Time = 0.4 seconds:

Scan speed = 2040 - 50/0.4+0.1 = 3980 amu/sec

The scan speed range for this calibration is 197 to 3980 amu/sec.

#### **Checking the Calibration**

Select **Process > Verification From File** from the Calibration window (<u>Figure 4-1</u>) to view a successful or failed calibration. The Display Calibration Graphs dialog box appears (<u>Figure 4-11</u>). From it you can select a calibration type for viewing. With the required calibration selected, MassLynx automatically displays the correct calibration file.

Display Calibration Graphs
Select Calibration Type
C Static
© Scanning
<ul> <li>Scan Speed Compensation</li> </ul>
Select Calibration File
Combine scans in data file:
Erom 1 Io 1
Browse Raw Data
OK Cancel

Figure 4-11 Display Calibration Graphs Dialog Box

**Note:** Data for the selected calibration type must appear in the lower frame of the dialog box. If it does not, click **Browse** to find the correct file.

Click OK to repeat the calibration and display a Calibration report.

#### **Calibration Report**

MassLynx momentarily displays a successful calibration and then prints its Calibration report. However, if the standard deviation of a calibration's residuals exceeds the preset maximum, the software displays a set of calibration graphs in the Calibration window (Figure 4-12). These help you identify the problem.

The top calibration graph shows the calibration file. The peak-matching algorithm, which matches calibration peaks to reference peaks, shows the peaks in a contrasting color.

The Data and Reference file graphs show the calibration spectrum with its highlighted peaks matched with reference peaks of a contrasting color. Other graphs show mass difference and residuals.

#### Updating the Calibration Report

- Select Edit > Calibration Parameters from the Calibration window. The Calibration Parameters dialog box appears (Figure 4-7).
- 2. Enter changes, and click OK.

#### **Printing the Calibration Report**

- 1. Click the Calibration window (Figure 4-1) to activate it.
- 2. Click Yes to save changes in the calibration file. The Save As dialog box appears.
- 3. Enter a file name in the File name field.

*Note:* Waters recommends you incorporate the current date in every file you name.

- 4. Click Save or Cancel. The Automatic Calibration dialog box appears (Figure 4-9).
- 5. Select Scan Speed Compensation as the calibration type.
- 6. Select Acquire & Calibrate and Acquire & Verify as the process methods.
- 7. Select **Print Report**, and click **OK**. The Calibrate window appears displaying calibration graphs (Figure 4-12).
- 8. Check the Mean Residual Error parameter, which must be equal to or less than ± 0.2 for a calibration to be successful.



Figure 4-12 Calibrate Window Showing ZQ-4000 Calibration Graphs

#### **Calibration Failure**

Calibration failures result from many causes. If too many mass peaks are missed, check data in the on-screen Calibration report. If the missed masses appear there, one or more of the first three causes listed in <u>Table 4-2</u> likely apply. If the data include these mass peaks, but they nevertheless go unrecognized during calibration, one or more of the subsequently listed causes probably induced the failure.

Symptom	Possible Cause	Corrective Action
No peaks.	No reference sample.	Add reference sample.
	No solvent flow into the source.	Check LC components, syringe pump, and liquid line connections.
	Multiplier set too low.	Adjust multiplier.

Table 4-2	Calibration	Failure	Troubleshooting
-----------	-------------	---------	-----------------

Symptom	Possible Cause	Corrective Action
Too many consecutive peaks missed.	Reference solution in low supply or concentration.	Increase reference solu- tion or its concentration.
	Multiplier set too low to detect the less intense peaks.	Reset the Multiplier parameter.
	Incorrect ionization mode selected.	Make sure the ionization mode matches the calibra- tion file.
	Intensity threshold too high.	Lower threshold, or increase reference concentration.
	Initial Error and/or Peak Window parameter set too low.	Increase parameter limits.
	Maximum Std Deviation exceeded.	Widen (increase) the Maximum Std Deviation parameter.
	The wrong reference file has been selected.	Select the appropriate reference file.

Table 4-2 Calibration Failure Troubleshooting (Continued)

Having taken the necessary action, proceed as follows:

- 1. If the values in the Intensity threshold, Initial error, and Peak windows are adjusted to obtain a successful calibration, check the on-screen Calibration report to ensure the correct masses have been matched.
- 2. Click **OK** in the Calibrate window to accept the new calibration, or click **Cancel** to keep the previous calibration.

#### Incorrect Calibration

The instrument can sometimes meet the calibration criteria, despite failing to match the correct peaks. Such error can arise when you specify these settings:

- Intensity threshold set to 0
- Initial error greater than 2.0
- Peak window greater than 1.5
- Maximum Std Deviation greater than 0.2

Always examine the on-screen Calibration report (Figure 4-12) for correct peak matches. The calibration is correct when the acquired spectrum looks like the reference spectrum, and it highlights all the expected peaks.

Contamination or background peaks can also invalidate a calibration. For example, a contamination or background peak that lies within a peak-matching window might be more intense than its neighboring reference peak. Thus the software can choose the contamination or background peak instead of the reference peak. In such a case, if the reference peak is closer to the peak window's center, narrow the peak window to exclude the contamination or background peak. If, on the other hand, the reference peak is not closer to the window's center, and you cannot narrow the window without affecting it, edit the calibration manually.

#### **Manually Editing Matched Peaks**

You can manually exclude incorrectly matched peaks from the on-screen Calibration report. Manually editing a single peak will not affect other matched peaks in the calibration.

To exclude an incorrectly matched peak in an acquired spectrum, place the cursor over it and right-click. This removes the peak's highlighting and excludes it from reported data.

To include a peak as a reference, place the cursor over it and right-click. This matches the selected peak to the closest peak in the reference spectrum.

#### **Saving the Calibration**

You can save and reuse calibrations.

Mass range and scan speed limits remain unchanged for retained calibrations. So do ion energy and resolution settings for calibration acquisition, which can affect mass assignment.

#### **Verifying the Calibration**

Once you perform and save a full instrument calibration, repeating it is not always necessary. Instead, you can make a scanning acquisition and verify a saved calibration, following this procedure:

- Open the Calibration Parameters dialog box by selecting Edit > Calibration Parameters from the Calibration window (Figure 4-1).
- 2. Set all peak matching parameters to those used in the calibration.
- Select Calibrate > Start Acquisition from the Calibration dialog box (Figure 4-1) to open the Automatic Calibration dialog box (Figure 4-9).
- 4. Enable these parameter fields in the Automatic Calibration dialog box:
  - Scanning Calibration
  - Acquire & Verify
  - Print Report
- 5. Disable these parameter fields:
  - Static Calibration

- Scan Speed Compensation
- Acquire & Calibrate
- Click the Acquisition Parameters button to open the Calibration Acquisition Setup dialog box (Figure 4-10).
- Set Scan From, Scan To, Run Duration, Data Type, Slow Scan Time, Fast Scan Time, and Inter Scan Delay to agree with the parameters you will use for data acquisition.

*Note:* No other options apply when you select Scanning Calibration.

- 8. Click **OK**. The Automatic Calibration dialog box reappears.
- 9. Click **OK** to start the verification procedure.

The instrument performs a scanning acquisition. Then MassLynx combines the data acquired into a single spectrum, which it compares with the reference file. MassLynx then creates and prints a calibration curve. Unlike the original calibration procedure, the instrument's calibration remains unchanged, the printed report serving as verification.

**Note:** Do not adjust analyser settings (LM, HM resolution, and ion energy) after calibrating. Use only source parameters to optimize signal intensity.
# Chapter 5 Maintaining

This chapter includes routine maintenance procedures.

# 5.1 Considerations

#### **Safety and Handling**

**Caution:** Observe good laboratory practice when you handle solvents, change tubing, or operate the instrument. Refer to Material Safety Data Sheets for the solvents you use, and know their chemical properties.



Attention: Do not touch integrated circuit chips or other circuit board components that do not specifically require manual adjustment. Static electric charge can damage electronic components.

Attention: Never open the instrument's top or side panels to access power supplies or other components. The supplies do not contain user-serviceable parts.



Attention: Never disconnect an electrical assembly while the system is plugged in. This could damage electrical parts.

Attention: Also, turn off the power and wait 10 seconds before disconnecting an assembly.

#### **Maintenance Equipment**

Routine parts cleaning requires the following equipment:

- An ultrasonic bath with a minimum chamber size of 12 in. x 6 in. x 4 in. (300 mm x 150 mm x 100 mm)
- Glass vessels, approximately 4 in. (100 mm) in diameter and 4 3/4 in. (120 mm) high
- A 500-mL graduated cylinder (to use when cleaning the hexapole assembly)

#### **Contacting Waters Technical Service**

North American customers who experience maintenance problems they cannot resolve should contact Waters Technical Service at 800 252-4752. Others should phone their local Waters subsidiary or Waters corporate headquarters in Milford, Massachusetts (U.S.A.).

<u>Table 5-1</u> lists periodic maintenance schedules you should observe to ensure the instrument's optimal performance.

**Note:** The maintenance frequencies shown apply to instruments that normally receive moderate use.

Maintenance Procedure	Frequency
Gas-ballast the rotary pump.	Daily (APCI)
	Weekly (ESI)
Check the rotary pump oil, and top-off when necessary.	Weekly
Replace the rotary pump oil.	Every 3 months of continuous
	appears markedly discolored
Clean the cone gas nozzle, sample cone, and	When sensitivity decreases to
Clean the ESI APCI probe tip, or replace the ESI	When sensitivity decreases to
Replace the APCI fused silica.	
Clean the corona discharge needle (APCI mode).	When sensitivity decreases to
	unacceptable levels
Clean the ion block assembly.	When it is visibly fouled
	When background or peak
	contaminants are unaccept- ably high
Clean all source components.	When sensitivity decreases to
	When cleaning the cone gas
	nozzle, sample cone, and baffle
	plate fails to improve results

Table 5-1 Maintenance Schedule

# 5.2 Routine Maintenance

Routine maintenance comprises these tasks:

- Checking the rotary pump oil (<u>Section 5.2.1</u>)
- Changing the pump oil (<u>Section 5.2.2</u>)
- Gas-ballasting the rotary pump (<u>Section 5.2.3</u>)
- Replacing the oil mist filter (<u>Section 5.2.4</u>)
- Cleaning the source assembly (<u>Section 5.2.5</u>)
- Cleaning the APCI probe tip (<u>Section 5.2.6</u>)
- Cleaning and replacing the corona discharge needle (<u>Section 5.2.7</u>)

Perform routine maintenance at scheduled intervals (<u>Table 5-1</u>), or when troubleshooting indicates it is necessary.

#### 5.2.1 Checking the Rotary Pump Oil

*Note:* You can check the oil level while the pump is operating.

The rotary pump oil level appears in the rotary pump's oil level sight glass. Check the oil level at weekly intervals. Maintain it at or near the MAX level. If you must add oil, vent and shut down the detector before removing the oil filler plug.

Examine the oil each time you check its level. It should be colorless, free of visible contaminants. If it is discolored, change it, following the procedure in <u>Section 5.2.2</u>.

#### 5.2.2 Replacing the Pump Oil

Note: You must vent the instrument and shut it down before adding oil.

Change the rotary pump oil after every 3 months of continuous operation, or whenever it becomes markedly discolored. Gas-ballast following every oil change.

#### **Required Materials**

- Latex or vinyl gloves
- Flat-head screwdriver
- · Container to catch used oil
- Funnel
- Vacuum oil (use only Ultragrade 19 or Inland Q45 (Edwards 45) vacuum pump oil)
- 4-mm hex wrench

5

#### Procedure

Refer to Figure 5-1 and Figure 5-2 when performing the following procedure.

Note: Operate the pump to warm the oil before you drain it.

Caution: Wear latex or vinyl gloves when changing the pump oil.



Figure 5-1 Rotary Pump Assembly Fitted with Oil Mist Filter

*Note:* Vent the instrument according to the procedure in <u>Section 5.2.5</u>, and proceed with these steps.

- 1. Turn the instrument's power switch to Off.
- 2. Place something under the pump motor, tilting the unit so that the oil drain plug is at the lower end.



Figure 5-2 Rotary Pump Oil Filler Plug, Drain Plug, and Sight Glass

- 3. Remove the drain plug with the slotted screwdriver.
- 4. Remove the oil filler plug to facilitate drainage.
- 5. Drain the oil completely.
- 6. Flush residual oil from the pump as follows:
  - a. Refit the oil drain plug.
  - b. Add about 300 mL of oil to fill the pump to the MIN mark on the sight glass.
  - c. Refit the oil filler plug.
  - d. Select Options > Pump from the Tune window (Figure 3-3) to start the pump operating. Continue to run the pump for 10 to 15 minutes.
  - e. Select Options > Vent and click Yes at the prompt to stop the pump.
  - f. Let the oil drain completely.
- 7. Use a funnel to add about 1 L of oil to the pump, until the level in the sight glass reaches the MAX mark.
- 8. Allow a few minutes for the oil to fully drain to the bottom of the pump, then recheck the level. Add more, if necessary, but do not overfill.

*Note:* The oil level normally drops about 10% after the pump starts.

- 9. Refit the oil filler plug.
- 10. Select Options > Pump from the Tune window (Figure 3-3) to start the pump.
- 11. Gas-ballast (Section 5.2.3).

## 5.2.3 Gas-Ballasting the Rotary Pump

The rotary pump draws large quantities of solvent vapors. The vapors tend to condense in the pump oil, diminishing pumping efficiency. Gas-ballasting purges condensed contaminants from the oil.

Gas-ballast the rotary pump when these conditions apply:

- With ESI operation, once a week
- With frequent APCI operation, once a day
- · When the pump oil appears cloudy
- · When the vacuum pressure is higher than normal
- · When condensate forms in the rotary pump exhaust line
- When you change the rotary pump oil



Attention: Failure to routinely gas-ballast the rotary pump shortens oil life and, consequently, pump life.





Attention: Do not gas-ballast the rotary pump while the detector is in operational mode.

 Shut the vacuum system isolation valve, moving its handle fully to the right (Figure 5-1).



Attention: Never gas-ballast the rotary pump for more than 2 hours.

- 2. When the oil is clear, return the gas-ballast control to its normal position.
- 3. Open the vacuum system isolation valve (Figure 5-3).



Figure 5-3 Isolation Valve

4. Open the gas ballast knob on the top of the pump. Run the pump for 30 to 60 minutes, then close the gas ballast knob.

## 5.2.4 Replacing the Oil Mist Filter

*Caution:* Wear latex or vinyl gloves when replacing the oil mist filter.

**Note:** Vent the instrument according to the procedure in <u>Section 5.2.5</u>, and proceed with these steps.

- 1. Turn the instrument's power switch to Off.
- 2. Unscrew the NW25 clamp, and remove the exhaust line from the top of the oil mist filter housing (Figure 5-4).
- 3. Loosen the four bolts that secure the top and bottom covers of the oil mist housing with a 4-mm hex wrench.



Figure 5-4 Oil Mist Filter Assembly

4. Remove the top cover, and discard the odor filter.

Note: The odor filter will likely stick to the cover.

- 5. Pull upward on the oil mist filter, removing and discarding it along with its O-ring.
- 6. Slip the new O-ring over the support post in the bottom of the filter housing, then press the new oil mist filter on top.
- 7. Refit the top cover, securing it with the four screws.
- 8. Reattach the exhaust hose to the NW25 clamp.
- 9. Evacuate the system by selecting **Options > Pump** from the Tune window.

### 5.2.5 Cleaning the Source Assembly

Clean the sample cone and cone gas nozzle when they are visibly fouled, or when you eliminate LC and sample-related causes for decreased signal intensity. When cleaning these parts fails to increase signal sensitivity, also clean the extraction lens, hexapole, and ion block.

#### **Required Materials**

- Lint-free cotton or powder-free nitrile gloves
- 6-in. forceps or needle-nose pliers
- 2.5-mm hex wrench
- 3-mm hex wrench
- 6 mm-hex wrench

- Small, flat-head screwdriver
- Small, Phillips-head screwdriver
- Clean, 1000-mL beaker
- Clean, 500-mL graduated cylinder

Note: Use only glassware you have not cleaned with surfactants.

- HPLC-grade methanol
- HPLC-grade water
- Formic acid
- Ultrasonic bath
- Source of oil-free, inert gas (nitrogen or helium) for drying (air-drying optional)
- Lint-free paper towels

#### **Spare Parts**

You might need to replace these spare parts when cleaning source components.

Item	Waters Part Number	Reference Number
Ion block D-ring (AS035)	700001192	5711312
Viton O-ring (AS214)	700000902	5711294
Extraction cone O-ring	700001897	5711967
Sample cone O-ring	700001194	5711321

#### **Source Cleaning Procedure**

- 1. Disassemble the source components (see <u>"Disassembling Source Components" on page 82</u>).
- 2. Clean the source components (see <u>"Cleaning the Source Components" on page</u><u>88</u>).
- 3. Remove and clean the hexapole assembly (see <u>"Removing and Cleaning the</u> <u>Hexapole Assembly" on page 89</u>).
- Reassemble the source components (see <u>"Reassembling the Source Components"</u> on page 90).

#### **Disassembling Source Components**

The source components comprise the pumping block and ion block assemblies, which you must disassemble before cleaning. Consult <u>Figure 5-5</u> and <u>Figure 5-6</u> when disassembling and assembling these assemblies.

1. Stop the liquid flow, then disconnect the LC line from the probe.



Attention: Never remove either probe before the source heater cools to below 100 °C and the source heaters cool to below 50 °C. Doing so will shorten the probe heater life. To reduce cooling time, continue flowing API gas.



TP02001

- 1 Ion Block Assembly 2 – O-Ring, Viton (AS016) 3 – O-Ring, Viton (2-030) 4 – Ion Block Support 5 – O-Ring, Viton (2-039) 6 – O-Ring, Viton (AS016) 7 – O-Ring, Viton (AS017)
- 7 O-Ring, Viton 42.0 × 1.5 mm

Figure 5-5 Pumping Block Assembly

- 8 PTFE Encapsulated O-Ring
- 9 Source Pogo Pins
- 10 PEEK Connector Body
- 11 O-Ring, Viton 2-103
- 12 Threaded Plug
- 13 Pumping Block
- 14 Hexapole



- 1 Ion Block Fastener
- 2 BS 10 Black Viton O-Ring
- 3 Screws, M3 imes 20 Cross
- $4 M3 \times 6 PH$
- 5 Washer, M3 SS
- 6 Cover Plate
- 7 Heater Assy
- 8 O-Ring, Viton, 2-035
- 9 Cone Gas Spring Clip
- 10 Terminal Block
- 11 Ion Block Plug
- 12 Ion Block Plug Seal
- 13 Ion Block
- 14 Valve Stem

- 15 Large Valve Stem Washer
- 16 O-Ring, Kalrez, 0.240  $\times$  0.063
- 17 Seal Ring
- 18 Black Viton O-ring PTFE Encapsulated
- 19 Extraction Retainer
- 20 Extraction Cone
- 21 Sample Cone O-Ring
- 22 Sample Cone
- 23 Cone Gas Nozzle
- 24 O-Ring, Kalrez, AS007
- 25 Small Valve Stem Washer
- 26 Flat Washer, M4
- 27 Metric Hex Nut, M4

Figure 5-6 Ion Block Assembly

- Click Press For Standby on the Tune window. The icon changes from green to red. This means all high voltages are turned off, as well as the ESI desolvation /APCI probe heater.
- 3. Switch off the source heater by setting a temperature of 20 °C and pressing Enter.
- 4. Before you remove the ESI or APCI probe, stop the nitrogen flow by toggling on the toolbar to Off.
- Select Options > Vent and Yes, at the prompt. Click OK when the message box appears.



Attention: If you are cleaning only the sample and cone gas nozzles, you need not vent the system.

**Note:** Venting interrupts electrical power to the turbomolecular pump. Nevertheless, the pump continues to operate briefly, until rotational speed decreases to less than 50%, at which point its vent valve opens.

- 6. Disconnect the appropriate front panel gas and electrical connections.
- Unscrew the probe's two knurled thumbscrews, and retract it from the source (Figure 5-7)



Caution: The probe might be too hot to handle. Allow time for it to cool.



**Attention:** Never remove a probe before it cools to below 100 °C and the source heater cools to below 50 °C. Doing so shortens the life of the probe heater. To reduce cooling time, continue flowing API gas.



Figure 5-7 Probe Assembly in Position on the Source

8. Remove the center panel (Figure 5-8) from the front of the instrument by grasping its sides and pulling it toward you.



Figure 5-8 ZQ Detector Front View

9. Unfasten the two clip fasteners (Figure 5-7) so that the source enclosure opens downward on its hinge, allowing access to the source contents.



**Attention:** Wear lint-free cotton or powder-free nitrile gloves for the rest of the cleaning procedure.

 If you are using an APCI probe, carefully remove the corona discharge needle (<u>Figure 5-9</u>).



Figure 5-9 Source Showing the Corona Discharge Needle

11. Remove the PTFE tubing attached to the cone gas nozzle (Figure 5-10).



Figure 5-10 Ion Block

- Remove the two 2.5-mm hex screws securing the cone gas spring clip (Figure 5-10).
- 13. Remove the cone gas nozzle, O-ring, and sample cone from the ion block.



- 14. Carefully separate the sample cone from the cone gas nozzle (<u>Figure 5-6</u>). Remove the sample cone O-ring (<u>Figure 5-11</u>).
- 15. Remove the baffle plate, and set all pieces aside.



Figure 5-11 Sample Cone and Cone Gas Nozzle

- 16. Remove the two 6-mm hex screws that secure the ion block.
- 17. Remove the ion block from the ion block support (<u>Figure 5-5</u>). The ion block fits snugly on the support, so separating them can require considerable effort.
- 18. Place the ion block on a flat surface, and remove any remaining O-rings that remain on it.
- 19. Use the Phillips-head screwdriver to remove the hold-down screw from the PEEK extraction cone retainer (Figure 5-12).



Figure 5-12 Ion Block Rear View

Note: Do not damage the ion block surface and insulator O-ring.

- 20. Grasp the extraction cone pin with the needle-nose pliers. Lift the extraction cone from the ion block.
- 21. Insert a suitable nonmetallic implement under the inner edge of the polymeric seal ring to pry the seal ring and O-ring out of the ion block.

*Note:* Avoid damaging the seal and O-ring on the ion block.

22. Remove the D-shaped O-ring from the front of the ion block.

#### **Cleaning the Source Components**

- 1. Place the ion block in a beaker with methanol : water (1:1).
- 2. Place the beaker containing the ion block and methanol/water mixture in an ultrasonic bath for 20 minutes.
- 3. Remove the ion block from the methanol/water mixture, and place it in a beaker containing 100% methanol.
- 4. Place the sample cone, cone gas nozzle, and extraction cone in a beaker containing methanol : water (1:1). If debris have accumulated at the sample cone orifice, place a drop of formic acid on the orifice before placing it in the beaker.

**Note:** If the parts are obviously fouled, use a mixture of 45% methanol, 45% water, and 10% formic acid.



*Caution:* Be extremely careful when working with formic acid. Use a fume hood and appropriate protective equipment.

- 5. Expose all parts to ultrasound for about 30 minutes. If you used formic acid in the cleaning solution, rinse the parts well, immersing them in a beaker of water and setting the beaker in an ultrasonic bath for about 20 minutes to remove all traces of the acid. Then displace the water by immersing the parts in a beaker of methanol and setting the beaker in the ultrasonic bath for 10 minutes.
- 6. Carefully remove the parts from the beaker. Blow-dry them using inert, oil-free gas. Alternatively, arrange the parts on lint-free towels and let them air dry. Wipe off water spots with a lint-free cloth.

#### **Removing and Cleaning the Hexapole Assembly**

- Remove the 3-mm screws securing the ion block support, and remove the ion block support from the pumping block. Remove any O-rings that remain stuck to the pumping block surface (Figure 5-5).
- 2. Use your hand to gently grasp the hexapole (Figure 5-13), and retract it carefully from the analyser assembly.



Figure 5-13 Hexapole Assembly



Attention: Never squeeze the hexapole rods together when you remove the hexapole. Their orientation relative to one another is critical to the instrument's performance. Also, avoid scratching the bored surfaces of the pumping block as you withdraw the hexapole.

- 3. Bend a length of stainless steel tubing into a hook shape. Insert the hook into one of the holes in the assembly's rear support ring.
- 4. Carefully suspend the assembly in a graduated cylinder. Add methanol to the vessel until it covers the assembly.
- 5. Place the graduated cylinder in an ultrasonic bath for 30 minutes.

- 6. Remove the hexapole assembly from the graduated cylinder, and place it on a lint-free cloth. Allow it to air-dry, or use a nitrogen flow to dry it.
- Insert the assembly, aligning the notches in the differential aperture at its rear with the two bottom support rails on the analyser assembly. Carefully slide the assembly into place (Figure 5-13). Be sure to insert the assembly fully.
- 8. Check the condition of the three, rear, ion block support O-rings. Replace them with new ones, if necessary. Ensure the O-rings are properly installed before reattaching the ion block support.

**Note:** The pilot pin in the ion block support must align with the notch on the ion block.

9. Secure the ion block by alternately tightening its retaining screws.

#### **Reassembling the Source Components**

- Visually examine the condition of the three, front, ion block support O-rings (Figure 5-5). Replace them, if necessary. Be sure they are properly installed before you proceed.
- 2. Refit the PEEK seal ring and O-ring on the ion block.
- 3. Press the extraction cone into the ion block support. Secure it with the PEEK retainer and screw.
- 4. Refit the sample cone and its O-ring into the cone gas nozzle. Secure all parts with the cone gas spring clip and two 2.5-mm hex screws.
- 5. Refit the ion block assembly to the PEEK support block. Secure it with two 6-mm hex screws. Avoid overtightening.
- If you have cleaned only the sample and cone gas nozzles, turn the isolation valve (<u>Figure 5-10</u>) back to <u>Open</u>.
- If you are using the APCI probe, replace the corona discharge needle (<u>Section 5.2.7</u>).
- 8. Refit the PTFE tube to the cone gas nozzle.
- 9. Close the source enclosure, reattaching its clip fasteners.
- 10. Refit the probe assembly, and reconnect the LC line.
- 11. Refit the center panel (Figure 5-8) on the front of the instrument.
- 12. Reconnect the front panel gas and electrical lines.
- 13. Evacuate the instrument, turn on the API gas, and check for nitrogen leaks around the source enclosure.

## 5.2.6 Cleaning the APCI Probe Tip

Clean the APCI probe tip when buffer residue accumulates on the probe tip or when signal intensity weakens.

- 1. Stop the liquid flow.
- 2. Open the source enclosure cover, and close the isolation valve (Figure 5-10). Close the source enclosure cover, reattaching its clip fasteners.
- 3. Click **(API Gas)** in the Tune window to start nitrogen flowing.
- 4. Adjust the nitrogen flow to approximately 650 liters per hour, as indicated by the Tune window (Figure 3-3) desolvation gas meter. Set the APCI probe heater temperature to 650 °C, and press Enter.
- 5. Click the **Operate** icon, and wait 10 minutes with the APCI probe heater at 650 °C. This will remove any chemical contamination from the probe tip.

## 5.2.7 Cleaning and Replacing the Corona Discharge Needle

Clean or replace the needle if it looks corroded or black, and you have noticed decreased signal intensity.

#### **Required Materials**

- Lint-free cotton or powder-free nitrile gloves
- Lapping film
- HPLC-grade methanol
- Lint-free tissue

#### **Procedure**

To clean and replace the corona discharge needle (Figure 5-9):

- 1. Remove the center panel from the front of the detector (Figure 5-8).
- 2. Remove the two knurled thumbscrews from the top of the probe.
- 3. Disconnect the front panel gas and electrical connections (Figure 5-8).
- 4. Open the source enclosure cover.



Attention: Wear lint-free cotton or powder-free nitrile gloves for the rest of the cleaning procedure.

5. Remove the corona discharge needle from the source, pulling it straight out.



**Caution:** The inner surfaces of the source enclosure and its constituent components are hot.

- 6. Clean and sharpen the tip of the needle with the lapping film, then wipe the needle clean with a methanol-saturated tissue. Replace the needle if it is deformed or otherwise damaged.
- 7. Reinstall the needle. Point the tip toward the sample cone.
- 8. Close the source enclosure cover, reattaching its clip fasteners.
- 9. Reinstall the probe assembly.

# 5.3 Replacing Parts

## 5.3.1 Replacing the Ion Block Cartridge Heater

Replace the cartridge heater when it fails to heat.

#### **Required Materials and Spare Part**

- 3-mm hex wrench
- 1.5-mm hex wrench
- Slotted screwdriver
- Needle-nose pliers
- Ion block cartridge heater (Figure 5-11), spare part (part number 700001201, reference number M955504AD1)



Figure 5-14 Ion Block Cartridge Heater

#### Procedure

**Note:** Vent the instrument according to the procedure in <u>Section 5.2.5</u>, and proceed with these steps.

- 1. Remove the cover plate (shown as a cutaway in Figure 5-15) from the ion block.
- 2. Remove the two screws securing the heater cartridge ring tags from the PEEK terminal block (Figure 5-15).
- 3. Carefully bend the ring tags out of the terminal block.
- 4. Use the needle-nose pliers to gently retract the heater cartridges from the ion block.



Figure 5-15 Replacing the Heater Cartridge

- 5. Slide the new heater cartridge into the ion block with the needle-nose pliers.
- 6. Place the two heater cartridge ring tags onto the PEEK block terminals.
- 7. Tighten the two terminal block screws with a 20-mm hex wrench.
- 8. Reinstall the ion block cover plate. Secure it with the four hex screws.
- 9. Reinstall the source enclosure, probe adjustor assembly, and probe.

### 5.3.2 Replacing the Stainless Steel Capillary

Replace the stainless steel sample capillary on the ESI probe when it permanently clogs, becomes contaminated, or sustains damage.

#### **Required Materials**

- Flat-head screwdriver
- Needle-nose pliers

- 1.5-mm hex wrench
- 6-mm hex wrench
- 5/16-in. wrench
- 7/16-in. wrench
- Capillary tube (part number 700000341, reference number M955088AD)
- Loupe

#### Procedure

- 1. Remove the two end-cover retaining screws on the ESI probe with the slotted screwdriver.
- 2. Loosen the set screw on the LC PEEK union with the 1.5-mm hex wrench, and remove the probe's end-cover.
- 3. Unscrew the probe tip with the 6-mm wrench, and remove it.
- 4. Remove the LC union with the 5/16-inch and 7/16-inch wrenches.
- 5. Unscrew the coupling, and withdraw the capillary from the probe.
- 6. Remove the capillary and O-ring from the coupling. Discard the capillary, PTFE liner, and ferrule assembly.
- 7. Use the needle-nose pliers to remove the conductive sleeve from the inner bore of the probe assembly fitting.
- 8. Slide a GVF16 ferrule onto the liner tube.
- 9. Press the O-ring into the groove facing the short end of the coupling.
- 10. Slide the coupling, short end first, onto the capillary, followed by the new PTFE liner tube and ferrule.
- 11. Slide a compression screw and ferrule onto a piece of 0.007-inch PEEK tubing. Connect it to the opposite side of the LC union.
- 12. Press the capillary into the union until it seats.
- 13. Tighten the adaptor nut on the LC union so that it is snug but not tight.
- 14. Gently tug the capillary to make sure it remains secure.
- 15. Remove the PEEK tubing from the union.
- 16. Slide the conductive sleeve onto the capillary, then feed the capillary through the probe.
- 17. Attach the coupling nut to the probe, and gently tighten it with the 7/16-inch wrench.
- Replace the probe tip, then screw down until a 0.5-mm section of the capillary protrudes from its end (<u>Figure 5-16</u>). Use the loupe, provided in the Startup Kit, to ascertain its length.



Figure 5-16 ESI Probe Tip with Capillary Protruding 0.5 mm

- 19. Replace the probe end cover and secure it with the two slotted screws. Tighten the set screw to clamp the LC union in place.
- 20. Before reinstalling the probe, attach it to the nebulizer gas connection and turn on the nitrogen by selecting the API gas icon on the Tune page.
- 21. Make sure no nitrogen escapes from the probe tip. If you find a leak, replace the probe tip assembly and O-ring.



Attention: Check carefully for leaks! Leakage can destroy a probe.

## 5.3.3 Replacing the ESI Probe Tip

Replace the probe tip if you encounter any of these problems:

- Gas leaks from the O-ring.
- A blockage in the internal metal sheathing through which the stainless steel capillary passes.
- The threads are damaged.

#### **Required Materials and Spare Part**

- 6-mm hex wrench
- Loupe
- ESI probe tip, spare part (part number 750000337, reference number M955016BC1)

#### Procedure

- 1. Remove the ESI probe from the source.
- 2. Unscrew and remove the probe tip with the 6-mm hex wrench.
- 3. Install the new probe tip, and screw down until 0.5 mm of the capillary protrudes from the end. Use the Startup Kit loupe to ascertain the capillary position.

## 5.3.4 Replacing the APCI Fused Silica Capillary and Filter Pad

Replace the fused silica capillary, and/or filter pad, when signal intensity decreases and backpressure increases.

#### **Required Materials**

- 1.5-mm hex wrench
- 5/16-in. open-end wrench
- 7/16-in. open-end wrenches (2)
- Ceramic capillary cutter (part number WAT250324 in the Tools Kit)
- Butane lighter or match
- HPLC-grade methanol
- Slotted screwdriver
- Lint-free paper towels
- Loupe

#### **Spare Parts**

- Two GVF004 ferrules (part number 700000344, reference number 6070737)
- Fused silica (part number 430000109)
- Filter pad (part number 600000103)

#### **Procedure**

- 1. Slide the probe tip and heater assembly off the probe.
- 2. Remove the two screws from the probe's end-cover with the slotted screwdriver.
- 3. Loosen the two set screws that hold the LC filter, and remove the probe end with the hex wrench.
- 4. Use the two 7/16-inch wrenches to unscrew the filter from the coupling. Then remove the ferrule from inside the filter.
- 5. Use the two 7/16-inch wrenches, to unscrew the two halves of the filter cartridge and replace the filter pad. Reconnect the two filter cartridge halves.
- 6. Remove the old filter pad and replace it with a new one.

- 7. Use a 7/16-inch wrench to unscrew the coupling from the probe. Unscrew the adaptor nut from the probe with a 5/16-inch wrench.
- 8. Remove and discard the fused silica capillary, ferrule, and O-ring.
- 9. Examine the end of a length of new  $300 \times 100 \ \mu m$  fused silica capillary. Use the loupe to ascertain that you cut the end squarely.

**Note:** Always make perpendicular cuts in the capillary, afterward examining them for squareness with the loupe.

- 10. Feed the fused silica capillary through the probe.
- 11. Press the new O-ring into the groove facing the short end of the coupling.
- 12. Slide the coupling (short end first) onto the capillary and finger tighten.
- 13. Slide a GVF004 ferrule onto the capillary.
- 14. Position the filter with the flow direction arrow pointing towards the coupling. Connect the coupling to the filter, making sure the capillary is fully seated inside the filter.
- 15. Tighten only until the capillary is snug in its fitting, then gently tug it to be sure it remains in place.



Attention: Overtightening the fitting can damage the capillary.

- 16. Tighten the coupling to the probe with a 7/16-inch wrench.
- 17. Run your fingers across the edges of the ceramic capillary cutter to determine which edge is the smooth one. Use the smooth edge to cut the capillary so that 0.5 to 1 mm extends beyond the nebulizer tip.

**Note:** The capillary should extend 0.5 to 1.0 mm from the end of the nebulizer. If it does not, reinstall the capillary.

- 18. Unscrew the coupling from the probe and withdraw the capillary from it.
- 19. Slide a GVF004 ferrule, followed by the adaptor nut and then another GVF004 ferrule, onto the capillary.
- 20. Use a flame to remove about 20 mm of polyamide coating from the end of the capillary. Clean with a methanol-saturated tissue.
- 21. Carefully reinsert the capillary into the probe. Tighten the coupling with a 7/16-inch wrench.
- 22. Replace the probe end-cover and retaining screws.
- 23. Use the 1.5-mm hex wrench to tighten the set screws in the probe end-cover, clamping the filter in place.
- 24. Replace the probe heater assembly.

5

## 5.3.5 Replacing the APCI Probe Heater

Replace the APCI probe heater if it fails to heat.

#### **Required Material and Spare Part**

- 0.89-mm hex wrench or slotted screwdriver, depending on the type of screw that secures the probe tip
- APCI probe heater, spare part (part number 700000337)

#### **Procedure**

- 1. Loosen the two set screws at the base of the probe tip assembly, and slide the probe jacket off the assembly.
- 2. Separate the heater from the probe body, pulling it parallel to the axis of the probe.
- 3. Install the new heater onto the probe end-cover.
- 4. Replace the probe tip assembly, securing it with the two set screws.

# Chapter 6 Troubleshooting

This chapter describes how to troubleshoot the instrument using recommended troubleshooting procedures. This chapter addresses the following topics:

- Component hardware troubleshooting
- Checking the APCI probe

# 6.1 Safety and Handling

*Caution:* Observe good laboratory practice when you handle solvents, change tubing, or operate the instrument. Refer to Material Safety Data Sheets for the solvents you use, and know their chemical properties.

**Caution:** To avoid possible electric shock, never disconnect an electrical assembly while power is applied to the instrument. Once power is turned off, wait about 10 seconds before you disconnect an assembly.



**Caution:** To avoid possible electric shock, do not open the power supply cover. The power supply does not contain user-serviceable parts.

**Attention:** To prevent circuit damage from static charges, do not touch integrated circuit chips or other components that do not require manual adjustment.

#### **Proper Operating Procedures**

To maintain optimal performance, follow the operating procedures and guidelines in this guide.

#### **Spare Parts**

See <u>Appendix C, Accessories and Spare Parts</u>, for spare parts information. Parts not included in <u>Appendix C</u> are not recommended for replacement by the customer.

#### **Contacting Waters Technical Service**

You can easily correct many problems. Nevertheless, when North American customers cannot correct a condition, they should contact Waters Technical Service at 800 252-4752. All other customers should call their local Waters subsidiary or Waters corporate headquarters in Milford, Massachusetts U.S.A.

When you contact Waters, be ready with this information:

- · Completed normal operation checklist for the method you are using
- Nature of the symptom
- Waters Micromass ZQ Detector model number and serial number
- Flow rate
- Operating pressure
- Mobile phase(s)
- Detector settings
- Type and serial number of column (if using an HPLC system)
- Sample description
- Control mode (MassLynx, No interaction, other)
- Software version and serial number

# 6.2 Component Hardware Troubleshooting

<u>Table 6-1</u> contains suggestions for resolving hardware problems.

Symptom	Possible Cause	Corrective Action
No ion peaks on the Tune window (No Ion Beam)	Operating parameters (Capillary/Corona, Cone, Extractor, RF Lens, Ion Energy, Gas Nitrogen, and Heaters) on the Tune window are improperly set.	Optimize parameters. See <u>Section 3.2.1</u> , <u>Section 3.3.2</u> , and <u>Section 3.3.3</u> .
	Cables are not properly connected.	Ensure all cables are correctly attached to the source and probe.
	Instrument is not in the operational mode.	Click Press for Operate (Tune window) to put the instrument in operational mode. The button's associ- ated icon turns from red to green, and the Operate LED on the instrument's front panel reads "On."
	Communication failure.	Reinitialize. (Select Options > Reinitialize from the Tune window.)
	No sample is present.	Make sure sample is loaded in the autosampler or syringe.
	Isolation valve is closed.	Open the isolation valve.
	The source components are dirty	Clean the source compo-

Table 6-1	Hardware	Troubleshooting
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Symptom	Possible Cause	Corrective Action
No ion peaks on the Tune window (No Ion Beam) <i>Continued</i>	Insufficient nitrogen flow.	Ascertain nitrogen pressure is 90 to 100 psi and the gas flow rate on the Tune window is >100 L/hr. The desolvation and probe heaters shut off when the nitrogen flow rate falls below 100 L/hr.
	No LC flow.	Check solvent flow from the autosampler or syringe pump.
	A fluid leak developed in the HPLC system.	Examine the HPLC system for leaks and correct any you find.
	Broken fused silica capil- lary in the APCI probe.	Replace the fused silica capillary. See <u>Section 5.3.4</u> .
	Source components have been incorrectly assembled.	Ensure the source and probe voltage readbacks vary with Tune window settings. If any of these voltages are absent, disassemble and correctly reassemble the source and hexapole lens assemblies.
	Permanently blocked ESI or APCI capillary.	Replace capillary. See <u>Section 5.3.4</u> .

Symptom	Possible Cause	Corrective Action
Unsteady or low intensity peaks (Ion Beam)	Poor nebulization due to incorrect temperature and gas flow settings.	Check temperature and gas flow settings. Liquid inside the source enclosure is an indication that the tempera- ture is too low. The analyser pressure should be less than $1 \times 10^{-4}$ mbar. Nitrogen pres- sure should be from 90 to 100 psi, and the nitrogen flow rate should be greater than 100 L/hr. Check the stability of the nitrogen flow with a good quality, two-stage regulator.
	Problem with sample delivery (autosampler, syringe pump).	Troubleshoot the autosam- pler or check the syringe and syringe pump.
	Source components are dirty.	Clean source components. See <u>Section 5.2.5</u> .
	Cone voltage ramp is set to On.	Set the cone voltage ramp to Off.
	ESI or APCI capillary is not properly installed.	Ensure a capillary height setting of 0.5 mm. See <u>Section 5.3.4</u> .
Unusually high LC back- pressure	Blockage in the capillary transfer line or injection loop due to particulate matter from the sample.	Remove the probe from the source, and try to clear the blockage by increasing the solvent flow rate to 50 $\mu$ L/min.

Symptom		Possible Cause	Corrective Action
	Unusually high LC back- pressure	APCI probe filter pad is blocked.	Replace the filter pad. See <u>Section 5.3.4</u> .
	Continued	Tubing from LC system is blocked.	Remove the finger-tightened nut and tubing from the back of the probe. If the back pres- sure remains relatively high, replace the tubing.
		The ESI stainless steel sample capillary inside the probe is blocked.	Try reversing the capillary to clear any blockage. If this fails to correct the problem, replace the capillary.
		The ESI capillary is not fully seated in the LC union or the APCI capillary is not properly seated in the filter.	Remove and disassemble the probe, and reseat the capillary correctly in union.
	Unusually low LC back- pressure	Leaking connector.	Check all fittings and tighten where necessary.
		Problem with LC solvent delivery.	Troubleshoot the LC system.
		Broken flow cell in UV detector.	Replace the flow cell.
	Insufficient vacuum – (any reading higher than $1 \times 10^{-4}$ mbar on the Pirani	Leaking ion block O-rings.	Disassemble the source, and examine the ion block O-rings. See <u>Section 5.2.5</u> .
	gauge)	Roughing pump not oper- ating correctly.	Examine the vacuum pump oil. If it is dirty, flush the pump with clean oil. Then refill it with fresh oil. Repeat the procedure, if necessary.
		Leak in vacuum backing line.	Examine the vacuum hose for cracks or vacuum leaks.
		Restriction in vacuum pump exhaust tubing.	Examine the exhaust line for restrictions.
		The turbomolecular pump does not operate properly.	Check for operating defects.

Symptom	Possible Cause	Corrective Action
Leaking nitrogen (hissing sound or solvent smell)	The seal around the source enclosure is imperfect. Examine the source enclo- sure sealing surfaces for imperfections or nicks. Also examine the condition of th encapsulated O-rings.	
Vacuum oil accumulated in the exhaust tubing	Oil mist filter needs replacing.	Replace oil mist filter element and odor filter. See <u>Section 2.3.1</u> .
Vacuum oil accumulating inside oil mist housing	Oil return system works only when pump is ballasted.	Ballast the pump for 20 to 30 minutes. See <u>Section 5.2.3</u> .
The Source heaters do not operate.	Nitrogen flow is below 100 L/hr.	Set nitrogen flow to greater than 100 L/hr.
	A high ambient tempera- ture or obstructed air flow causes overheating.	Lower the ambient tempera- ture. Ensure sufficient air flow around the instrument. Remove and clean the fan screens.
The APCI heater does not operate.	If desolvation heater oper- ates, the APCI heater might need replacing.	Replace the APCI heater.
The instrument vents for no apparent reason.	The power supply is being interrupted.	Troubleshoot the line power.
Roughing pump fuse fails	The oil mist filter element is oil-saturated.	Replace the oil mist filter element and odor filter. Replace the fuse.
	The system needs ballasting.	Ballast the pump for 20 to 30 minutes. See <u>Section 5.2.3</u> .
	The line voltage is less than 208 VAC.	Ascertain that line voltage is within specifications.
	Vacuum pump oil is dirty.	Change vacuum pump oil. See <u>Section 5.2.2</u> .

6

Symptom	Possible Cause	Corrective Action
Ion Mode drop-down menu options are disabled or instrument spontane- ously switches probe type.	One or both of the probe contact pins jammed inside the probe and do not contact the probe support plate.	Remove probe cover, free the contact pin, and ensure that both pins and associ- ated springs move freely within the bushing.
	Probe adjustor contact pin not making contact.	Remove the probe adjustor assembly and check the position of the contact pin and support plate.
Failure to recognize one particular probe type.	Problem with the probe.	Remove and try another probe of the same type.
Ripple – peaks and base-	Erratic LC solvent flow.	Troubleshoot the LC system.
line appear to vary cyclically in intensity.	Poor nebulization due to incorrect temperature and gas flow settings.	Adjust temperature and gas flow settings. Liquid on the baffle plate indicates the temperature is too cold.
	Unstable power supplies in the source supplies or the RF/DC generator or unstable photomultiplier supply.	Check power supplies and RF generator voltages.
	Vibration from the rotary pumps or even other equip- ment in the same building.	Check for and eliminate excessive benchtop and instrument vibration.
Loss of communication with instrument	Instrument to MassLynx host communication failed.	Reset the workstation and restart MassLynx.
IEEE communication errors	Instruments powered up in the wrong sequence.	Shut off individual system components and start the system in this order: Work- station, ZQ Detector, Inlet modules. Start MassLynx, then wait 3 minutes for the audible signal.
	Wrong IEEE address or conflicting address.	Check system IEEE settings and enter the correct addresses.
	Faulty IEEE cable in IEEE chain.	Systematically replace IEEE cables until you isolate the problematic one.

Table 6-1	Hardware	Troubleshooting	(Continued)
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# 6.3 Inspecting the APCI Probe

- 1. Switch the instrument to **Standby** from the Tune window.
- 2. Make sure the probe heater is off.
- 3. Unplug the probe from the instrument front panel, and remove it from the source.
- 4. Connect the PTFE tube to the nebulizer outlet on the front panel.
- 5. Remove the probe heater assembly, carefully loosening the two set screws.
- 6. Disconnect the heater from the probe body by pulling it parallel to the axis of the probe.
- Ensure that 0.5 to 1 mm of fused silica protrudes from the stainless steel nebulizer tube.
- Connect the LC pump to the probe with a flow of acetonitrile : water, 1:1, at 1 mL/min.
- Make sure the liquid jet flows freely from the end of the capillary and that the LC pump pressure reads 250 to 400 psi.
- 10. Check that the nitrogen supply pressure is 90 to 100 psi (6 to 7 bar).
- 11. Click (API Gas) to start the nitrogen flow.
- 12. Make sure the liquid jet converts to a fine, uniform aerosol.
- 13. Stop the liquid flow.
- 14. Click (API Gas) to stop the nitrogen flow.
- 15. Reconnect the probe heater assembly.
- 16. Insert the APCI probe into the source, securing it by tightening the two thumbscrews.
- Connect the probe cable to APCI/ESI on the instrument's front panel. (Note that you
  must first remove the plug labeled ESI.)

# Appendix A Using the ESCi Multi-Mode Ionization Source

When fitted with the ESCi<sup>™</sup> Multi-Mode Ionization Source, the Waters<sup>®</sup> Micromass<sup>®</sup> ZQ<sup>™</sup> Mass Detector alternates between acquiring data in ESI and APCI ionization modes within the same run. This software-mediated capability depends on a high voltage power supply, which in ESCi mode switches continuously between supplying voltage to the ESI capillary and current to the APCI corona needle. Scanning occurs with the ESI probe installed and interscan delay times as short as 100 msec. The operating software, MassLynx<sup>™</sup> version 4.0, lets you select ionization modes and polarities in varying combinations and durations.

# A.1 Preparing for Operation

Preparing the ZQ Detector for ESCi operation entails these activities:

- · Installing the corona discharge needle
- Setting up the MassLynx software
- Performing the Daidzein test

#### A.1.1 Installing the Corona Discharge Needle

If you have been operating the instrument in ESI mode, you must install the corona discharge needle in the instrument's source. (If you have been operating it in APCI mode, the corona needle should be installed already.)

1. Remove the center panel from the front of the instrument, grasping its sides and pulling it toward you (Figure A-1).


Figure A-1 ZQ Mass Detector, Front View

2. Open the source enclosure cover.



**Caution:** Wear vinyl or latex gloves when working inside the source.

3. If the ESI probe is installed, loosen the two knurled thumbscrews that secure it to the source. Withdraw the probe several inches, enough to allow your hand easy access to the blanking plug.

If the APCI probe is installed, remove it, and replace it with the ESI probe. See the *Waters ZQ Detector Operator's Guide* for details about installing the ESI probe.



*Caution:* If you recently operated the instrument, you will find the source enclosure's inner surfaces hot.

- 4. Remove the blanking plug from the mounting contact.
- 5. Install the corona discharge needle on the mounting contact, orienting its tip toward the sample cone (Figure A-2).
- 6. Secure the ESI probe, source enclosure, and center panel.



Figure A-2 Installing the Corona Needle

### A.1.2 Setting Up MassLynx

Follow steps 1 through 5 in <u>Section 3.1</u> to open the MassLynx Tune window (Figure A-3).

ZQ - c:\masslynx\default.pro\acqudb\default.ipr	
File Ion Mode Calibration Gas Ramps Options Help	
ES+ Source         Analyser         Diagnostics           Voltages	Image         Span         Gain         Ion Mode           Image         1772         5         156         ES         Ion Mode           Image         2         772         5         156         APcl         Image: APcl<
BF Lens (V) 0 0.0	172.0 X 772.0 X 1622.0 X 3920.0
Temperatures           Source I emp (°C)         0           Desolvation I emp (°C)         0           Gas Flow         0           Desolvation (L/hr)         Cone (L/hr)           OFF         0FF           0FF         0FF	
Acquire	Press for Standby

Figure A-3 Tune Window

 Select Options > ESCi mode (Figure A-4). A check mark appears on the drop-down list, beside ESCi, and the Ion Mode column appears in the Peak Editor.

File Ion Mode Calibration Gas Ramps Options Help	
D 📽 🖩 🚳 水 〓 🗽 Readbacks Ion	Mode
Auditure. Auditure. Discontine. Score Barenbarr. Marc. Score Date Marce	moue
ES (dages) pragram dam infinition of the contraction of the contractio	umn
Contraction (Contraction) Contraction (Contr	
Applay (W) Processor Vent Vent Processor	
Cone (V) de 38 Vacuum Monitor	
APCI Voltages Swippe Type Vice Type	
Corona (uA) 7.7 Tes Set Instrument Threshold	
Cone (V) 36 31 Acquisition Status 172.0 56 772.0 56 1522.0 56 3920.0 56	
Communications Status 100.0% x100 45.5% x160 41.2% x131 54.1% x160	
Souce vorages much second	
Temperatures	
Servers Temp (**) 70 00	
and the second	
Desolvation Temp 119 120	
Gas Fow	
Desolvation (L/hr)         Cone (L/hr)	
261 0FF	
1/0.0 1/2.5 1/0.0 1/2.5 1/0.5 39200 392	
Acoure Press for Standby	

Figure A-4 Tune Window Showing Options List with ESCi Mode Selected

**Note:** During instrument operation, the Diagnostics page displays voltage-equivalent output in current (APCI) mode and current-equivalent output in voltage (ESI) mode (*Figure A-5*). Thus the Current and Voltage values appear to interchange, suggesting that current and voltage are simultaneously present. This, however, is not the case. These readings reflect only the power supply output, not voltage output at the capillary and current output at the corona needle.

**Note:** A blinking diagnostic indicator light, which in conventional ESI and APCI operation denotes an inoperative negative mode, is normal in ESCi operation.

ZQ - c:\masslynx\default.pro\acqudb\default.ipr Fie IonMode Calibration Gas Ramps Options Help	
D 🕫 🖬 🖨 🛝 🗮 🗽 🏋 😥 🔯 🖷 🕘 🎗	
ESCi+ Source         Analyser         Diagnostics           Tubb Speeds         Source Recognition         Source Recognition           Source Speed (%)         100         Source ID(v)         1.43           Pressues         Reference Volts         Reference Volts         195.7           Analogue         +430 Volts         438.7         193.7	Mass         Span         Gain         Ion Mode           IV         1         175         5         64         ES         Image: Span state           IV         2         607.5         5         64         APcI         Image: Span state         Image:
+28V DK -1350 Volts 1363.0 +24V DK +1350 Volts 1363.6	175.0         607.5         1082.0         607.5         1082.0           66.5%         >>64         363.%         >>64         10.8%         >>2
Misc. RF         Detector Supplies           RF Gen DC1         42.9	
RF Gen DC2 33.9 Diagnostic 2	
+5 Vols 5.01 Corona (uA) 90.39 Capillary (kV) 3.20	
	2.5 175.0 177 5.0 607.5 61C 080.0 1082.5 5.0 607.5 61C
Acquire	Press for Standby

Figure A-5 Tune Window, Diagnostics Page

Select Ion Mode > ESCi+ or ESCi- to enable the positive or negative ESCi ionization mode (Figure A-6).

20 - c:\masslymx\default.pro\acqudb\default.ipr		<u>_</u> _×
Prie Ion Mode Caloradon Gas Kamps Options Hep □ ✓ ESO+	2	
ESC Source   Analyser   Diagnostics   ES-V-Source   Analyser   Diagnostics   ES-Vollages Gapilay (IV)   320   313	Mate         Span         Gen         Est           172         5         20         55         56 <t< td=""><td>ion Mode 5 ¥ 6 ¥</td></t<>	ion Mode 5 ¥ 6 ¥
Cgne (V) 42 38 -	IF 3 1522 5 313 E	
APCI Voltages Corona (uA) 0.4 7.3	IF <u>4</u>  3920  5  156   <b>3</b>	
Cane (V) 42 31 -	172.0 🐹 772.0 🐹	1522.0 💥 3920.0 🕱
Source Voltages  Extractor (V)  BF Lens (V)  0.0	79.2% 200 100.0% 2000	X126 X120
Temperatures Source Temp (*C) 79 [90		
Desolvation Temp		
Gas Flow Desolvation (L/hr) Cone (L/hr)		
251 OFF		
250.0 - 0.0 -		
	170.0 172.5 770.0 772.5	520.0 1522.5 17.5 3920.0 392
Acquire		Press for Standby
Select ESCI+ ionisation mode	Vacuum OK	Operate

Figure A-6 Selecting the ESCi+ or ESCi- Ionization Mode

- 3. Optimize the fast-transitioning values, **ES Voltages**, **APCI Voltages**, and **Source Voltages**, for each mode and polarity (<u>Figure A-7</u>).
- Optimize the slow-transitioning values, Temperatures and Gas Flow, for both ESI and APCI performance (Figure A-7).

**Note:** The ESCi+ and ESCi– pages should display identical slow-transitioning values.

 Select APcI or ESI for each row in the Ion Mode column by clicking the drop-down list in each row (Figure A-7).

**Note:** You cannot select polarity from the Ion Mode column. Instead, click **Ion Mode** in the menu bar, and select mode and polarity from the drop-down list. This introduces the ESI+, ESI–, APCI+, or APCI– Source page, which determines an acquisition's polarity.

	Image: Synth default.pro (acquidb/default.ipro)         File Ion Mode Calibration Gas Ramps Options Help         Image: Image: Synthesis Control (Section Contro	
Fast- Transitioning Variables	ESC+5 Surce         Analyser         Diagnostics         Mass         Span         Gain         Ion Mode           ES Voltages         ES Voltages         I         172         5         20         ES         I         ICO	ו Mode List
Slow- Transitioning Variables	Temperatures         Souce Lenp (°C)       73       90       Peratorna       P	ak splay
	Acquire Press for Standby Press for Standby	
	roday yaddaiii ok joperace //	

Figure A-7 Selecting the Ion Mode in the MassLynx Peak Editor

Figure A-8 shows the Tune window as it appears while the instrument operates in ESCi mode.

**Note:** In the peak display, the APCI peaks appear in blue, the ESI peaks in red (*Figure A-11*).





 Select MS Method from the Main window shortcut bar (Figure 3-2). The MassLynx Function List Editor window appears, its list area blank (Figure A-9). See the MassLynx 4.0 Guide to ZQ Data Acquisition for details about this window.

☑ Experiment Setup - c:\masslynx\esci.pro\acqudb\esci me	thod.exp	
<u>File Edit Options Toolbars Functions</u>		MS Scan
	Scan	Button
Total Run Time: 0.00		
No. Type Information	Time	
😭 Start 🕅 MassLynx - DEFAULT - D [ 🔄 ZQ	🖉 Experiment Setup - c	

A

Figure A-9 Function List Editor Window (Blank)

7. Click **MS Scan**. The Function: *n* MS Scan dialog box (where n = the function list number) appears (Figure A-10).

Function:1 MS Scan	×	
Mass (m/z)	Method	
Start 100	Ionization Mode ES+  ES+	Function Modes and
End 1500	Data Centroid API+ API-	— Polarities
Time (Mins)	Scan Duration (secs)	
Start 0	Scan Time 0.5	
End 60	Inter-Scan Delay 0.1	
Cone Voltage	APcl Probe	
🗖 Use Tune Page	🔲 Use Tune Page Settings	
Cone Voltage (V) 31	Probe Temp 100	
🔲 Use Cone Voltage Ramp		
CV Ramp	OK Cancel	

Figure A-10 Function: *n* MS Scan Dialog Box

8. Enter parameter values for your sample, then click OK.

**Note:** Because you must complete this dialog box for each function you specify, runs with multiple functions require multiple iterations of this process.

Each time you click OK, the MassLynx Function List Editor window reappears listing the scan function you just specified (Figure A-11).



Figure A-11 Function List Editor Window Showing Specified Functions

 Select Options > ESCi mode to disable ESCi mode and resume normal ESI or APCI operation (Figure A-12). The check mark associated with ESCi on the drop-down list disappears, as does the Ion Mode column from the Peak Editor.

**Note:** You can perform ESI-only acquisitions in ESCi mode without compromising instrument performance. In doing so, however, be sure to remove the corona discharge needle and replace it with the blanking plug.

ZQ - c:\masslynx\default.pro\acqudb\del	fault.ipr											_	
File Ion Mode Calibration Gas Ramps Optic	ons Help												
	eadbacks												
ES+Source Analyser Diagnostics So	cope Parameters		Mass		Span	Gain							
Voltages Re	einitialize		172	5		20							
Capillary (kV) 3.20 3.13	ent	₹ 2	772	5		156							
Cone (V) 42 38	acuum Monitor	₩ 3	1522	5		313							
Extractor (V) 5 5	ringe Tune	✓ 4	3920	5		156							
BF Lens (V) 0.0 5e	et Instrument Threshold												
Temperatures	equisition Status		172.0	8		772.0	8		1522.0	8		3920.0	8
Serves Terrs (°C)	ommunications Status	100.0%	3	20	88.4%		×156	92.2%		×313	96.6%		×156
	strument Name												
Descrivation Temp (C) 118 11.	agnostics Page			-1			-				<u> </u>		
- Das Flow	5Ci mode												
Desolvation (L/hr) Cone (L/h	n) hà												
							-			-			
							_						
261 0FF										_			
				_			_	<u> </u>		_	<u> </u>		
				-1			-			_			
		170.0	470.5	-		770.5		500.0	4500		17.5	2020.0	
			172.5	1	170.0	172.5		520.0	1522.	0	17.5	3920.0	392
Acquire										P	ress for S	tandby	
			Vacuum Ok	<					Operate				

Figure A-12 ESCi Mode Disabled

### A.2 Daidzein Test

Use the Daidzein (Figure A-13) test to prepare the instrument for ESCi operation, evaluate its performance, and determine the ratio of peak heights to the level of noise in a mass chromatogram (signal-to-noise ratio).



Figure A-13 Daidzein (m/z = 255.2 [M + H] and 253.2 [M - H])

### A.2.1 Test Conditions

#### Table A-1 HPLC Conditions

Parameter	Setting
Column	2.1 × 50 mm, Xterra <sup>®</sup> C <sub>18</sub> (3 $\mu$ m)
Mobile phase	Water: acetonitrile, 70:30
Flow rate	0.5 mL/min
Back pressure	~ 2100 psi
Injection volume	5 μL
Temperature	Ambient
Run time	3.0 min.

#### Table A-2 MS Conditions

Parameter	Setting
lon modes	ES+/ ES-/ API-/ API+
Scan range	150 to 500 amu
Scan time	0.2 sec.
Interscan delay	0.1 msec
Photomultiplier tube	450 V
Acquisition time	3.0 min.

### A.2.2 Signal-to-Noise Ratio

See the MassLynx User's Guide for signal-to-noise theory and the Waters Micromass ZQ with MassLynx v4.0 Software and Instrument Verification Procedure for details about calculating the signal-to-noise ratio as it applies to the ZQ Detector.

A

#### Specifications 119

This appendix describes specifications for the Waters Micromass ZQ Detector and its ESCi option.

### **B.1 ZQ Detector Specifications**

Table B-1	ZQ Detector	Operational S	Specifications
-----------	-------------	---------------	----------------

Condition	Specification
Mass Range:	
2000 model	2 to 2000 amu
4000 model	2 to 4000 amu
ESI Sensitivity:	
Positive ion	Typically, 1 pg (5- $\mu$ L injection of 0.2 pg/ $\mu$ L) reserpine at 300 $\mu$ L/min flow rate = 50:1 S/N-RMS.
APCI Sensitivity:	
Positive ion	Typically, 1 pg (5- $\mu$ L injection of 0.2 pg/ $\mu$ L) reserpine at 1 mL/min flow rate = 50:1 S/N-RMS.
Dynamic range	Five orders of magnitude.
Spectral stability	Mass accuracy to within 0.2 da over a 24-hour period.
Adduct formation	The adduct ion at 273 da will be less than 30% of the molec- ular ion (232 da) for an infusion of 1 ng fenfluoramine in APCI positive mode using 1:1 acetonitrile : water as the solvent system.
Resolution	The peaks at 1080.8 and 1081.8 from an infusion of PA beta cyclodextrin shall be resolved with a valley between them of no more than 10% of the average height of the two peaks.

Table B-2 ZQ Detector Environmental Specifications

Condition	Specification
Operating temperature range	15 to 28 °C (59 to 82.4 °F)
Operating humidity range	20 to 80%, noncondensing
Shipping and storage temperature	–20 to 60°C (–4 to 140 °F)

#### Table B-3 ZQ Detector Dimensions

Condition	Specification
Height	23 in. (57.2 cm)
Depth	26 in. (66.1 cm)
Width	15.2 in. (38.1 cm)
Weight (excluding data system and rotary pump)	210 lb. (95.3 kg)

#### Table B-4 ZQ Detector Electrical Specifications

Condition	Specification
Line frequency	50 Hz, 47 to 53 Hz 60 Hz, 57 to 63 Hz
Fuse rating	F 2 A, 250 VAC
Analog inputs	To 1 VAC
Power consumption	140 VA (Nominal)

### **API Setup Solution**

Polypropylene glycol/reserpine/cyclodextrin

### B.2 ESCi Multi-Mode Ionization Source Specifications

#### Table B-5 ESCi Environmental Specifications

ltem	Specification
Operating temperature range	0 to 45 °C
Altitude	Up to 2000 m
Maximum relative humidity	80% for temperatures up to 31 $^{\circ}$ C, decreasing linearly to 50% at 40 $^{\circ}$ C

Table B-6 ESCi Electrical Specifications

ltem	Specification	
Input voltage	+24 VDC ±10%	
Current	1.7 A, maximum	

# Appendix C Accessories and Spare Parts

The tables in this appendix list and indicate the part number for recommended spare parts and accessories.

Table	C-1	Fuses
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Item	Part Number
Fuse, 0.5A VFA, SMT little fuse 154.500	700001371
Fuse, 1A, SB, SMT little fuse 154 00IT	700001364
Fuse, 1.5A, SB, SMT little fuse 154 01.5T	700001365
Fuse, 2.5A SB, SMT little fuse 154 02.5T	700001366
Fuse, 2A VFA, SMT R451002	700001370
Fuse, 2A VFA, SMT little fuse 154.002	700001372
Fuse, 3.5A SB, SMT little fuse 154 03.5T	700001367
Fuse, 3A G, FA, 1.25 x 0.25-in. little fuse 312 010	700001362
Fuse, 5A SB, SMT little fuse 154 005T	700001368
Fuse, 8A, Anti-surge Ceramic 1.25 in.x 0.25 in.	700001363
Fuse, A1, FB, SMT little fuse 154, 1F 664-844	700001369
Fuse, Anti-surge 0.375 SMT 154	700001361
ZQ Spare Fuse Kit	700001716

#### Table C-2 Probe Components

Item	Part Number
APCI Probe	70000290
Assy., Heater Shroud, APCI	700000441
Coupling, APCI-ESI, Probe	700000966
ESI Probe	70000289
ESI Probe Assy.	70000289
ESI Probe End Cover	700001667
ESI Probe PEEK Shaft	700002056
Ferrule, 1/16 x 0.3 Bore, GVF	415000110
Ferrule, APCI-GV004, ZQ	700000344

### Table C-2 Probe Components (Continued)

Item	Part Number
Ferrule, ESI-GVF16-16	700000343
Heater, APCI Probe	279000112
In-line Filter Anachem A314	600000106
Liner, Ferrule, ESI Probe	430000110
O-Ring, APCI-ESI, Coupling	700000968
Pad, Filter	600000103
Pin, Corona APCI	700000354
Probe adjustor Assy.	700001714
Silica, Fused, APCI Probe	430000109
Sleeve, ESI Conductive	700000969
Tip, Probe Assy.	700000337
Tube, Liner (Normal Flow)	700000640
Tube, PTFE, ESI Probe	430000111

#### Table C-3 Vacuum Components

Item	Part Number
Adaptor NW25, 1/2 Hose	WAT240495
Clamp, Hose 1 in. SS Box 10	WAT241163
Clamp, Hose, 5/8 in. SS Box 10	WAT241162
Controller, Turbo Pump, 24 V	700001127
Edwards 45 Oil, 1 Gallon	70000870
Edwards, ISO 100 Ctr Ring, Viton	700001314
Edwards Oil Return Kit	700000573
Edwards Pump Kit	700001289
Gauge, Pirani, APG L, NW15	700001129
Installation & Oil Filters	700001288
Jubilee Hose Clamp #2	700000468
<i>Note:</i> You can use this part instead of part number WAT241162.	
LCZ/ZMD Filter, Odor Element	70000871
Ms Oil Mist Filter Housing w/Element	WAT241107
Ms VAC Hose PVC 1 in., 25 ft.	WAT241165
Ms VAC Hose PVC 1/2 in., 30 ft.	WAT241168

### Table C-3 Vacuum Components (Continued)

Item	Part Number
Ms VAC Hose PVC ¾ in., 20 ft.	WAT241397
NW25 Blanking Cap	WAT240463
NW25 Center Ring, Viton	WAT241160
NW25 Elbow 90 Deg.	WAT240443
NW25 Swing Clamp	WAT240696
NW25 Tee	WAT240462
Oil Mist Filter Element	WAT240447
Pump, Split Flow Turbo, Edwards 200/200H	700001128
Tailpiece, NW25-NW25	700001157
VAC NW25 Flange, Lg Butt	WAT240828

#### Table C-4 Valves and Flow Meters

Item	Part Number
Kalrez, 0.145 x 0.070 mm	70000903
Kalrez, 0.240 x 0.063 mm	70000902
Tailpiece, NW25-NW25	700001157
Viton, 0.984 x 0.139	700000901
Viton, 12 x 1.5 mm	700001194
Viton, 12 x 1.5 mm	700001167
Viton, 2-010	700000452
Viton, 2-013	WAT241666
Viton, 2-025	WAT241431
Viton, 2-035	700000904
Viton, 2-039	425000112
Viton, 39 x 2.5 mm	700001056
Viton, AS016	700000355
Viton, AS035, PTFE	700001192
Viton, AS039	700001199
Viton, AS214	700001199

Table C-5 Kits

Item	Part Number
APCI Probe, Kit Consumable	700000464
APCI Probe, Spare Assy. Kit	700000338
API Calibration Kit	700001593
API Calibration Solution (NACsi)	700001594
API Test Kit	70000889
Basic Spares & Tool Kit	700001287
Edwards Oil Return Kit	700000573
Edwards Pump Kit (contains the oil return connection kit, reference number A5042000)	700001289
ESI Probe Assy. Spares Kit	70000336
ESI Probe Installation Kit	700001290
FractionLynx Dye Kit	716000765
Installation & Oil Filters Kit	700001288
Kit, Consumable, ESI Probe	700000463
PM Kit Protocol (ZQ)	715000336
Reserpine Kit	700001673
ZQ PM Kit	201000123
ZQ Source Consumables Kit	700001296
ZQ Source Spares Kit	700001291

### Table C-6 Power Supplies

Item	Part Number
1000 W DC Power Supply	700001334
DC Power Supply, CA 10000 PSU	700001134
Embedded PC, ZQ	700001606
Generator, RF ZQ 4000	700001300
Generator, RF, ZQ 2000	700001139
GPIB Bd & S/W	186000825
Headamp, Assy., ZQ	700000281
Low Noise Headamp	700001123
Pcb, Ext. Conn., ZQ	700001135
Power Supply, 28,28,24	700001125
Power Supply, 3.3,5,15,15,28	700001126

#### Table C-6 Power Supplies (Continued)

Item	Part Number
Switch, Main	700001146
ZQ A/D Main Sys Board	700001804
ZQ Brandenburg PSU DN 1044	700001140
ZQ Brandenburg PSU DN1043	700001141
ZQ Brandenburg PSU DN1047 Mux Type	700001142
ZQ Single PS Main Wiring	700001360
ZQ Single PS Wiring	700001359
ZQ, I/O Board	700001805

Table C-7 Source and Analyser Components

Item	Part Number
Assy., Detector, ZQ	700001161
Block, Sample Cone Locator	700001245
Block, Terminal	700001251
Cone, Gas Nozzle, ZQ	700001236
Cone, Sample, ZQ	700001237
Connector, PEEK, Body Assy.	700001202
Cover Plate	700001234
Extraction Cone, ZQ	700001238
Fastener, ION Block, ZQ	700001241
Heater Assy., ZQ Source	700001201
Hexapole, Assy., ZQ	700001132
ION Block Assy. ZQ	700001198
ION Block, ZQ	700001235
Lens, Phosphor, Platform LC	279000128
Lid, Assy., Quad, ZQ	700001134
Photomultiplier Platform LC	2790000127
Photomultiplier Platform LC	2790000127
Plug M10	700000906
Plug, Hole, Peek, ION Body ZQ	700001243
Retainer, Extractor, ZQ	700001242
Retainer, Sleeve, Z-couple	700001239

### Table C-7 Source and Analyser Components

Item	Part Number
Seal Ring, ZQ	700001244
Seal, Plug	700000907
Spring Clip Cone Gas, ZQ	700001232
Stem, Valve	700001240
Support, ION Block ZQ	700001233
ZQ 4000, Lid, Quad Assy.	700001301
ZQ Source Assy.	700001222
ZQ Source Spares Kit	700001291

Table C-8 Washers, Screws, and Nuts

Item	Part Number
Hex, Nut, M4, Metric	WAT410400
Hex, Nut, M4, Metric	WAT410400
LCZ/ZMD/ZA Attach Screw APCI Probe	700000577
LCZ/ZMD/ZQ APCI Knob Grub Screw M3x3	700000579
LCZ/ZMD/ZQ Attach Screw ESI Probe	700000576
LCZ/ZMD/ZQ Attach Screw Knob APCI	700000578
M2x2 APCI Grub Screw Slotted	700000967
Screw Flat HD, M3x6	WAT041240
Screw, M2x6 CH HD, S.S.	700001195
Screw, M2x6 SKT HD CAP	700001168
Screw, M3x20 Cross	410000578
Screw, M4x40 S/H, SST	410000523
Screw, Metric, M3x6 PH	WAT411306
Washer Teflon, Ion block fastener	700001929
Washer, Flat, M4	WAT410410
Washer, Large Valve Stem	700001038
Washer, Lock Split M2	WAT410240
Washer, M3 S.S	700001197
Washer, Small, Valve Stem	700001039

### Table C-9 Miscellaneous Components

Item	Part Number
Basic Spares & Tool Kit	700001287
Cover, LHS, ZQ	700001120
Card, INTEL Pro100	210000169
Cover, RHS, ZQ	700001121
Cover, Top, ZQ	700001119
Guard, Fan 120MM, RS2584639	700001151
Kit, Rebuild, Rheo, RV700-100	700001303
LCZ/ZMD/ZQ Glass Eye Loupe	70000883
Molding, Front, ZQ	700001122
Sample Loop, 10 µL S.S. Rheo, RV700-100	700001302
Stud, Locating Pho/Bronze	700001781
ZQ Instrument Spares Kit	700001118
ZQ Syringe Pump	700001316
ZQ, Power Cord Euro	700001293
ZQ, Power Cord UK	700001292

# Index

# A

Accessories 122 Acquisition parameters, specifying 64 Acquisitions, scanning 71 Adding rotary pump oil 33 Analyzer components 126 Analyzer page 57 APCI theory 29 tuning in APCI mode 47 APCI probe 109 cleaning the tip 91 inspecting 107 replacing heater 98 APCI+ Source page parameters 49 Apply span correction 60 Auto peak matching 61 Automatic Calibration Check dialog box 59 Automatic Calibration dialog box 63

# B

Bench space <u>31</u> Blanking plug <u>109</u>

# С

Calibrating mass scale <u>54</u> Calibration checking <u>67</u> failure <u>69</u> incorrect <u>70</u> saving <u>71</u> verifying <u>71</u> Calibration Acquisition dialog box <u>64</u> Calibration acquisition ranges, check <u>60</u> Calibration Acquisition Setup dialog box <u>64</u> Calibration file, setting up 54 Calibration Parameters dialog box <u>60</u> Calibration parameters, setting 55 Calibration procedure 63 Calibration report 67 printing 68 updating 68 Capillary replacing fused silica 96 replacing stainless steel 93 Check calibration acquisition ranges 60 Checking calibration 67 oil level 33 rotary pump oil 75 Cleaning APCI probe tip 91 cone gas nozzle 84 corona discharge needle 91 hexapole assembly 89 source 81 source assembly 80 Clearance 31 Components analyzer 126 miscellaneous 128 probe 122 source 126 vacuum 123 Cone extraction 88 gas cone 87 retainer 87 Cone gas nozzle, cleaning 84 Connecting nitrogen supply and exhaust 35 rheodyne injector 37 Contacting Waters Technical Service 33, 74, 100

Conventions, documentation <u>24</u> Corona discharge needle installing <u>108</u> removing <u>108</u>

## D

Daidzein test 117 conditions 118 signal-to-noise ratio 118 Damage, reporting 33 Data type 66 Default threshold parameters, adjusting 58 Diagnostics page 112 Dialog boxes Automatic Calibration 63 Automatic Calibration Check 59 Calibration Acquisition 64 Calibration Acquisition Setup 64 Calibration Parameters 60 Display Calibration Graphs 67 Function: *n* MS Scan 115 Instrument Threshold Settings 58 Login 41 Mass Measure 62 Open 55 Readback 52 Save As 68 Syringe Selection 45 Dimensions, ZQ Detector 120 Display Calibration Graphs dialog box 67 Documentation conventions 24 related 23 Drying gas exhaust bottle, installing 36 Duration, run 66

# E

Electrical specifications, ESCi <u>121</u> Electromagnetic fields <u>31</u> Electrospray ionization <u>28</u> Environmental specifications, ESCi <u>121</u> Environmental specifications, ZQ Detector <u>120</u> ES+ Source page parameters <u>43</u> ESCi- mode <u>112</u> ESCi Multi-Mode Ionization Source <u>108</u> ESCi+ mode <u>112</u> ESI probe <u>109</u> installing <u>38</u> replacing tip <u>95</u> ESI tuning <u>44</u> Exhaust systems, separation of <u>37</u>

### F

Fast scan time <u>66</u> Filter, replacing oil mist <u>79</u> Flow meters <u>124</u> Function List Editor window <u>116</u> Function: *n* MS Scan dialog box <u>115</u> Fuses <u>122</u>

### G

Gas-ballasting 76, 78

## Η

Hardware, troubleshooting <u>101</u> Hexapole assembly, removing and cleaning <u>89</u>

# 1

Initial error <u>61</u> Injector, connecting <u>37</u> Inspecting APCI probe <u>107</u> Installing corona discharge needle 108 drying gas exhaust bottle 36 ESI probe 38 Oil return connection kit 34 rotary pump 33 Instrument Threshold Settings dialog box 58 Intensity threshold 61 Intensity weighting 61 Inter scan delay 66 Ion block 87 Ion block assembly 83 Ion block cartridge heater, replacing 92 Ion mode, selecting in MassLynx Peak Editor 113 Ionization mode, selecting ESCi+ or ESCi-112 Isolation valve 78

# K

Kits <u>125</u> Startup <u>32</u>

## L

Login dialog box 41

# M

Mass analyser <u>28</u> Mass Measure dialog box <u>62</u> Mass scale, calibrating <u>54</u> MassLynx Diagnostics page <u>112</u> Function List Editor window <u>116</u> Login dialog box <u>41</u> Main window <u>42</u> opening <u>41</u> security <u>41</u> Matched peaks, manually editing 71 Maximum standard deviation 60 Miscellaneous components 128 Missed reference peaks 59 Modes ESCi- 112 ESCi+ 112 ion 113 ionization 112 Multi-Mode Ionization Source 108

# N

Needle corona discharge <u>85</u> corona discharge, clean and replace <u>91</u> Nitrogen stud <u>36</u> Nitrogen supply and exhaust, connecting <u>35</u> Nuts <u>127</u>

## 0

Pump oil <u>34</u> Oil level, checking <u>33</u> Oil mist filter, replacing <u>79</u> Oil return connection kit, installing <u>34</u> Open dialog box <u>55</u> Opening MassLynx <u>41</u> Operating specifications, ZQ Detector <u>119</u> Operation, preparing for <u>108</u> Overview ESCi Multi-Mode Ionization Source <u>108</u> ZQ Detector <u>27</u>

### P

Parameters acquisition <u>64</u> Analyser page <u>46</u> APCI+ source <u>49</u> calibration <u>55</u> ES+ Source page <u>43</u> Parts fuses 122 power supplies 125 spare 122 Startup Kit 32 Peak window 61 Peaks matched 71 missed reference 59 Periodic maintenance schedules 74 Pirani gauge 28 Plug, blanking 109 Polynomial order 61 Power requirements 31 Power supply connecting to 40 parts 125 Preparing for operation 108 syringe and syringe pump 40 Printing Calibration report 68 Probe adjustor 46, 51 Probes APCI 109 components 122 ESCi 109 Pump oil, replacing 75 Pumping block assembly 82 Pumps rotary 33, 75 syringe 40 turbomolecular 27 vacuum 33

## R

Readback dialog box <u>52</u> Readbacks <u>52</u> Reference solution, suggested values <u>56</u> Related documentation <u>23</u> Relative humidity <u>31</u> Removing corona discharge needle 108 hexapole assembly 89 ZQ Detector center panel 108 Replacing APCI probe heater 98 corona discharge needle 91 ESI probe tip 95 fused silica capillary 96 ion block cartridge heater 92 oil mister filter 79 pump oil 75 stainless steel capillary 93 Rheodyne injector, connecting 37 Rotary pump adding oil 33 checking oil 75 checking oil level 33 installing 33 Run duration 66

# S

Sample cone, cleaning 84 Save As dialog box 68 Saving calibrations 71 Scan from parameter 66 Scan speeds for calibrants other than sodium cesium iodide 66 Scan times 66 Scan to parameter 66 Scanning acquisitions 71 Screws 127 Security, MassLynx 41 Selecting ESCi+ or ESCi- 113 ion mode 113 Setting calibration parameters 55 Setting up calibration file 54 MassLynx 41, 110

Settings calibration 55 Tune page 55 Shipping damage, reporting 33 Signal-to-noise ratio 118 Slow scan time 66 Software, MassLynx 4.0 29 Source assembly, cleaning 80 Source cleaning 81 Source components 126 Source parameters, APCI+ Source page 49 Span correction, apply 60 Spare parts 122 Specifications 119 ESCi electrical 121 ESCi environmental 121 ZQ Detector environmental 120 ZQ Detector operating 119 Startup Kit 32 Static dwell 66 Static electricity 31 Static span 66 Syringe pump, preparing 40 Syringe Selection dialog box 45 Syringe stop 40

# T

Temperature, operating <u>31</u> Test, Daidzein <u>117</u> Threshold, intensity <u>61</u> Troubleshooting <u>99</u> hardware <u>101</u> Tune page Analyzer tab <u>57</u> settings <u>55</u> Tune window <u>42</u>, <u>110</u> Analyser page <u>46</u> APCI+ page <u>49</u> Diagnostics page <u>112</u> during ESCi operation <u>114</u> ES+ Source page <u>43</u> Tuning in ESI <u>44</u> Turbomolecular pump <u>27</u>

# U

Unpacking ZQ Detector <u>32</u> Updating Calibration report <u>68</u>

# V

Vacuum components <u>123</u> Vacuum pump oil level, checking <u>33</u> Vacuum system <u>27</u> Valves <u>124</u> Venting the source <u>83</u> Verifying calibrations <u>71</u> Vibration <u>31</u> Voltage <u>31</u>

# W

Washers <u>127</u> Windows Function List Editor <u>115</u> Main <u>42</u> Peak <u>61</u> Tune <u>42</u>, <u>110</u>

### Ζ

ZQ Detector dimensions <u>120</u> overview <u>27</u> removing center panel <u>85</u>, <u>90</u> removing center panel in ESCi Multi-Mode Ionization Source <u>108</u> unpacking <u>32</u>