

Ion Max and Ion Max-S API Source

Hardware Manual

97055-97044 Revision C

January 2008



© 2008 Thermo Fisher Scientific Inc. All rights reserved.

PhotoMate is a trademark of Syagen Technology. Kimwipe is a trademark of Kimberly-Clark Corporation. Granville-Phillips 342 is a trademark of Helix Technology Corporation.

The following are registered trademarks in the United States and other countries: Microsoft and Windows are registered trademarks of Microsoft Corporation. Unimetrics is a registered trademark of Unimetrics Company. Tygon is a registered trademark of Saint-Gobain Performance Plastics Company. Granville-Phillips and Convectron are registered trademarks of Helix Technology Corporation. Agilent is a registered trademark of Agilent Technologies, Inc. Intel and Pentium are registered trademarks of Intel Corporation. KEL-F is a registered trademark of 3M Company. Swagelok is a registered trademark of Crawford Fitting Company. Rheodyne is a registered trademark of Rheodyne, L.P. Delrin, Kalrez, Teflon, Tefzel, and Viton are registered trademarks of E.I. du Pont de Nemours & Co. Upchurch Scientific is a registered trademark of Upchurch Scientific, Inc.

Thermo Fisher Scientific Inc. provides this document to its customers with a product purchase to use in the product operation. This document is copyright protected and any reproduction of the whole or any part of this document is strictly prohibited, except with the written authorization of Thermo Fisher Scientific Inc.

The contents of this document are subject to change without notice. All technical information in this document is for reference purposes only. System configurations and specifications in this document supersede all previous information received by the purchaser.

Thermo Fisher Scientific Inc. makes no representations that this document is complete, accurate or errorfree and assumes no responsibility and will not be liable for any errors, omissions, damage or loss that might result from any use of this document, even if the information in the document is followed properly.

This document is not part of any sales contract between Thermo Fisher Scientific Inc. and a purchaser. This document shall in no way govern or modify any Terms and Conditions of Sale, which Terms and Conditions of Sale shall govern all conflicting information between the two documents.

Release history: Revision A released 2005, Revision B released 2006, Revision C released 2008.

For Research Use Only. Not regulated for medical or veterinary diagnostic use by U.S. Federal Drug Administration or other competent authorities.



Regulatory Compliance

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations. When the system is delivered to you, it meets all pertinent electromagnetic compatibility (EMC) and safety standards as described below.

EMC Directive 89/336/EEC as amended by 92/31/EEC and 93/68/EEC

EMC compliance has been evaluated by U.L. Underwriter's Laboratory Inc.

EN 55011: 1998 EN 61000-4-3: 2002

EN 61000-3-2: 1995, A1: 1998, A2: 1998, A14: 2000 EN 61000-4-4: 1995, A1: 2001, A2: 2001

EN 61000-3-3: 1998 EN 61000-4-5: 1995, A1: 2001 EN 61326-1: 1998 EN 61000-4-6: 1996, A1: 2001 EN 61000-4-2: 2000 EN 61000-4-11: 1994, A1; 2001

FCC Class A, CFR 47 Part 15 and Part 18 CISPR 11: 1999, A1: 1999, A2: 2002

Low Voltage Safety Compliance

This device complies with Low Voltage Directive 73/23/EEC and harmonized standard EN 61010-1:2001.

Changes that you make to your system may void compliance with one or more of these EMC and safety standards. Changes to your system include replacing a part or adding components, options, or peripherals not specifically authorized and qualified by Thermo Fisher Scientific. To ensure continued compliance with EMC and safety standards, replacement parts and additional components, options, and peripherals must be ordered from Thermo Fisher Scientific or one of its authorized representatives.

FCC Compliance Statement

THIS DEVICE COMPLIES WITH PART 15 OF THE FCC RULES. OPERATION IS SUBJECT TO THE FOLLOWING TWO CONDITIONS: (1) THIS DEVICE MAY NOT CAUSE HARMFUL INTERFERENCE, AND (2) THIS DEVICE MUST ACCEPT ANY INTERFERENCE RECEIVED, INCLUDING INTERFERENCE THAT MAY CAUSE UNDESIRED OPERATION.



CAUTION Read and understand the various precautionary notes, signs, and symbols contained inside this manual pertaining to the safe use and operation of this product before using the device.



Notice on Lifting and Handling of Thermo Fisher Scientific Instruments

For your safety, and in compliance with international regulations, the physical handling of this Thermo Fisher Scientific instrument *requires a team effort* to lift and/or move the instrument. This instrument is too heavy and/or bulky for one person alone to handle safely.

Notice on the Proper Use of Thermo Fisher Scientific Instruments

In compliance with international regulations: Use of this instrument in a manner not specified by Thermo Fisher Scientific could impair any protection provided by the instrument.

Notice on the Susceptibility to Electromagnetic Transmissions

Your instrument is designed to work in a controlled electromagnetic environment. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

For manufacturing location, see the label on the instrument.



WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the following symbol:



Thermo Fisher Scientific has contracted with one or more recycling or disposal companies in each European Union (EU) Member State, and these companies should dispose of or recycle this product. See www.thermo.com/WEEERoHS for further information on Thermo Fisher Scientific's compliance with these Directives and the recyclers in your country.

WEEE Konformität

Dieses Produkt muss die EU Waste Electrical & Electronic Equipment (WEEE) Richtlinie 2002/96/EC erfüllen. Das Produkt ist durch folgendes Symbol gekennzeichnet:



Thermo Fisher Scientific hat Vereinbarungen mit Verwertungs-/Entsorgungsfirmen in allen EU-Mitgliedsstaaten getroffen, damit dieses Produkt durch diese Firmen wiederverwertet oder entsorgt werden kann. Mehr Information über die Einhaltung dieser Anweisungen durch Thermo Fisher Scientific, über die Verwerter, und weitere Hinweise, die nützlich sind, um die Produkte zu identifizieren, die unter diese RoHS Anweisung fallen, finden sie unter www.thermo.com/www.therm



Conformité DEEE

Ce produit doit être conforme à la directive européenne (2002/96/EC) des Déchets d'Equipements Electriques et Electroniques (DEEE). Il est marqué par le symbole suivant:



Thermo Fisher Scientific s'est associé avec une ou plusieurs compagnies de recyclage dans chaque état membre de l'union européenne et ce produit devrait être collecté ou recyclé par celles-ci. Davantage d'informations sur la conformité de Thermo Fisher Scientific à ces directives, les recycleurs dans votre pays et les informations sur les produits Thermo Fisher Scientific qui peuvent aider la détection des substances sujettes à la directive RoHS sont disponibles sur www.thermo.com/WEEERoHS.

CAUTION Symbol	CAUTION	VORSICHT	ATTENTION	PRECAUCION	AVVERTENZA
4	Electric Shock: This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	Elektroschock: In diesem Gerät werden Hochspannungen verwendet, die Verletzungen verursachen können. Vor Wartungsarbeiten muß das Gerät abgeschaltet und vom Netz getrennt werden. Betreiben Sie Wartungsarbeiten nicht mit abgenommenem Deckel. Nehmen Sie die Schutzabdeckung von Leiterplatten nicht ab.	Choc électrique: L'instrument utilise des tensions capables d'infliger des blessures corprelles. L'instrument doit être arrêté et débranché de la source de courant avant tout intervention. Ne pas utiliser l'instrument sans son couvercle. Ne pas elensver les étuis protecteurs des cartes de circuits imprimés.	Descarga eléctrica: Este instrumento utiliza altas tensiones, capaces de producir lesiones personales. Antes de dar servicio de mantenimiento al instrumento, éste debera apagarse y desconectarse de la línea de alimentacion eléctrica. No opere el instrumento sin sus cubiertas exteriores quitadas. No remueva las cubiertas protectoras de las tarjetas de circuito impreso.	Shock da folgorazione. L'apparecchio è alimentato da corrente ad alta tensione che puo provocare lesioni fisiche. Prima di effettuare qualsiasi intervento di manutenzione occorre spegnere ed isolare l'apparecchio dalla linea elettrica. Non attivare lo strumento senza lo schermo superiore. Non togliere i coperchi a protezione dalle schede di circuito stampato (PCB).
	Chemical: This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	Chemikalien: Dieses Gerät kann gefährliche Chemikalien enthalten. Tragen Sie Schutzhandschuhe beim Umgang mit toxischen, karzinogenen, mutagenen oder ätzenden/reizenden Chemikalien. Entsorgen Sie verbrauchtes Öl entsprechend den Vorschriften in den vorgeschriebenen Behältern.	Chimique: Des produits chemiques dangereux peuven se trouver dans l'instrument. Proted dos gants pour manipuler tous produits chemiques toxiques, cancérigènes, mutagènes, ou corrosifs/irritants. Utiliser des récipients et des procédures homologuées pour se débarrasser des déchets d'huile.	Química: El instrumento puede contener productos quimicos peligrosos. Utilice guantes al manejar productos quimicos tóxicos, carcinogenos, mutagenos o corrosivos/irritantes. Utilice recipientes y procedimientos aprobados para deshacerse del aceite usado.	Prodotti chimici. Possibile presenza di sostanze chimiche pericolose nell'apparecchio. Indossare dei guanti per maneggiare prodotti chimici tossici, cancerogeni, mutageni, o corrosivi/irritanti. Utilizzare contenitori aprovo e seguire la procedura indicata per lo smaltimento dei residui di olio.
<u></u>	Heat: Before servicing the instrument, allow any heated components to cool.	Hitze: Warten Sie erhitzte Komponenten erst nachdem diese sich abgekühlt haben.	Haute Temperature: Permettre aux composants chauffés de refroidir avant tout intervention.	Altas temperaturas: Permita que lop componentes se enfríen, ante de efectuar servicio de mantenimiento.	Calore. Attendere che i componenti riscaldati si raffreddino prima di effetturare l'intervento di manutenzione.
	Fire: Use care when operating the system in the presence of flammable gases.	Feuer: Beachten Sie die einschlägigen Vorsichtsmaßnahmen, wenn Sie das System in Gegenwart von entzündbaren Gasen betreiben.	Incendie: Agir avec précaution lors de l'utilisation du système en présence de gaz inflammables.	Fuego: Tenga cuidado al operar el sistema en presencia de gases inflamables.	Incendio. Adottare le dovute precauzioni quando si usa il sistema in presenza di gas infiammabili.
	Eye Hazard: Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	Verletzungsgefahr der Augen: Verspritzte Chemikalien oder kleine Partikel können Augenverletzungen verursachen. Tragen Sie beim Umgang mit Chemikalien oder bei der Wartung des Gerätes eine Schutzbrille.	Danger pour les yeux: Dex projections chimiques, liquides, ou solides peuvent être dangereuses pour les yeux. Porter des lunettes de protection lors de toute manipulationde produit chimique ou pour toute intervention sur l'instrument.	Peligro par los ojos: Las salicaduras de productos químicos o particulas que salten bruscamente pueden causar lesiones en los ojos. Utilice anteojos protectores al mnipular productos químicos o al darle servicio de mantenimiento al instrumento.	Pericolo per la vista. Gli schizzi di prodotti chimici o delle particelle presenti nell'aria potrebbero causare danni alla vista. Indossare occhiali protettivi quando si maneggiano prodotti chimici o si effettuano interventi di manutenzione sull'apparecchio.
<u>^</u>	General Hazard: A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual.	Allgemeine Gefahr: Es besteht eine weitere Gefahr, die nicht in den vorstehenden Kategorien beschrieben ist. Dieses Symbol wird im Handbuch außerdem dazu verwendet, um den Benutzer auf Anweisungen hinzuweisen.	Danger général: Indique la présence d;un risque n'appartenant pas aux catégories citées plus haut. Ce symbole figure également sur l'instrument pour renvoyer l'utilisateur aux instructions du présent manuel.	Peligro general: Significa que existe un peligro no incluido en las categorias anteriores. Este simbolo también se utiliza en el instrumento par referir al usuario a las instrucciones contenidas en este manual.	Pericolo generico. Pericolo non compreso tra le precedenti categorie. Questo simbolo è utilizzato inoltre sull'apparecchio per segnalare all'utente di consultare le istruzioni descritte nel presente manuale.
	When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific San Jose Products.	Wenn Sie sich über die Sicherheit eines Verfahrens im unklaren sind, setzen Sie sich, bevor Sie fortfahren, mit Ihrer lokalen technischen Unterstützungsorganisation für Thermo Fisher Scientific San Jose Produkte in Verbindung.	Si la sûreté d'un procédure est incertaine, avant de continuer, contacter le plus proche Service Clientèle pour les produits de Thermo Fisher Scientific San Jose.	Cuando la certidumbre acerca de un procedimiento sea dudosa, antes de proseguir, pongase en contacto con la Oficina de Asistencia Tecnica local para los productos de Thermo Fisher Scientific San Jose.	Quando e in dubbio la misura di sicurezza per una procedura, prima di continuare, si prega di mettersi in contatto con il Servizio di Assistenza Tecnica locale per i prodotti di Thermo Fisher Scientific San Jose.

AUTION Symbol	CAUTION	危険警告	危險警告
4	Electric Shock: This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	電撃:この計測器は高電圧を使用し、人体に危害を与える可能性があります。 保守・修理は、必ず操業を停止し、電源を切ってから実施して下さい。上部カ バーを外したままで計測器を使用しないで下さい。プリント配線 板の保護カバーは外さないで下さい。	電擊:儀器設備使用會造成人身傷害的高伏電壓。在維修之前, 必須先關儀器設備並切除電源。務必要在頂蓋蓋上的情況下操作 儀器。請勿拆除PCB保護蓋。
	Chemical: This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	化学物質: 危険な化学物質が計測器中に存在している可能性があります。毒性、 発がん性、突然変異性、腐食・刺激性などのある薬品を取り扱う際は、手袋を 着用して下さい。廃油の処分には、規定の容器と手順を使用して下さい。	化學品:儀器設備中可能存在有危險性的化學物品。接觸毒性 致癌、誘變或腐蝕/刺激性化學品時,請配帶手套。處置廢油 時,請使用經過許可的容器和程序。
	Heat: Before servicing the instrument, allow any heated components to cool.	熱:熱くなった部品は冷えるのを待ってから保守・修理を行って下さい。	高溫:請先等高溫零件冷卻之後再進行維修。
	Fire: Use care when operating the system in the presence of flammable gases.	火災 : 可燃性のガスが存在する場所でシステムを操作する場合は、充分な注意 を払って下さい。	火災:在有易燃氣體的場地操作該系統時,請務必小心謹慎。
	Eye Hazard: Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	眼に対する危険:化学物質や微粒子が飛散して眼を傷つける危険性があります。化学物質の取り扱い、あるいは計測器の保守・修理に際しては防護眼鏡を着用して下さい。	眼睛傷害危險:飛濺的化學品或顆粒可能造成眼睛傷害。處理化學品或維儀器設備時請佩戴安全眼鏡。
<u>^</u>	General Hazard: A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual.	一般的な危険:この標識は上記以外のタイプの危険が存在することを示します。また、計測器にこの標識がついている場合は、本マニュアル中の指示を参照して下さい。	一般性危險:説明未包括在上述類別中的其他危險。此外,儀器 設備上使用這個標誌,以指示用戶本使用手册中的説明。
	When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific San Jose Products.	安全を確保する手順がよくわからない時は、作業を一時中止し、お近く のサーモエレクトロンサンローゼプロダクトのテクニカールサポートセ ンターごご連絡ください。	如对安全程序有疑问,请在操作之前与当地的菲尼根技术服务中心联系。

Contents

	Preface	xi
	About This Guide	xi
	Safety and Special Notices	xi
	Safety Precautions	xii
	Contacting Us	xiii
Chapter 1	Introduction	1
Chapter 2	Electrospray Ionization	3
	Theory of Electrospray Ionization	4
	Functional Description of the ESI Probe	6
	Removing the ESI Probe	7
	Installing the ESI Probe	9
	Maintaining the ESI Probe	12
	Trimming the ESI Sample Tube	12
	Removing the ESI Nozzle, Needle, and Needle Seal	13
	Cleaning the ESI Manifold	15
	Cleaning the ESI Nozzle	15
	Reassembling the ESI Probe	
	Installing a New Fused-Silica Sample Tube and PEEK Safety Sleeve	16
	Installing an Optional Stainless Steel Needle Sample Tube	18
	Replaceable Parts	19
Chapter 3	Atmospheric Pressure Chemical Ionization	21
•	Theory of Atmospheric Pressure Chemical Ionization	
	Functional Description of the APCI Probe	
	Removing the APCI Probe	
	Installing the APCI Probe	
	Maintaining the APCI Probe	31
	Removing the APCI Nozzle	
	Cleaning the APCI Probe Components	
	Removing the APCI Sample Tube	
	Installing a New APCI Sample Tube	
	Reassembling the APCI Probe	
	Replaceable Parts	35
	-	

Contents

Chapter 4	Ion Max-S Ion Source Housing		
•	Removing the Ion Max-S Ion Source Housing	38	
	Installing the Ion Max-S Ion Source Housing		
	Ion Max-S Housing Maintenance		
Chapter 5	Ion Max Ion Source Housing	43	
	Removing the Ion Max Ion Source Housing	44	
	Installing the Ion Max Ion Source Housing	45	
	Housing Maintenance	47	
	Index	49	

Preface

About This Guide

This manual describes how to install and maintain the ESI and APCI probes.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:



CAUTION Highlights hazards to humans, property, or the environment. Each CAUTION notice is accompanied by an appropriate CAUTION symbol.

IMPORTANT Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or may contain information that is critical for optimal performance of the system.

Note Highlights information of general interest.

Tip Highlights helpful information that can make a task easier.

Safety Precautions

Observe the following safety precautions when you operate the instrument or perform maintenance on the API probes:



CAUTION Do not perform any servicing other than that contained in the instrument-name. To avoid personal injury or damage to the instrument, do not perform any servicing other than that contained in the *Ion Max and Ion Max-S API Source Hardware Manual* or related manuals unless you are qualified to do so.



CAUTION Install a safety sleeve over the ESI capillary. You could receive an electrical shock if the fused-silica capillary tube breaks during ESI operation. Therefore, for your safety and in compliance with international safety standards, you **must** cover the fused-silica capillary tube with the PEEK safety sleeve (P/N 00301-22806) and associated PEEK ferrules (P/N 00101-18119) provided in the Safety Sleeve Kit (P/N 70005-62015) before you operate the instrument.



CAUTION Respect heated zones. Treat heated zones with respect. The ion transfer capillary and the APCI vaporizer might be very hot and might cause severe burns if touched. Allow heated components to cool before you service them.



CAUTION Place the MS detector in Standby (or Off) before you open the atmospheric pressure ionization (API) source. The presence of atmospheric oxygen in the API source when the MS detector is on could be unsafe. The MS detector automatically goes into Standby when you open the API source; however, it is best to take this added precaution.



CAUTION Handle the corona discharge needle with care. The corona discharge needle is very sharp and can puncture your skin.

Contacting Us

There are several ways to contact Thermo Fisher Scientific.

❖ To contact Technical Support

Phone 800-685-9535 Fax 561-688-8736

E-mail TechSupport.C+MS@thermofisher.com

Knowledge base www.thermokb.com

Find software updates and utilities to download at www.mssupport.thermo.com.

❖ To contact Customer Service for ordering information

Phone 800-532-4752 Fax 561-688-8731

Web site www.thermo.com/finnigan

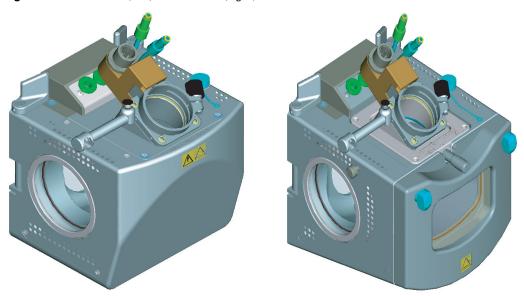
❖ To suggest changes to documentation or to Help

- Fill out a reader survey online at www.thermo.com/lcms-techpubs.
- Send an e-mail message to the Technical Publications Editor at techpubs.finnigan-lcms@thermofisher.com.

Introduction

All mass spectrometers require an ionization source (ion source) to generate ions. The popularity of LC/MS techniques in mass spectrometry has made atmospheric pressure ionization (API) sources the industry standard. The specific process used to ionize the sample is referred to as the ionization mode. The Ion Max™ and Ion Max-S™ API sources can be configured to operate in any of several API modes, including electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI), and atmospheric pressure photo-ionization (APPI). The ion guides transmit the ions produced in the API source into the mass analyzer, where they are separated according to their mass-to-charge ratios.

Figure 1. Ion Max-S (left) and Ion Max (right) API sources



With both of the Ion Max ion source housings you can quickly switch between ionization modes without the need for specialized tools. The ventilation of the ion source housing ensures that the housing is always cool and easy to handle. Pressure in the ion source housing is kept at atmospheric levels, which reduces the chemical noise that nebulized gases can cause when they are not properly evacuated from the ion source. The probe mounting angle is fixed at the optimum angle for signal intensity and ion source robustness. You can adjust the probe position, marking the adjustments to allow for referencing the probe position during ionization optimization. View ports on the ion source housing allow you to view the probe while it is being positioned. These ports also help you add accessories more easily.

1 Introduction

The lifetime of the ion source is excellent due to several special features:

- The drain size and angle prevent ion source corrosion by allowing eluants to flow directly from the probe into the drain when auxiliary gases are off.
- For liquids that do not enter the drain directly, the floor of the ion source interior is specially sloped to enable maximum drainage of collected eluants.
- The zero dead volume LC grounding union that connects the LC flow to the ESI sample inlet is offset from the ion source to prevent LC leaks from dripping directly onto the ion source housing.

Both of the Ion Max API sources incorporate a universal mounting platform and interface for use with ESI, APCI, and APPI ionization sources. For more information on the analysis of ions produced by the Ion Max API sources, refer to the hardware manual that comes with your mass spectrometer.

Electrospray Ionization

This chapter describes the principles of electrospray ionization (ESI), and how to install and maintain the ESI probe for the Ion Max and Ion Max-S API sources. The end of the chapter contains a list of replaceable parts that are available for the maintenance of the probe.

Contents

- Theory of Electrospray Ionization
- Functional Description of the ESI Probe
- Removing the ESI Probe
- Installing the ESI Probe
- Maintaining the ESI Probe
- Installing an Optional Stainless Steel Needle Sample Tube
- Replaceable Parts

Theory of Electrospray Ionization

The electrospray ionization (ESI) mode transforms ions in solution into ions in the gas phase. Many samples that previously were not suitable for mass analysis (for example, heat-labile compounds or high molecular weight compounds) can be analyzed by the use of ESI. ESI can be used to analyze any polar compound that makes a preformed ion in solution. The term *preformed ion* can include adduct ions. For example, polyethylene glycols can be analyzed from a solution containing ammonium acetate because of adduct formation between the NH₄⁺ ions in the solution and oxygen atoms in the polymer. With ESI, the range of molecular weights that the mass spectrometer can analyze is greater than 100000 u, due to multiple charging. ESI is especially useful for the mass analysis of polar compounds, which include biological polymers (for example, proteins, peptides, glycoproteins, and nucleotides), pharmaceuticals and their metabolites, and industrial polymers (for example, polyethylene glycols).

In ESI, ions are produced and analyzed as follows:

- 1. The sample solution enters the ESI needle to which a high voltage is applied.
- 2. The ESI needle sprays the sample solution into a fine mist of droplets that are electrically charged at their surface.
- 3. The electrical charge density at the surface of the droplets increases as solvent evaporates from the droplets.
- 4. The electrical charge density at the surface of the droplets increases to a critical point known as the Rayleigh stability limit. At this critical point, the droplets divide into smaller droplets because the electrostatic repulsion is greater than the surface tension. The process is repeated many times to form very small droplets.
- 5. From the very small, highly charged droplets, sample ions are ejected into the gas phase by electrostatic repulsion.
- 6. The sample ions enter the mass spectrometer and are analyzed.

Figure 2 shows the steps in the formation of ions from highly charged droplets.

¹ Refer to the following papers for more information on the electrospray ionization process: Fenn, J. B.; Mann, M.; Meng, C. K.; Wong, S. F.; Whitehouse, C. M. *Mass Spectrom. Reviews* 1990, *9*, 37; Smith, R. D.; Loo, J. A.; Edmonds, C. G.; Barinaga, C. J.; Udseth, H. R. *Anal. Chem.* 1990, *62*, 882; Ikonomou, M. G.; Blades, A. T.; Kebarle, P. *Anal. Chem.* 1991, *63*, 1989.

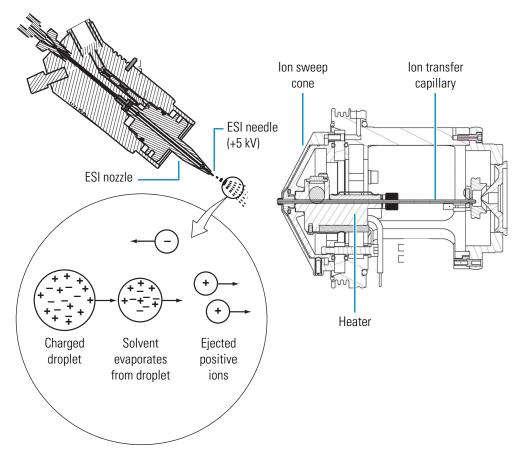


Figure 2. ESI process in the positive ion polarity mode

You can operate the ESI probe in either the positive or negative ion polarity mode. The polarity of the preformed ions in solution determine the ion polarity mode of choice: acidic molecules form negative ions in solution, and basic molecules form positive ions. The ejection of sample ions from droplets is facilitated if the ionic charge and surface charge of the droplet are of the same polarity. Thus, a positively charged needle is used to analyze positive ions and a negatively charged needle is used to analyze negative ions.

Sample ions can carry a single charge or multiple charges. The number of charges carried by the sample ion depends on the structure of the analyte of interest and the carrier solvent. (In ESI, the buffer and the buffer strength both have a noticeable effect on sensitivity, so choosing these variables correctly is important.) In the case of higher molecular weight proteins or peptides, the resulting mass spectrum consists typically of a series of peaks corresponding to a distribution of multiply charged analyte ions.

The ESI process is affected by droplet size, surface charge, liquid surface tension, solvent volatility, and ion solvation strength. Large droplets with high surface tension, low volatility, strong ion solvation, low surface charge, and high conductivity prevent good electrospray.

Organic solvents such as methanol, acetonitrile, and isopropyl alcohol are superior to water for ESI. Volatile acids and bases are good, but salts above 10 mM concentration and strong acids and bases are extremely detrimental.

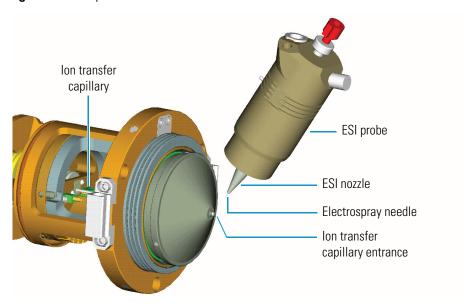
The rules for achieving a good electrospray are as follows:

- Keep salts out of the solvent system.
- Use organic/aqueous solvent systems and volatile acids and bases.
- Optimize the pH of the solvent system.

Functional Description of the ESI Probe

The ESI probe produces charged aerosol droplets that contain sample ions. See Figure 3. The ESI probe accommodates liquid flows of 1 µL/min to 1 mL/min without splitting.

Figure 3. ESI probe and ion source interface

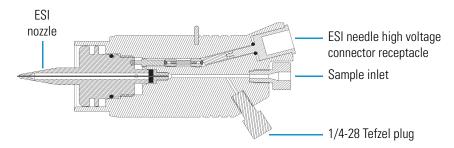


The ESI probe includes the ESI sample tube, needle, nozzle, and manifold. See Figure 4. Sample and solvent enter the ESI probe through the sample tube. The sample tube is a short section of 0.1-mm ID fused-silica tubing that extends from a fitting secured to the ESI source housing, through the ESI probe and into the ESI needle, to within 1 mm from the end of the ESI needle. The ESI needle, to which a large negative or positive voltage is applied (typically ± 3 to ± 5 kV), sprays the sample solution into a fine mist of charged droplets. The ESI nozzle directs the flow of sheath gas and auxiliary gas at the droplets. The ESI manifold houses the ESI nozzle and needle, and includes the sheath gas and auxiliary gas plumbing. The sheath gas plumbing and auxiliary gas plumbing deliver dry nitrogen gas to the nozzle.

The ESI probe has inlets for the introduction of sample solution, sheath gas, and auxiliary gas into the API source. The sheath gas is the inner coaxial nitrogen gas that sprays (nebulizes) the sample solution into a fine mist as it exits the sample tube. Typical sheath gas flow rates for ESI are 10 to 30 units for sample flow rates of less than 10 μ L/min, and 30 to 60 units for sample flow rates greater than 400 μ L/min. When you tune the mass spectrometer, you should adjust the sheath gas flow rate until the ion signal is stable.

The auxiliary gas is the outer coaxial nitrogen gas that assists the sheath gas in the nebulization and evaporation of sample solutions. The auxiliary gas also helps lower the humidity in the ion source. Typical auxiliary gas flow rates for ESI and APCI are 10 to 20 units. Auxiliary gas is usually not needed for sample flow rates below 50 μ L/min.

Figure 4. Cross sectional view of the ESI probe



The angle of the ESI probe is fixed at approximately sixty degrees. You can make small changes to probe orientation with adjustment screws to help optimize spray stability. The fixed angle, off-axis spraying affords long-term signal stability (robustness) for most solutions that contain non-volatile matrix components, mobile phase buffers, or ion-pairing reagents.

Removing the ESI Probe

❖ To remove the ESI probe

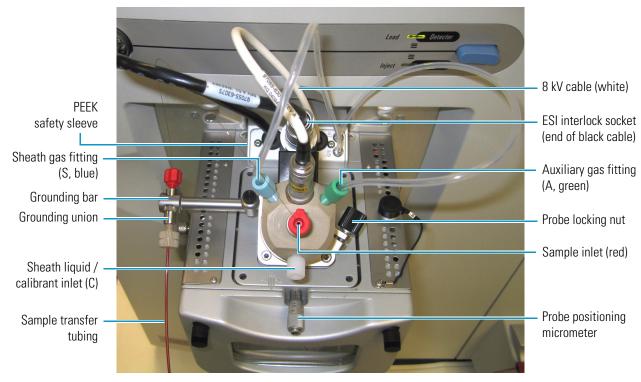
1. Place your mass spectrometer in Standby.



CAUTION If your ESI probe does not already have a sample tube (fused-silica capillary) and safety sleeve attached, you must follow the procedure for installing a sample tube and PEEK safety sleeve that is outlined in the topic "Installing a New Fused-Silica Sample Tube and PEEK Safety Sleeve" on page 16.

- 2. Disconnect the sample transfer tubing from the stainless steel ZDV fitting (grounding union). See Figure 5.
- 3. Remove the 8 kV cable from the ESI needle high voltage receptacle as follows (see Figure 5):
 - a. Unlock the cable by twisting the locking ring counter-clockwise.
 - b. Unplug the 8 kV cable from the ESI needle high voltage receptacle.
- 4. Disconnect the AUX Gas fitting (green) from the auxiliary gas inlet (A) on the probe manifold (Figure 5).
- 5. Disconnect the Sheath Gas fitting (blue) from the sheath gas inlet (S) on the probe manifold.
- 6. Remove the stainless steel fitting (grounding union) from the grounding bar on the ion source housing.

Figure 5. Ion Max ion source housing with ESI probe installed



- 7. Unlock the probe by loosening the probe locking nut.
- 8. Carefully pull the probe straight back in the port in the housing until it meets with the slot in the ESI interlock block.
 - The guide pin on the probe manifold prevents you from twisting the probe until the pin is aligned with the slot in the ESI interlock block.
- 9. Once the probe is all the way back and aligned with the slot, turn the probe 45 degrees counter-clockwise to free the probe from the alignment notch. Be careful not to break the fused-silica sample tube or PEEK safety sleeve.
- 10. Pull the probe straight out to remove it from the ion source housing.
- 11. Store the ESI probe in its original shipping container.

Installing the ESI Probe

❖ To install the ESI probe

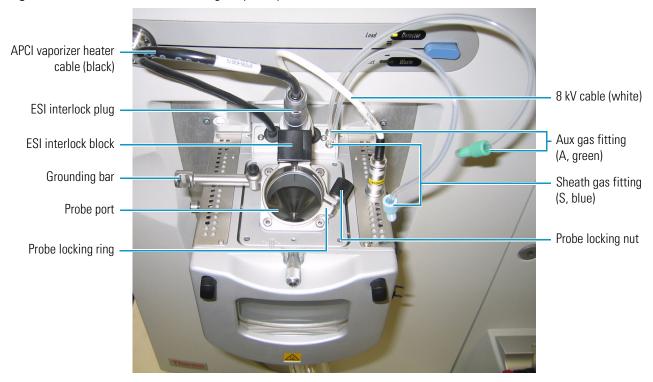
1. Remove the ESI probe from its storage container. Inspect and clean it if necessary.



CAUTION If your ESI probe does not already have a sample tube (fused-silica capillary) and safety sleeve attached, you must follow the procedure for installing a sample tube and PEEK safety sleeve that is outlined in the topic "Installing a New Fused-Silica Sample Tube and PEEK Safety Sleeve" on page 16.

2. To ensure that the probe locking ring is opened wide enough to accept the ESI probe, loosen the probe locking nut. See Figure 6.

Figure 6. Ion Max ion source housing, no probe present

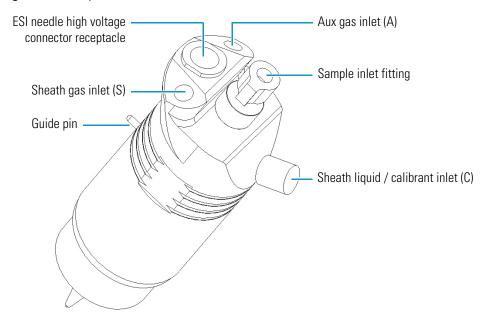


2 Electrospray Ionization

Installing the ESI Probe

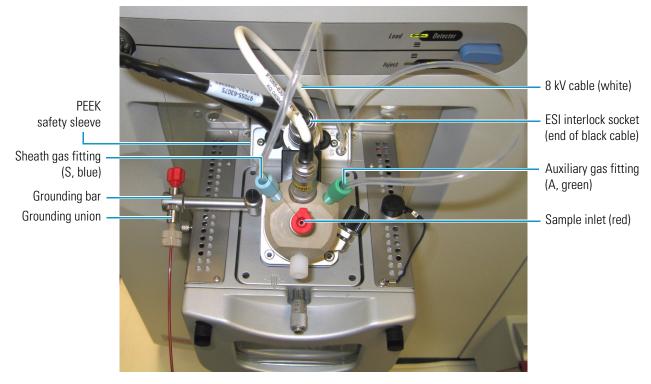
3. Insert the ESI probe into the port in the ion source housing, align the guide pin on the probe body at a minus 45-degree angle from the ESI interlock block. Figure 7shows the location of the guide pin.

Figure 7. ESI probe, side view



- 4. Push the probe into the port until the guide pin meets with the locking ring on the ion source housing.
- 5. Turn the probe 45 degrees clockwise and align the guide pin with the slot in the ESI interlock block. (You might need to pull the probe towards you slightly to properly align the pin with the notch).
- 6. Once you have turned the probe far enough to align the pin with the alignment notch at the rear of the port, push the probe straight in until the guide pin stops at the bottom of the alignment notch.
- 7. Lock the probe in place by tightening the probe locking nut.
- 8. Insert the stainless steel ZDV fitting (grounding union) into the grounding bar on the ion source housing. See Figure 8.

Figure 8. Ion Max ion source housing with ESI probe installed



- 9. Connect the sheath gas fitting (blue) to the sheath gas inlet (S) on the probe manifold. See Figure 8.
- 10. Connect the AUX gas fitting (green) to the auxiliary gas inlet (A) on the probe manifold. Figure 8.
- 11. Insert the APCI vaporizer heater cable into the ESI interlock socket.
- 12. Connect the $8\ kV$ cable to the ESI needle high voltage receptacle on the ESI probe. Tighten the locking ring on the $8\ kV$ connector.
- 13. Connect the sample transfer tubing to the grounding union.

The ESI source is now properly installed on the mass spectrometer.

Note Before you analyze samples with the ESI source, you must change the ionization mode in Tune by choosing **Setup > Change to ESI**.

Maintaining the ESI Probe

The ESI probe requires a minimum of maintenance. If the fused-silica sample tube is plugged or broken, replace it. You can trim or replace the sample tube without disassembling the ESI probe. However, to clean interior surfaces or to replace the electrospray needle or needle seal, you must disassemble the ESI probe.

Note You should flush the ESI probe at the end of each working day by pumping a 50:50 HPLC-grade methanol\distilled water solution from the LC through the ESI probe.

Wear clean gloves when you handle ESI probe components.

To maintain the ESI probe, follow these procedures:

- Trimming the ESI Sample Tube
- Removing the ESI Nozzle, Needle, and Needle Seal
- Cleaning the ESI Manifold
- Cleaning the ESI Nozzle
- Reassembling the ESI Probe
- Installing a New Fused-Silica Sample Tube and PEEK Safety Sleeve

Trimming the ESI Sample Tube

Operating your instrument with acetonitrile in the mobile phase can cause the polyimide coating on the fused-silica sample tube to elongate. If the polyimide coating has elongated past the end of the electrospray needle, you must cut and reposition the end of the sample tube.

To cut and reposition the end of the sample tube 1 mm inside the end of the ESI needle

- 1. Remove the ESI probe from the ESI source by following the procedure described in "Removing the ESI Probe" on page 7.
- 2. Loosen the sample inlet fitting.
- 3. Gently pull back on the sample tube to free it from the fitting.
- 4. Push the sample tube forward so that it extends beyond the end of the electrospray needle.
- 5. Use a fused-silica cutting tool to cut off a small length of sample tube. Ensure that you cut the end of the sample tube squarely.
- 6. Pull the sample tube backwards until the exit end of the sample tube is recessed just inside the ESI needle by approximately 1 mm.

7. Tighten the sample inlet fitting securely to hold the sample tube in place.

Note The sample tube might move forward when you tighten the sample inlet fitting. Ensure that the sample tube is retracted into the ESI needle approximately 1 mm. If necessary, loosen the fitting and reposition the sample tube.

8. Reinstall the ESI probe as described in "Installing the ESI Probe" on page 9.

Removing the ESI Nozzle, Needle, and Needle Seal

Remove the nozzle, needle, and needle seal to clean the ESI manifold. If the needle is damaged, replace it. Replace the needle seal if the sheath gas is leaking at the interface between the needle seal and the needle. See Figure 9.

❖ To remove the ESI nozzle, needle, and needle seal

- 1. Unscrew the sample inlet fitting.
- 2. Remove the sample tube and sample inlet fitting from the ESI probe.
- 3. Use a 5/16-in. wrench to loosen and remove the ESI nozzle from the ESI manifold.
- 4. Remove the ESI needle and needle seal from the ESI manifold. If necessary, after you remove the needle use the needle or another appropriate tool to push the needle seal out of the ESI manifold.
- 5. If necessary, replace the needle seal (P/N 00950-00952), the ESI needle (P/N 00950-00990), or both. See Figure 9 for part names and Figure 14 for part numbers.

Figure 9. Exploded view of the ESI probe and parts (part numbers are given in Figure 14)

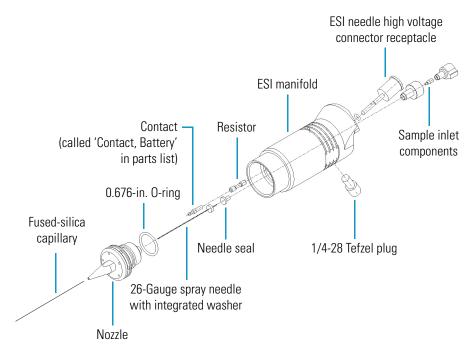
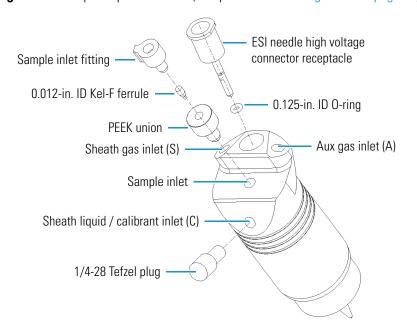


Figure 10. ESI probe part locations (find part numbers in Figure 15 on page 20)



Cleaning the ESI Manifold

To clean and dry the ESI manifold

- 1. If you have not already done so, remove the ESI probe as described in "Removing the ESI Probe" on page 7.
- 2. Unscrew the sample inlet fitting.
- 3. Remove the sample tube and sample inlet fitting from the ESI probe.
- 4. Use a 5/16-in. wrench to remove the ESI spray nozzle from the ESI manifold.
- 5. Remove the spring-loaded probe (P/N 00004-33012) and probe ACC receptacle (P/N 00004-33014) from the ESI manifold (from where it was seated behind the ESI nozzle).
- 6. Remove the high voltage connector receptacle (P/N 00004-89626), resistor assembly (P/N 97055-60058), and battery (P/N 00004-21402) from the ESI manifold. Place these parts on a clean surface.
- 7. Rinse the ESI manifold with distilled water and then with HPLC-grade methanol. Use a Kimwipe™ to remove excess methanol from the ESI manifold.
- 8. Dry the ESI manifold with nitrogen gas.
- 9. Inspect all of the O-rings and replace any that are damaged.
- 10. Replace the compression spring, resistor sleeve, resistor, and high voltage connector in the ESI manifold.
- 11. Replace the probe ACC receptacle and spring-loaded probe in the ESI manifold.

Cleaning the ESI Nozzle

If necessary, clean the bore of the ESI nozzle with an appropriate solvent. The choice of solvent depends on the solubility of the chemical deposits. Rinse the nozzle with methanol and dry with nitrogen gas.

Reassembling the ESI Probe

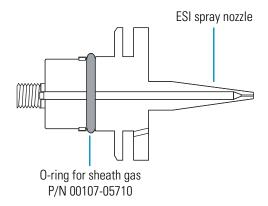
The following are instructions on how to reinstall the ESI sample tube, nozzle, needle, and needle seal (see Figure 9).

To reassemble the ESI probe

- 1. Inspect the Teflon® needle seal (P/N 00950-00952). If the needle seal is deformed, replace it.
- 2. Inspect the 26-gauge spray needle (P/N 00950-00990). If the spray needle is damaged, replace it.

- 3. Ensure that the 0.676-in. ID O-ring (P/N 00107-05710) for the sheath gas on the ESI nozzle is in good condition. In addition, ensure that the O-ring is placed into the precut groove on the ESI nozzle. See Figure 11.
- 4. Reinstall the ESI nozzle, needle, and needle seal as follows:
 - a. Insert the entrance end of the ESI needle into the needle seal.
 - b. Seat the ESI needle and needle seal in the ESI manifold.
 - c. Thread the ESI nozzle over the needle and into the ESI manifold. Slightly wet the nozzle threads with HPLC-grade methanol for lubrication.
 - d. With a 5/16-in. wrench, gently tighten the ESI nozzle until it is a little more than fingertight. Do not overtighten the nozzle.

Figure 11. ESI spray nozzle, showing the O-ring placed in the precut groove



Installing a New Fused-Silica Sample Tube and PEEK Safety Sleeve

❖ To install the new sample tube and PEEK safety sleeve

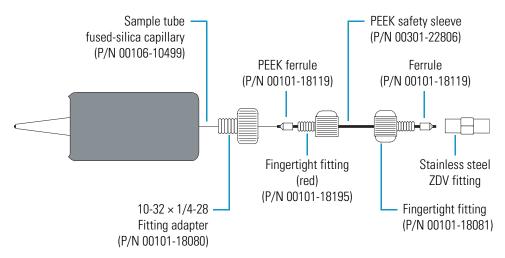


CAUTION AVOID ELECTRICAL SHOCK. When you are operating your instrument in the ESI mode, you could receive an electrical shock unless you install the safety kit discussed below. You could receive an electrical shock if the fused-silica capillary tube breaks during ESI operation. Therefore, for your safety and in compliance with international safety standards, you **must** cover the fused-silica capillary tube with the PEEK safety sleeve (P/N 00301-22806) and associated PEEK ferrules (P/N 00101-18119) provided in the Safety Sleeve Kit (P/N 70005-62015) before you operate the instrument. Installation instructions (P/N 70005-97009) are included in the kit. Operation of the instrument without the safety sleeve impairs the safety protection provided by the instrument and, thus, could lead to serious injury.

- Use a fused-silica cutting tool to cut a 30-cm (12-in.) piece of 0.1-mm ID × 0.19-mm OD fused-silica tubing (sample tube) (P/N 00106-10499). Ensure that you make square cuts to the ends of the fused-silica tubing.
- 2. Insert the sample tube through the exit end of the ESI needle and into the ESI probe.

- 3. Push the sample tube through the ESI probe until approximately 3.5 cm (1.5 in.) of the sample tube protrudes from the exit end of the ESI needle. The remaining length of sample tube should exit the ESI probe sample inlet.
- 4. Slide the $10-32 \times 1/4-28$ Kel-F fitting adapter (P/N 00101-18080) over the sample tube, and tighten the fitting onto the ESI probe sample inlet. See Figure 12.

Figure 12. Fused-silica sample tube and safety sleeve assembly



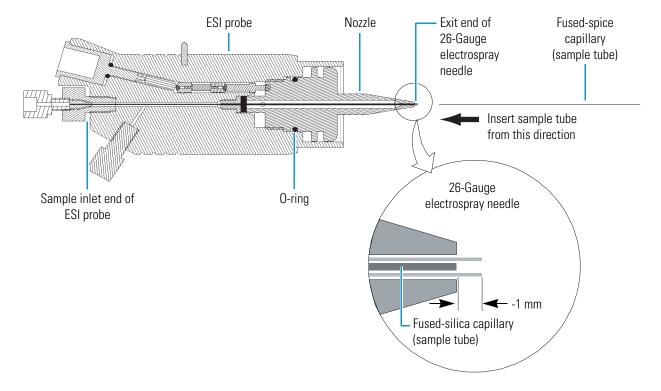
- 5. Slide the precut 25 cm (10 in.) 0.009-in. ID × 0.0240-in. OD PEEK safety sleeve (P/N 00301-22806) over the sample tube.
- 6. Slide the 0.027-in. ID PEEK ferrule (P/N 00101-18119), narrow end first, over the PEEK safety sleeve up to the $10-32 \times 1/4-28$ Kel-F° fitting adapter (P/N00101-18080).
- Slide the (red) fingertight fitting (P/N 00101-18195) onto the PEEK safety sleeve and into the ESI probe sample inlet (labeled **Sample**). Tighten the fitting slightly, but not completely.
- 8. Push the PEEK safety sleeve over the sample tube until it stops against the Teflon needle seal inside the ESI probe.
- 9. Pull the sample tube (from the ESI needle end) until the sample tube is flush with the precut square end of the PEEK safety sleeve.
- 10. Slide a fingertight fitting (P/N 00101-18081) and ferrule (P/N 00101-18119), wide end first, over the PEEK safety sleeve.
- 11. Connect the PEEK safety sleeve and ferrule to the (stainless steel) ZDV fitting by tightening the fingertight fitting. Ensure that the fingertight fitting is securely tightened around the PEEK safety sleeve, otherwise the sample stream might enter between the sample tube and the PEEK safety sleeve. Ensure that the sample tube is held tightly in the grounded fitting by gently pulling the sample tube from the exit end of the ESI needle.
- 12. Use a fused-silica cutting tool to cut the sample tube at the ESI needle so that only 2.5 cm of the sample tube protrudes from the exit end of the ESI needle.

Maintaining the ESI Probe

- 13. From the ESI sample inlet, pull the PEEK safety sleeve backwards so that the exit end of the sample tube is recessed just inside the ESI needle by approximately 1 mm. See Figure 13.
- 14. Tighten the (red) fingertight fitting securely to hold the PEEK safety sleeve and sample tube in place.

Note The sample tube might move forward when you tighten the sample inlet fitting. Ensure that the sample tube is retracted into the ESI needle approximately 1 mm. If necessary, loosen the fitting and reposition the sample tube.

Figure 13. Installing the ESI fused-silica sample tube



Installing an Optional Stainless Steel Needle Sample Tube

You can configure the Ion Max ESI probe to use a stainless steel metal needle rather than a fused-silica sample tube. Two kits are available, one that includes a 32-gauge metal needle (OPTON-53003) for typical flow rates used in ESI and another with a 34-gauge metal needle (OPTON-30004) used for low-flow applications. Both kits include instructions for installing the stainless steel needle sample tube.

Replaceable Parts

Please note that not all parts are available for purchase separately, some parts may only be available for purchase as part of a kit or assembly. Illustrations of these parts are in Figure 14 and Figure 15.

Probe, ESI OPTON-2001	1
Body-probe manifold	0
Nozzle-ESI probe 3-port	6
Fitting, union, 1/4-28, PEEK	4
Contact, Battery, BECU, 0.598 mml, 0.02 ohm@4A	2
Seal, standard needle, 5000 series	2
Needle, D point, 26 gauge, 2-in. length, .24D washer 00950-0099	0
Connector receptacle, high voltage, shielded 00004-8962	6
Ferrule, 0.012-in. ID, KEL-F HPLC	
Fitting, Fingertight 2 Upchurch®	
Fitting, plug, 1/4-28, Tefzel®, HPLC	
O-ring, 0.676-in. ID × 1/16-in. THK, Viton®	
O-ring, 0.125-in. ID × 1/16-in. THK, Viton	
Assembly-resistor contact-ESI probe	
Resistor FXD CC 1/4W 22M 5%, ROHS	
Fitting, HPLC adapter, 10-32 × 1/4-28, KEL-F	
Tubing, fused-silica, 0.10 mm ID × 0.19 mm OD	
Ferrule, 0.008-in. ID, KEL-F HPLC	
Safety Sleeve Kit70005-6201	
Ferrule, 0.27-in. ID, PEEK HPLC	
Tube, 0.009-in. ID \times 0.024-in. OD, 10-in. length, natural PEEK 00301-2280	6
Fitting, Fingertight 2 Upchurch	
Fitting, nut, finger, HPLC, 10-32, PEEK	
Stainless steel needle kit, 32 gauge OPTON-5300	3
Stainless steel needle kit, 34 gauge OPTON-3000	4

Figure 14. Exploded view of the ESI probe

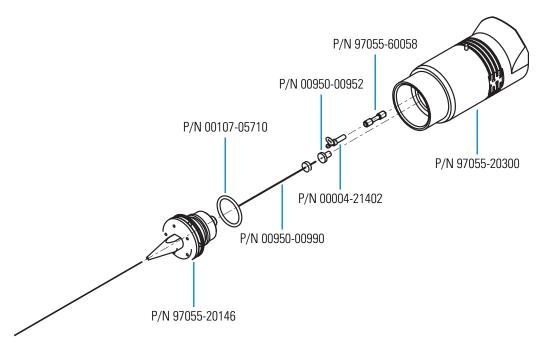
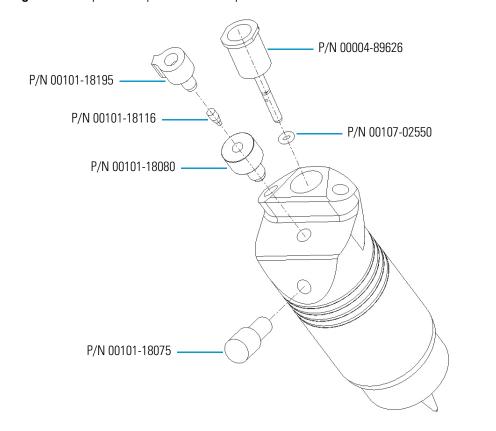


Figure 15. Replaceable parts for the ESI probe



Atmospheric Pressure Chemical Ionization

This chapter describes the principles of atmospheric pressure chemical ionization (APCI), and how to install and maintain the APCI probe for the Ion Max and Ion Max-S API sources. The end of the chapter contains a list of replaceable parts that are available for the maintenance of the probe.

Contents

- Theory of Atmospheric Pressure Chemical Ionization
- Functional Description of the APCI Probe
- Removing the APCI Probe
- Installing the APCI Probe
- Maintaining the APCI Probe
- Replaceable Parts

Theory of Atmospheric Pressure Chemical Ionization

Atmospheric pressure chemical ionization (APCI) is a soft ionization technique, but not as soft as ESI. APCI is used to analyze compounds of medium polarity that have some volatility.

In APCI, ions are produced and analyzed as follows:

- 1. The APCI nozzle sprays the sample solution into a fine mist of droplets.
- 2. The droplets are vaporized in a high temperature tube (the vaporizer).
- 3. A high voltage is applied to a needle located near the exit end of the tube. The high voltage creates a corona discharge that forms reagent ions through a series of chemical reactions with solvent molecules and nitrogen sheath gas.
- 4. The reagent ions react with sample molecules to form sample ions.
- 5. The sample ions enter the mass spectrometer and are analyzed.

Figure 16 shows the APCI process for a positive adduct ion formation.

APCI is a gas phase ionization technique. Therefore, the gas phase acidities and basicities of the analyte and solvent vapor play an important role in the APCI process.

In the positive-ion mode, sample ionization occurs in a series of reactions that start with the electron-initiated cation formation. The following are typical examples of primary, secondary, and adduct ion formation:

• Primary ion formation

$$e^{-} + N_2 \rightarrow N_2^{+} + 2e^{-}$$

• Secondary ion formation

$$N_2^{+} + H_2O \rightarrow N_2 + H_2O^{+}$$

 $H_2O^{+} + H_2O \rightarrow H_3O^{+} + HO^{-}$

• Proton transfer

$$H_3O^+ + M \rightarrow (M + H)^+ + H_2O$$

In negative-ion mode, $(M - H)^{-}$ is typically formed by the abstraction of a proton by OH⁻.

You typically use APCI to analyze small molecules with molecular weights up to about 1500 u. APCI is a very robust ionization technique; minor changes in most variables, such as changes in buffers or buffer strength, do not affect it.

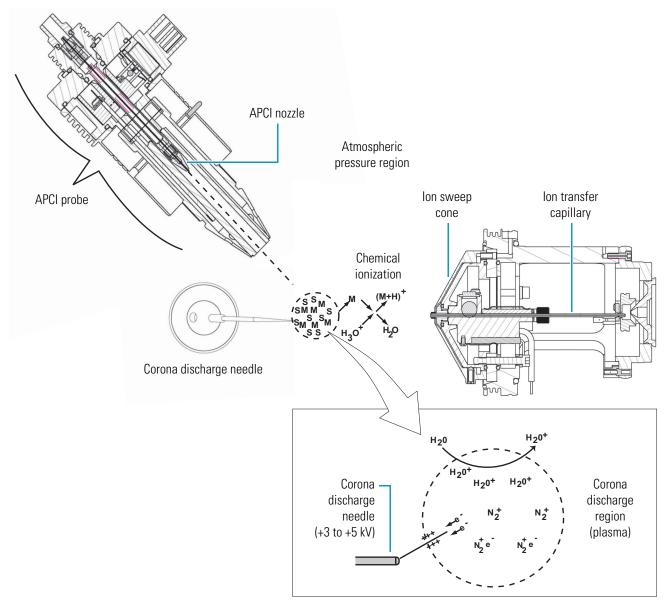


Figure 16. APCI process in the positive ion polarity mode

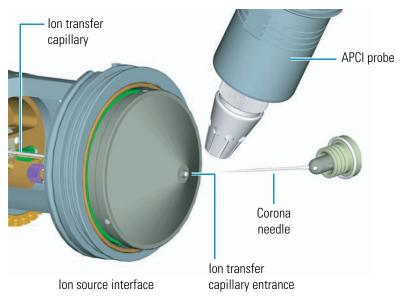
You can use APCI in positive or negative ion polarity mode. For most molecules, the positive-ion mode produces a stronger ion current. This is especially true for molecules with one or more basic nitrogen (or other basic) atoms. An exception to the general rule is that molecules with acidic sites, such as carboxylic acids and acid alcohols, produce more negative ions than positive ions.

Although, in general, fewer negative ions are produced than positive ions, negative ion polarity is sometimes the mode of choice. This is because the negative ion polarity mode sometimes generates less chemical noise than does the positive mode, which might improve selectivity.

Functional Description of the APCI Probe

The APCI probe ionizes the sample by atmospheric pressure chemical ionization. The APCI probe accommodates liquid flows of 100 μ L/min to 2 mL/min without splitting. See Figure 17.

Figure 17. APCI probe, corona discharge needle, and ion source interface



The APCI probe includes the APCI sample tube, nozzle, sheath gas and auxiliary gas plumbing, and vaporizer. See Figure 18.

Figure 18. Cross sectional view of the APCI probe

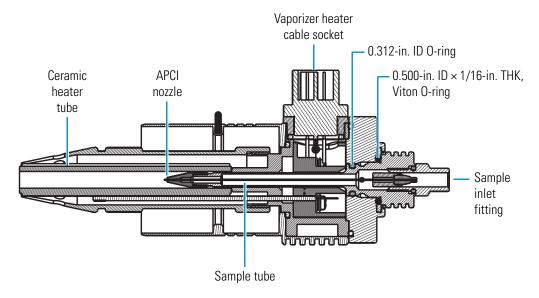
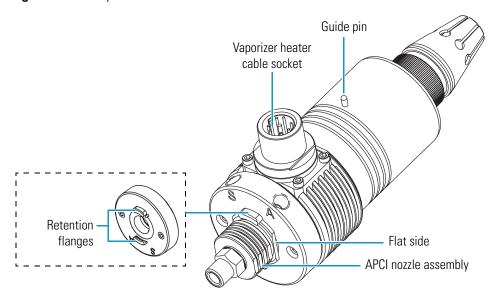


Figure 19. APCI probe exterior



Sample and solvent enter the APCI nozzle through the sample tube. The sample tube is a short section of 0.10-mm ID fused-silica tubing that extends from the sample inlet to 1 mm past the end of the nozzle. The manifold houses the APCI nozzle and includes the sheath gas and auxiliary gas plumbing. The APCI nozzle sprays the sample solution into a fine mist. The sheath gas and auxiliary gas plumbing deliver dry nitrogen gas to the nozzle. The droplets in the mist then enter the vaporizer. The vaporizer flash vaporizes the droplets at temperatures up to 500 °C.

Typical vaporizer temperatures are 350 °C to 450 °C for flow rates of 0.1 to 2 mL/min. The sample vapor is swept toward the corona discharge needle by the flow of the sheath and auxiliary gasses.

The corona discharge needle assembly is mounted inside the Ion Max API source housing. The tip of the corona discharge needle is positioned near the vaporizer. A high potential (typically ± 3 to ± 5 kV) is applied to the corona discharge needle to produce a corona discharge current of up to $100~\mu A$. (A typical value of the corona discharge current is $5~\mu A$.) The corona discharge from the needle produces reagent ion plasma primarily from the solvent vapor. Ion-molecule reactions with the reagent ions in the plasma ionize the sample vapor.

APCI requires a constant source of electrons for the ionization process. Thus, the corona discharge current is set to a specific value and regulated. The potential applied to the corona discharge needle varies, as needed, to provide the required current.

Removing the APCI Probe

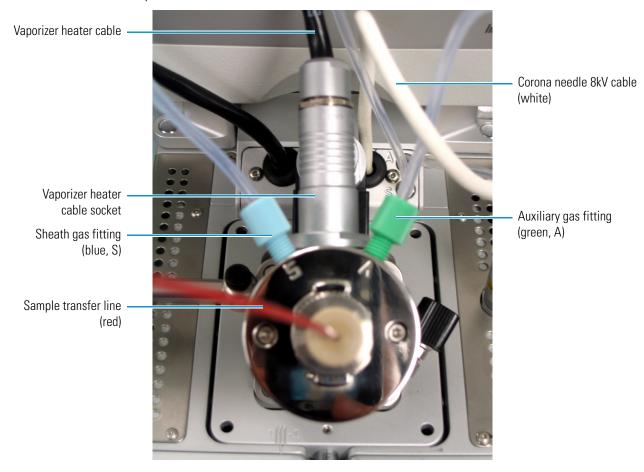
This topic describes how to remove the APCI probe.

Note The following procedure assumes that you are familiar with your instrument and software. If you need additional guidance, refer to the Help, the chapter in this manual that discusses your API source housing (Ion Max or Ion Max-S), or the *Getting Connected Guide* or *Hardware Manual* for your mass spectrometer.

❖ To remove the APCI probe

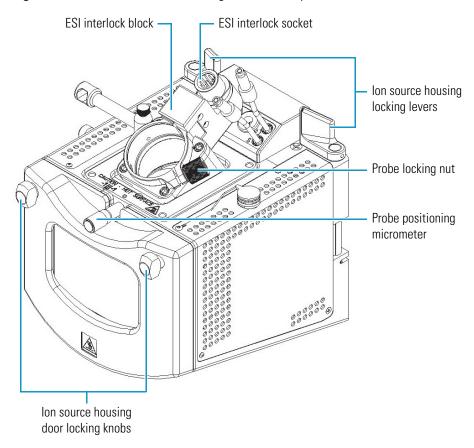
1. Unplug the vaporizer heater cable from the vaporizer heater cable socket on the APCI probe. See Figure 20.

Figure 20. Ion Max with APCI probe installed



2. Connect the vaporizer heater cable to the ESI interlock socket on the ion source housing. See Figure 21.

Figure 21. Ion Max ion source housing, detail of components



- 3. Disconnect the sample transfer line from the APCI probe. See Figure 20.
- 4. Remove the auxiliary gas line (green-colored fitting) from the APCI probe. See Figure 20.
- 5. Remove the sheath gas line (blue-colored fitting) from the APCI probe.



CAUTION AVOID BURNS. At operating temperatures, the APCI vaporizer can severely burn you. The APCI vaporizer typically operates between 400 °C and 600 °C. Always allow the heated vaporizer to cool to room temperature (for approximately 20 min) before you touch or remove this component.

- 6. Remove the APCI probe as follows:
 - a. Loosen the probe locking nut.
 - b. Carefully pull the probe straight back into the port in the housing until it meets with the slot in the ESI interlock block.

The guide pin on the probe manifold prevents you from twisting the probe until the pin is aligned with the slot in the ESI interlock block.

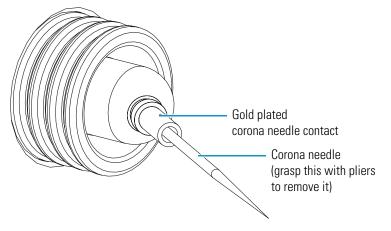
- c. Once the probe is all the way back and aligned with the slot, turn the probe 45 degrees counter-clockwise to free the probe from the alignment notch. Be careful not to break the fused-silica sample tube or PEEK safety sleeve.
- d. Pull the probe straight out to remove it from the ion source housing.
- e. Store the APCI probe in its original shipping container.
- 7. Remove the 8 kV cable from the corona needle high voltage receptacle as follows:
 - a. Unlock the cable by twisting the locking ring counter-clockwise.
 - b. Unplug the 8 kV cable from the corona needle high voltage receptacle.



CAUTION AVOID INJURY. The corona discharge needle is very sharp and can puncture your skin. Handle it with care.

- 8. Remove the corona needle as follows:
 - a. Unlock the ion source housing by turning the ion source locking levers 90 degrees. See Figure 21.
 - b. Remove the ion source housing by pulling the housing straight off of the ion source assembly.
 - The corona needle is in the corona assembly inside of the ion source housing across from the window.
 - c. Using pliers, grasp the needle and pull it straight out of the corona needle contact. See Figure 22.

Figure 22. Corona needle, view from inside the lon Max housing



- d. Remount the ion source housing or place it in a safe location for temporary storage.
- 9. Store the APCI probe in its original shipping container.

Installing the APCI Probe

❖ To install the APCI probe

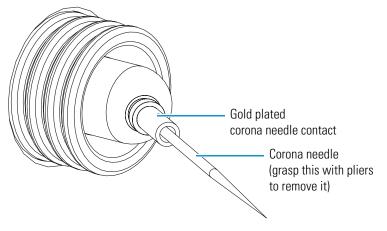
1. Install the corona needle as follows:



CAUTION AVOID INJURY. The corona discharge needle is very sharp and can puncture your skin. Handle it with care.

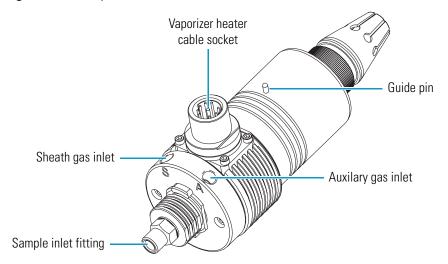
- a. Unlock the ion source housing by turning the ion source locking levers 90 degrees. See Figure 21.
- b. Remove the ion source housing by pulling the housing straight off of the ion source assembly.
- c. Using pliers, grasp the corona needle and push it straight into the corona needle contact. See Figure 23.

Figure 23. Corona needle, view from inside the lon Max housing



- d. Make sure the tip of the needle is aligned with the path of travel between the APCI probe and the ion source interface on the instrument.
- e. Remount the ion source housing.
- 2. Connect the 8 kV cable to the corona needle high voltage receptacle as follows:
 - a. Plug the 8 kV cable into the corona needle high voltage receptacle on the right side of the top of the ion source housing.
 - b. Lock the cable by twisting the locking ring clockwise.
- 3. Be sure to loosen the probe locking nut before attempting to install the probe.
- 4. Insert the APCI probe into the port in the ion source housing, and align the guide pin on the probe body at a 45-degree angle from the ESI interlock block. See Figure 24.

Figure 24. APCI probe



- 5. Push the probe into the port until the guide pin meets with the locking ring on the housing.
- 6. Turn the probe 45 degrees clockwise and align the guide pin with the slot in the ESI interlock block (you may need to pull the probe towards you slightly to properly align the pin with the notch).
- 7. Once you have turned the probe far enough to align the pin with the alignment notch at the rear of the port, push the probe straight in until the guide pin stops at the bottom of the alignment notch.
- 8. Seat the probe all the way down into the alignment notch.
- 9. Lock the probe in place by tightening the probe locking nut.
- 10. Unplug the vaporizer heater cable from the ESI interlock plug on the ion source housing.
- 11. Connect the vaporizer heater cable to the vaporizer heater cable socket on the APCI probe.
- 12. Connect the auxiliary gas line (green-colored fitting) to the inlet on the APCI probe marked **A**.
- 13. Connect the sheath gas line (blue-colored fitting) to the inlet on the APCI probe marked **S**.
- 14. Connect the sample transfer line to the APCI probe inlet.

The APCI source is now properly installed on the mass spectrometer.



CAUTION Prevent solvent waste from backing up into the ion source and mass spectrometer. Always ensure that liquid in the drain tube is able to drain to a waste container and that the outlet of the drain tube is above the level of liquid in the waste container.

Maintaining the APCI Probe

The APCI probe requires a minimum of maintenance. The APCI sample tube ($100-\mu m$ ID fused-silica tubing) is preloaded at the factory. However, if the sample tube becomes obstructed with salt precipitates or is broken, you need to replace it. Also, you might need to remove and clean the APCI nozzle.

Figure 18 and Figure 19 show the major components of the APCI probe.

Note You should flush the APCI probe at the end of each work day by pumping a 50:50 methanol/water solution through the APCI source.

Wear clean gloves when you handle APCI probe components.

To maintain the APCI probe, follow these procedures:

- Removing the APCI Nozzle
- Cleaning the APCI Probe Components
- Removing the APCI Sample Tube
- Installing a New APCI Sample Tube
- Reassembling the APCI Probe

Removing the APCI Nozzle

To remove the APCI nozzle from the APCI probe, do the following (see Figure 18 for the location of parts):

CAUTION



- 1. Until the APCI probe has cooled to room temperature, do not place, wrap, or store it in combustible materials (for example, plastic).
- 2. **AVOID BURNS.** At operating temperatures, the APCI vaporizer can severely burn you. The APCI vaporizer typically operates between 350 and 500 °C. **Always allow** the heated vaporizer to cool to room temperature (for approximately 20 minutes) before you touch or remove these components.

❖ To remove the APCI nozzle and sample tube from the probe

- 1. Place the instrument in Standby mode.
- 2. Hold onto the APCI probe body with one hand and grasp the head of the APCI nozzle assembly. Rotate the head of the nozzle assembly until the flat sides of the head are facing towards the retention flanges (see Figure 19). The nozzle assembly is now free of the probe.
- 3. Carefully pull the nozzle assembly straight out of the APCI probe.



CAUTION Do not break the APCI sample tube. Carefully pull the APCI nozzle straight back from the APCI probe to prevent the sample tube from touching the sides. If the sample tube hits the sides of the vaporizer, it can break.

4. Place the assembly on a clean, lint-free tissue.

Cleaning the APCI Probe Components

❖ To clean the APCI probe components

- 1. Remove the APCI nozzle from the probe body.
- 2. Check the condition O-rings on the APCI nozzle.
- 3. Clean the interior APCI components (excluding the ceramic heater) with a 50:50 solution of HPLC-grade methanol/HPLC-grade water and a lint-free swab. Dry the components with nitrogen gas and place them on a lint-free tissue.
- 4. Reinstall any O-rings you have removed while cleaning.

If you do not want to replace the APCI sample tube, reinstall the APCI nozzle (P/N 97055-60089) as described in the topic "Reassembling the APCI Probe" on page 34.

Removing the APCI Sample Tube

❖ To remove the APCI sample tube from the APCI manifold

- With a 3/8-in. open-end wrench, remove the sample tube inlet fitting (P/N 70005-20250), the 0.239-in. ID O-ring (P/N 00107-04000), and the sample tube from the APCI manifold. See Figure 25.
- 2. Remove the exit-end nut (P/N 70005-20220), the 0.016-in. ID, PEEK ferrule (P/N 00101-18120), and the sample tube from the sample tube inlet fitting.
- 3. Discard the old sample tube.

0.312-in. ID 0-ring
0.500-in. ID × 1/16-in. THK, Viton 0-ring

APCI nozzle

Fused silica sample tube

10-32 PEEK fitting
0.016-in. ID PEEK ferrule

APCI flange fitting

APCI flange fitting

Figure 25. APCI sample tube connection

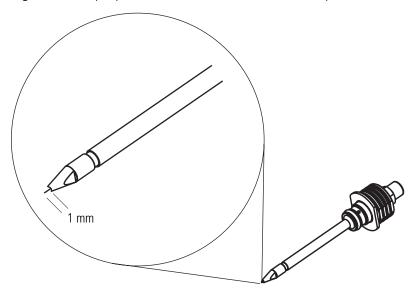
Installing a New APCI Sample Tube

❖ To install a new APCI sample tube

- 1. Use a fused-silica cutting tool to cut a piece of 100 μ m ID, 390 μ m OD fused-silica tubing (P/N 00106-10498) to a length of approximately 15 cm (6 in.). Ensure that you make square cuts to the ends of the fused-silica tubing.
- 2. Slide the PEEK fitting (P/N 70005-20220) and ferrule (P/N 00101-18120) onto the length of the fused-silica tubing. See Figure 25.
- 3. Check the condition of the 0.239-in. ID O-ring (P/N 00107-04000) on the sample tube inlet fitting. Replace it if necessary.
- 4. Insert the fused-silica tubing into the sample tube inlet fitting.
- 5. Slide the PEEK fitting and ferrule down the fused-silica tubing and into the sample tube inlet fitting.
- 6. Tighten the PEEK fitting to secure the new sample tube (fused-silica tubing).

7. Use a fused-silica cutting tool to cut the exit end of the sample tube so that approximately 1 mm protrudes past the tip of the APCI nozzle. See Figure 26.

Figure 26. Proper position of the exit end of the APCI sample tube



Reassembling the APCI Probe

❖ To reassemble the APCI probe

- 1. With one hand holding the APCI probe body to keep the probe from turning, carefully insert the APCI nozzle into the APCI probe.
- 2. With the flat sides of the APCI nozzle head facing up toward the retention flanges on the probe body, seat the nozzle head completely flat against the probe.
- 3. To secure the APCI nozzle in the probe, rotate the head of the nozzle 90 degrees to secure the round sides of the nozzle head in the retention flanges.

To reinstall the probe in the Ion Max API source housing, see "Installing the APCI Probe" on page 29.

Replaceable Parts

APCI probe	OPTON-20012
APCI probe nozzle assembly	97055-60089
Ferrule, 0.016-in. ID PEEK HPLC	00101-18120
Tubing, fused silica 0.15 mm × 0.39 mm	00106-10498
O-ring, 0.239-in. ID × 1/16-in. THK, Viton	00107-04000
O-ring, 0.312-in. ID × 1/16-in. THK	00107-04500
O-ring, 0.500-in. ID × 1/16-in. THK, Viton	00107-05600
Fitting, 10-32 male nut PEEK	70005-20220
Fitting, APCI flange	70005-20250
Nozzle, APCI probe	97055-20221

Figure 27. APCI Probe Assembly (OPTON-20012)

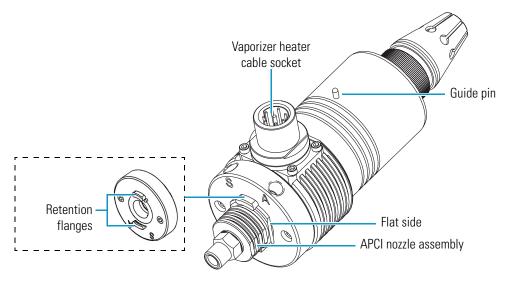
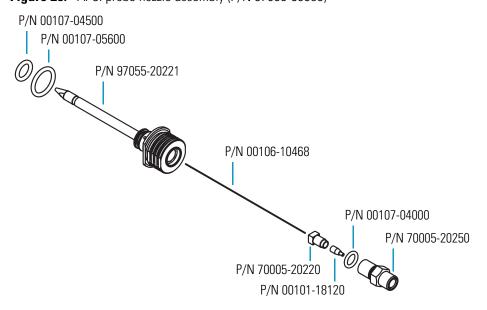


Figure 28. APCI probe nozzle assembly (P/N 97055-60089)



Ion Max-S Ion Source Housing

The Ion Max-S ion source housing seals the atmospheric pressure region of the API source. The ESI, APCI, and APPI probes are interchangeable and mount to the housing using toolless mounts. The Ion Max-S API source housing has two nitrogen inlets, a drain port for waste liquid, and high voltage electrical connections for the electrospray needle (ESI) and for the vaporizer and corona discharge needle (APCI). A high voltage safety interlock switch turns off the following voltages when the Ion Max API-S housing is removed:

- ESI spray voltage (or APCI corona discharge voltage)
- All API source and lens voltages, including the ion transfer capillary offset voltage
- The voltages on the ion guides

The above voltages are also turned off if the APCI vaporizer cable (APCI mode) is not plugged into the APCI vaporizer cable interlock connector on the Ion Max housing.

Contents

- Removing the Ion Max-S Ion Source Housing
- Installing the Ion Max-S Ion Source Housing
- Ion Max-S Housing Maintenance

Removing the Ion Max-S Ion Source Housing

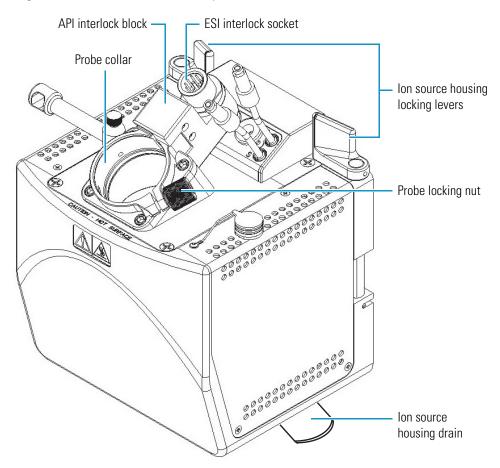
You must remove the Ion Max-S ion source housing to access the ion sweep cone (see Figure 31).

Note If an ion source probe is installed in the ion source housing, the external liquid lines should be disconnected before removing the ion source housing.

To remove the Ion Max-S ion source housing

1. Remove the drain tube from the ion source housing drain.

Figure 29. Ion Max-S, detail of components



- 2. Rotate the ion source housing locking levers 90 degrees to release the ion source housing from the ion source mount assembly.
- 3. Remove the ion source housing by pulling it straight off of the ion source mount assembly. Place the housing in a safe location for temporary storage.

Installing the Ion Max-S Ion Source Housing

❖ To reinstall the Ion Max-S ion source housing

1. Carefully align the two guide pin holes on the rear of the source housing with the ion source housing guide pins on the mass spectrometer. Carefully press the ion source housing onto the ion source mount. See Figure 30 and Figure 31.

Figure 30. Rear view of the lon Max-S ion source housing

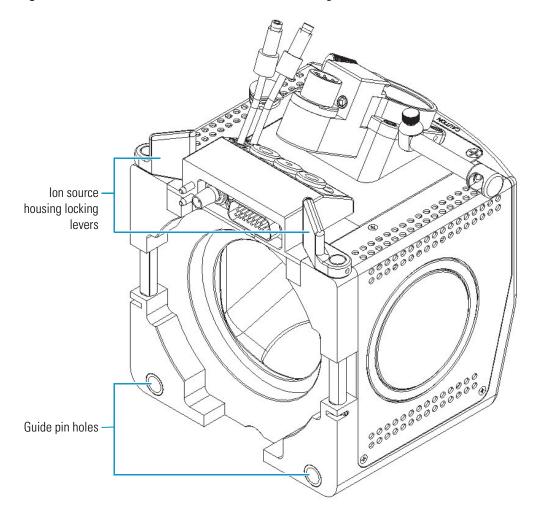
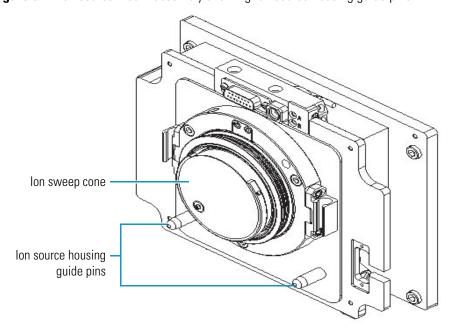


Figure 31. Ion source mount assembly showing ion source housing guide pins



Rotate the ion source housing locking levers 90 degrees to lock the ion source housing onto the ion source mount assembly.



CAUTION Prevent solvent waste from backing up into the ion source and mass spectrometer. Always ensure that liquid in the drain tube is able to drain to a waste container and that the outlet of the drain tube is above the level of liquid in the waste container.

2. Reinstall the source drain tube as follows:



CAUTION Do **not** vent the API source drain tube (or any vent tubing connected to the waste container) to the same fume exhaust system to which you have connected the forepumps. The analyzer optics can become contaminated if the API source drain tube and the (blue) forepump exhaust tubing are connected to the same fume exhaust system.

Your laboratory must be equipped with at least two separate fume exhaust systems. Route the (blue) forepump exhaust tubing to one of the fume exhaust systems. Route the drain tube from the API source to a waste container. Vent the waste container to the other fume exhaust system.

- a. Connect the 1-in. ID Tygon® tubing (P/N 00301-22922) to the ion source housing drain fitting.
- b. Attach the free end of the hose to a waste container. Ideally, the waste container should be vented to a fume exhaust system.

Ion Max-S Housing Maintenance

The Ion Max-S ion source housing is designed to be serviced by trained service engineers only. User maintenance is limited to cleaning the ion source housing as necessary. To clean the housing, remove the housing from the instrument and in an appropriate fume hood, rinse the interior of the housing with methanol and allow it to dry before you install it on the instrument. Be sure to follow all safety precautions in the sections regarding the installation and removal of the housing. For any additional service that might be required, contact your local Thermo Fisher Scientific field service engineer.

Ion Max Ion Source Housing

The Ion Max ion source housing seals the atmospheric pressure region of the API source. The ESI, APCI, and APPI probes are interchangeable and mount to the housing using toolless mounts. The Ion Max API source housing includes a door to access the API probe, a micrometer for precise probe positioning, two nitrogen inlets, a drain port for waste liquid, and high voltage electrical connections for the electrospray needle (ESI) and for the vaporizer and corona discharge needle (APCI). A high voltage safety interlock switch turns off the following voltages when the Ion Max API source door is open:

- ESI spray voltage (or APCI corona discharge voltage)
- All API source and lens voltages, including the ion transfer capillary offset voltage
- The voltages on the ion guides

The above voltages are also turned off if the APCI vaporizer cable (APCI mode) is not plugged into the APCI vaporizer cable interlock connector on the Ion Max housing.

Contents

- Removing the Ion Max Ion Source Housing
- Installing the Ion Max Ion Source Housing
- Housing Maintenance

Removing the Ion Max Ion Source Housing

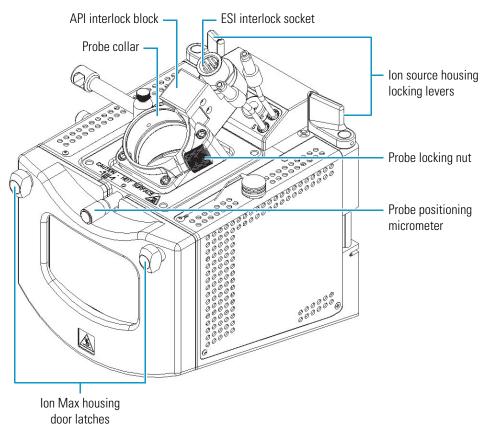
You need to remove the Ion Max ion source housing to access the ion sweep cone (see Figure 34).

Note If an ion source probe is still installed in the ion source housing, Thermo Fisher Scientific recommends disconnecting the external liquid lines first before removing the ion source housing.

❖ To remove the ion source housing

1. Remove the drain tube from the ion source housing drain.

Figure 32. Ion Max, detail of components



- 2. Rotate the ion source housing locking levers 90 degrees to release the ion source housing from the ion source mount assembly.
- 3. Remove the ion source housing by pulling it straight off of the ion source mount assembly. Place the housing in a safe location for temporary storage.

Installing the Ion Max Ion Source Housing

❖ To reinstall the Ion Max ion source housing

1. Carefully align the two guide pin holes on the rear of the source housing with the ion source housing guide pins on the mass spectrometer, and carefully press the ion source housing onto the ion source mount. See Figure 33 and Figure 34.

Figure 33. Rear view of the Ion Max ion source housing

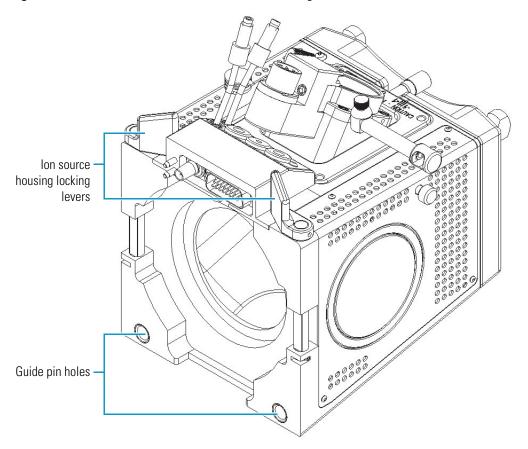
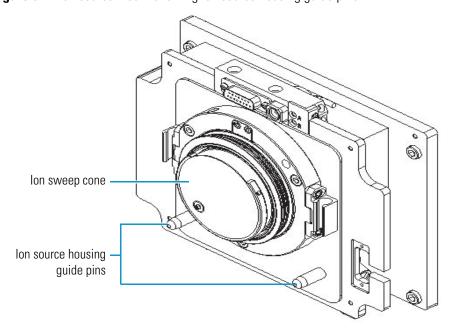


Figure 34. Ion source mount showing ion source housing guide pins



Rotate the ion source housing locking levers 90 degrees to lock the ion source housing onto the ion source mount assembly.



CAUTION Prevent solvent waste from backing up into the ion source and mass spectrometer. Always ensure that liquid in the drain tube is able to drain to a waste container and that the outlet of the drain tube is above the level of liquid in the waste container.

2. Reinstall the source drain tube as follows:



CAUTION Do **not** vent the API source drain tube (or any vent tubing connected to the waste container) to the same fume exhaust system to which you have connected the forepumps. The analyzer optics can become contaminated if the API source drain tube and the (blue) forepump exhaust tubing are connected to the same fume exhaust system.

Your laboratory must be equipped with at least two separate fume exhaust systems. Route the (blue) forepump exhaust tubing to one of the fume exhaust systems. Route the drain tube from the API source to a waste container. Vent the waste container to the other fume exhaust system.

- a. Connect the 1-in. ID Tygon tubing (P/N 00301-22922) to the ion source housing drain fitting.
- b. Attach the free end of the hose to a waste container. Ideally, the waste container should be vented to a fume exhaust system.

Housing Maintenance

The Ion Max ion source housing is designed to be serviced by trained service engineers only. User maintenance is limited to cleaning the ion source housing as necessary. To clean the housing, remove the housing from the instrument. In an appropriate fume hood, rinse the interior of the housing with methanol and allow it to dry before you install it on the instrument. Be sure to follow all safety precautions in the topics regarding the installation and removal of the housing. For any additional service that might be required, contact your local Thermo Fisher Scientific field service engineer.

Index

A	· ·
APCI manifold, location (figure) 24	CAUTIONS
APCI nozzle	avoiding burns from APCI vaporizer 27, 31
reinstalling 32	avoiding burns from ion transfer capillary 27, 31
removing 32	avoiding injury from corona discharge needle xii, 28, 29
APCI probe	Cautions
cleaning 32	solvent waste backing into ion source housing 30, 40, 46
corona discharge needle 25	venting ESI source 40
description 24	cleaning procedures
disassembling 31	APCI probe components 31, 32
figure 24	ESI probe components 12
maintenance 31	corona discharge needle
nozzle 25	description 25
reassembling 34	discharge current 25
removing 31	figure 24
vaporizer 25	corona discharge needle, avoiding injury from xii, 28, 29
vaporizer temperature 25	cross sectional views
APCI sample tube	APCI probe 24
connection (figure) 33	ESI probe 7
installation (figure) 34	•
installing 33	E
removing 32	L
replacing 31	electrospray ionization (ESI)
APCI vaporizer	discussed 4
avoiding burns from (CAUTION) 27, 31	electrospray process (figure) 5
cross sectional view (figure) 24	rules for good electrospray 6
API source	ESI needle
housing for Ion Max 43	location (figure) 14, 20
housing for Ion Max-S 37	reinstalling 15
atmospheric pressure chemical ionization (APCI)	removing 13
APCI process (figure) 23	voltages 6
discussed 22	ESI needle seal
auxiliary gas	location (figure) 14, 20
description 7	reinstalling 15
flow rates, ESI 7	ESI nozzle
auxiliary gas tube, APCI, location (figure) 24	cleaning 15
, 0	location (figure) 14, 20
	O-ring installation (figure) 16
	reinstalling 15
	removing 13

ESI probe	N
auxiliary gas plumbing 6	needle seal, ESI probe
cross sectional view (figure) 7	reinstalling 15
description 6	removing 13
exploded view (figure) 14, 20	-
figure 6	needle, ESI
installing 9	reinstalling 15
maintenance overview 12	removing 13
manifold 6	nozzle
needle 6	APCI, location (figure) 24
nozzle 6	APCI, removing 32
reassembling 15	ESI, reinstalling 15
removing 7	ESI, removing 13
sample tube 6	
sheath gas plumbing 6	S
	J
ESI sample tube, trimming 12	Safety Precautions
	discussion xii
F	MS detector in standby before opening API source xii
Eiguros	no unauthorized servicing xii
Figures	respect heated zones xii
APCI probe assembly 24	sample tube
APCI probe cross sectional view 24	APCI, installing 33
APCI process 23	ESI, installing 16
APCI sample tube connection 33	sample tube, repositioning (note) 13
ESI nozzle O-ring installation 16	
ESI probe 6	sheath gas, description 6
ESI process 5	source housing 37, 43
Ion Max ion source housing 39, 45	
fused-silica tubing	T
APCI, installing 33	
ESI, installing (figure) 18	temperatures range for APCI vaporizer 25
ESI, trimming 12	tuning, sheath gas flow rate 6
H	V
	vaporizer
heater coil, APCI probe, location (figure) 24	APCI probe, description 25
	location (figure) 24
1	_
	temperature 25
ion source	voltages
APCI probe, removing 26	corona discharge needle 25
ESI probe, installing 9	ESI needle 6
ion source interface, figure 6	
ion transfer capillary, avoiding burns from 27, 31	
ionization modes	
atmospheric pressure chemical ionization (APCI) 22	
defined 1	
electrospray ionization (ESI) 4	
М	
maintenance	
APCI probe 31	
ESI probe 12	