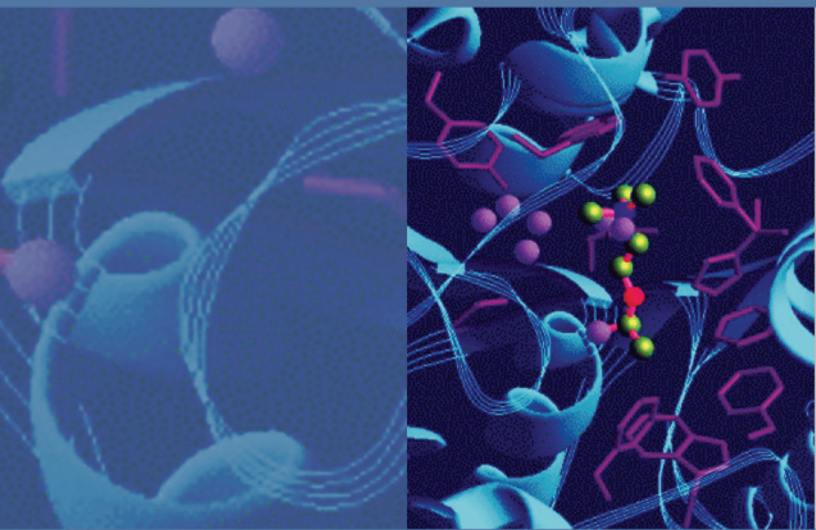

Ion Max and Ion Max-S API Source

Hardware Manual

97055-97044 Revision D November 2008

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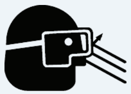
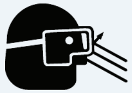
CAUTION Symbol	CAUTION	VORSICHT	ATTENTION	PRECAUCION	AVVERTENZA
	**Electric Shock:** This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not 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.
	**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 VorsichtsmaBnahmen, 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.
	**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 auBerdem 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.

CAUTION Symbol	CAUTION	危険警告	危險警告
	**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。
	**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：說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冊中的說明。
	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。

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Preface

This manual describes how to install the API source housing and how to install and maintain the ESI, HESI-II, 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.

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. For more information, refer to the Regulatory Compliance sheet in the hardware manual provided with your Thermo Scientific mass spectrometer.

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.

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 manual. 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 fused-silica sample tube. 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, the APCI vaporizer, and the HESI-II probe 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 mode (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 mode when you open remove API source housing, open the housing door (Ion Max), or unplug the vaporizer cable from the housing or the APCI or HESI-II probe; 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 for the information you need.

❖ **To contact Technical Support**

Phone	800-532-4752
Fax	561-688-8736
E-mail	us.techsupport.analyze@thermofisher.com
Knowledge base	www.thermokb.com

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

❖ To contact Customer Service for ordering information

Phone	800-532-4752
Fax	561-688-8731
E-mail	us.customer-support.analyze@thermofisher.com
Web site	www.thermo.com/ms

❖ To copy manuals from the Internet

Go to mssupport.thermo.com and click **Customer Manuals** in the left margin of the window.

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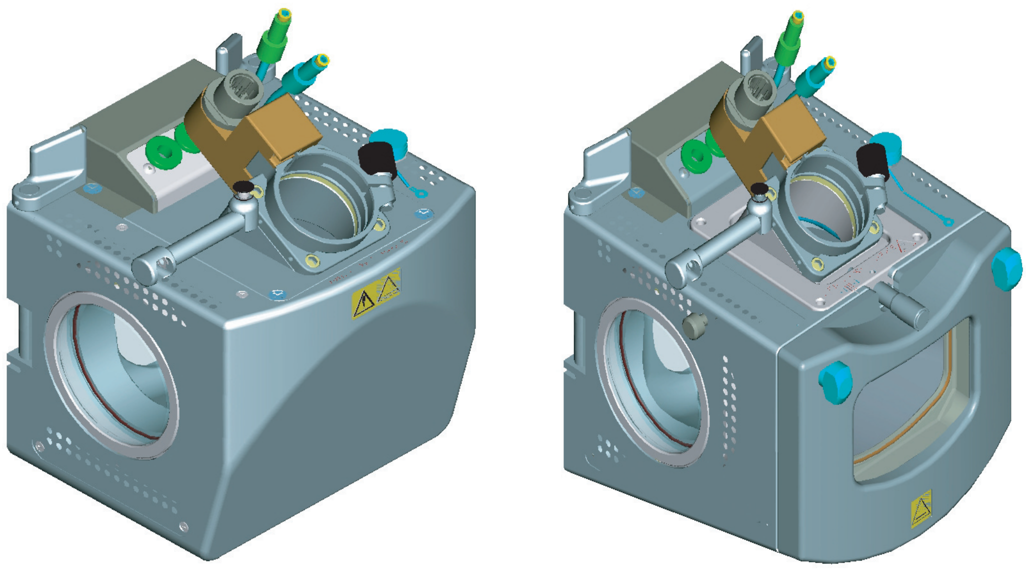
Ion Max and Ion Max-S API Source Housings

This chapter describes the Ion Max and Ion Max-S atmospheric pressure ionization (API) source housings (see Figure 1). The Ion Max and Ion Max-S API source housings provide the same functionality with the following exceptions: the Ion Max has an adjustable probe mount and a front door with a view window. The adjustable probe mount allows you to adjust the XY position of the probe and the front door makes it easier to install additional options. If your system uses the Ion Max-S API source housing, you can only adjust the probe depth.

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



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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), heated-electrospray ionization (H-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.

Table 1 lists which API source housing is provided with your mass spectrometer.

Table 1. API source housings provided with Thermo Scientific mass spectrometers

TSQ Series	LTQ Series	API source housing
TSQ Quantum Access	LCQ Fleet LXQ	Ion Max-S
TSQ Quantum Ultra TSQ Vantage	LTQ and LTQ XL LTQ Velos	Ion Max

With both of the Ion Max API source housings, you can switch between ionization modes without using specialized tools. 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. During operation, the external surface of the ion source housing can become hot. The ventilation holes in the ion source housing aid cooling when the system is placed in Standby mode. Allow the system to cool for a minimum of 20 minutes before touching the ion source housing.

CAUTION **Avoid burns**. Avoid touching the ion source housing when the mass spectrometer is in operation. In the APCI and H-ESI modes, the external surface of the housing can become hot enough to burn your skin.

The probe mounting angle is fixed at the optimum angle for signal intensity and ion source robustness. You can use the probe adjustment markers (A, B, C, and D depth markers) to record the probe position used during ionization optimization. View ports on the ion source housing allow you to view the probe while positioning it. These ports also help you add accessories.

Corrosion of the ion source caused by liquid leaks or poor drainage is prevented by these features:

- The drain size and angle allow 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 (ZDV) grounding unions that connect the solvent flow from the liquid chromatograph or the syringe pump to the ESI or HESI-II probe sample inlets are offset to prevent liquid leaks from dripping into the source housing.

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

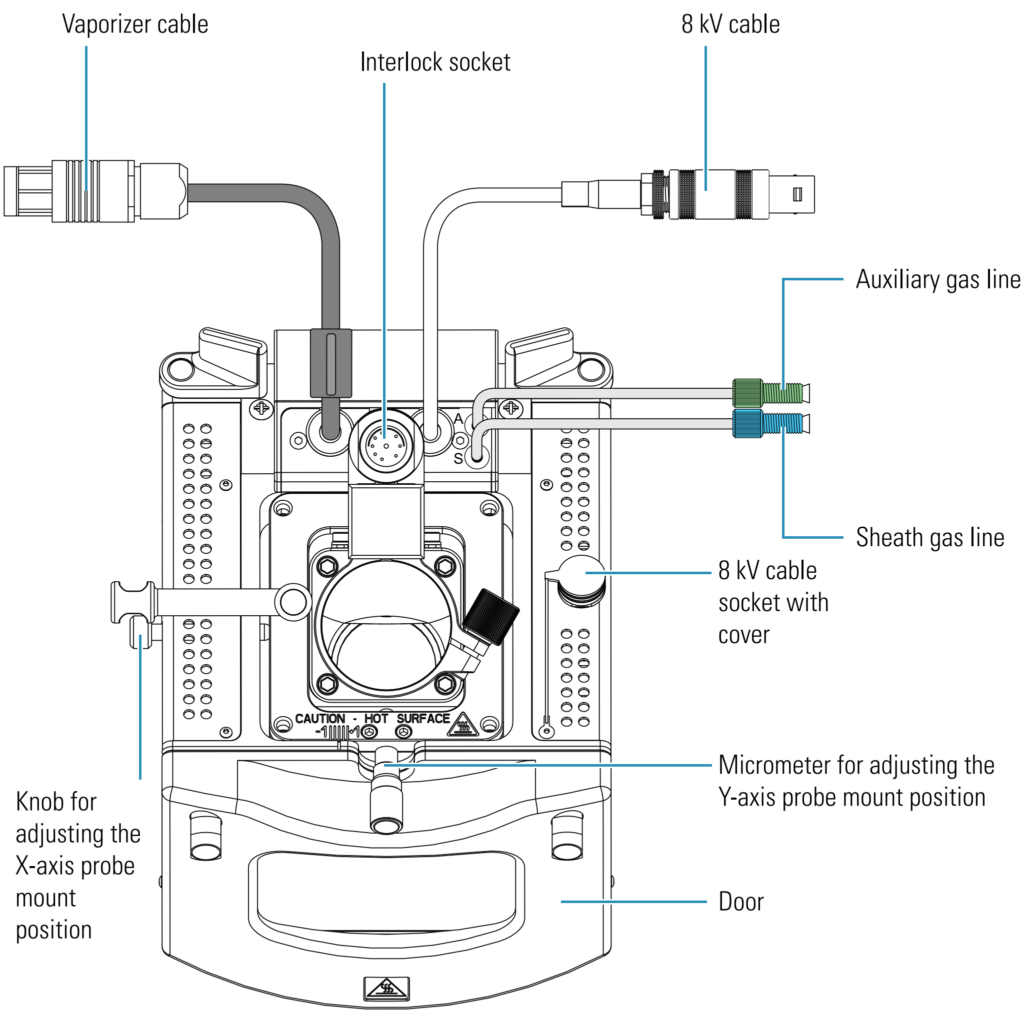
Functional Description of the API Source Housing

The Ion Max and Ion Max-S API source housings include two nitrogen inlets, a drain port for waste liquid, a vaporizer cable for the APCI or HESI-II probe heaters, and an 8 kV cable for the electrospray needle (ESI) or corona discharge needle (APCI).

The Ion Max housing also includes a door to access the API probe and a movable probe port for precise probe positioning. You use the micrometer in front of the probe port to move the probe nozzle closer to or farther away from the ion source interface.

Figure 2 shows the vaporizer cable, 8 kV cable, and nitrogen lines on the top portion of the Ion Max housing. In the H-ESI and APCI modes, the vaporizer cable provides power to the probe heater. In the ESI mode, the vaporizer cable remains connected to the interlock socket. In the ESI and H-ESI modes, the 8 kV cable supplies current to the ESI needle. In the APCI mode, the 8 kV cable supplies current to the APCI corona needle attached to the source housing. For all three ionization modes, the auxiliary and sheath gas lines supply nitrogen flow to the probes.

Figure 2. Ion Max housing viewed from the top



The API source housings have high voltage safety interlock switches that turn off the following voltages:

- ESI needle 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 following actions turn off these voltages:

- Removing the API source housing from the front of the mass spectrometer
- Removing the probe guide pin from the interlock block
- Disconnecting the vaporizer cable from the interlock socket on the API source housing (ESI mode) or from the probe (H-ESI or APCI mode)
- Opening the Ion Max housing door

Adjusting the Probe Position on the Ion Max API Source Housing

If you have the Ion Max API source housing, to maximize sensitivity, you can adjust the side-to-side and front-to-back probe position by a few millimeters. Typically, you adjust the probe position while optimizing the tune parameters for your analytes. For information on using the tune software for your mass spectrometer, refer to the getting started guide for your mass spectrometer.

Note The position of the probe port on the Ion Max-S API source housing is not adjustable.

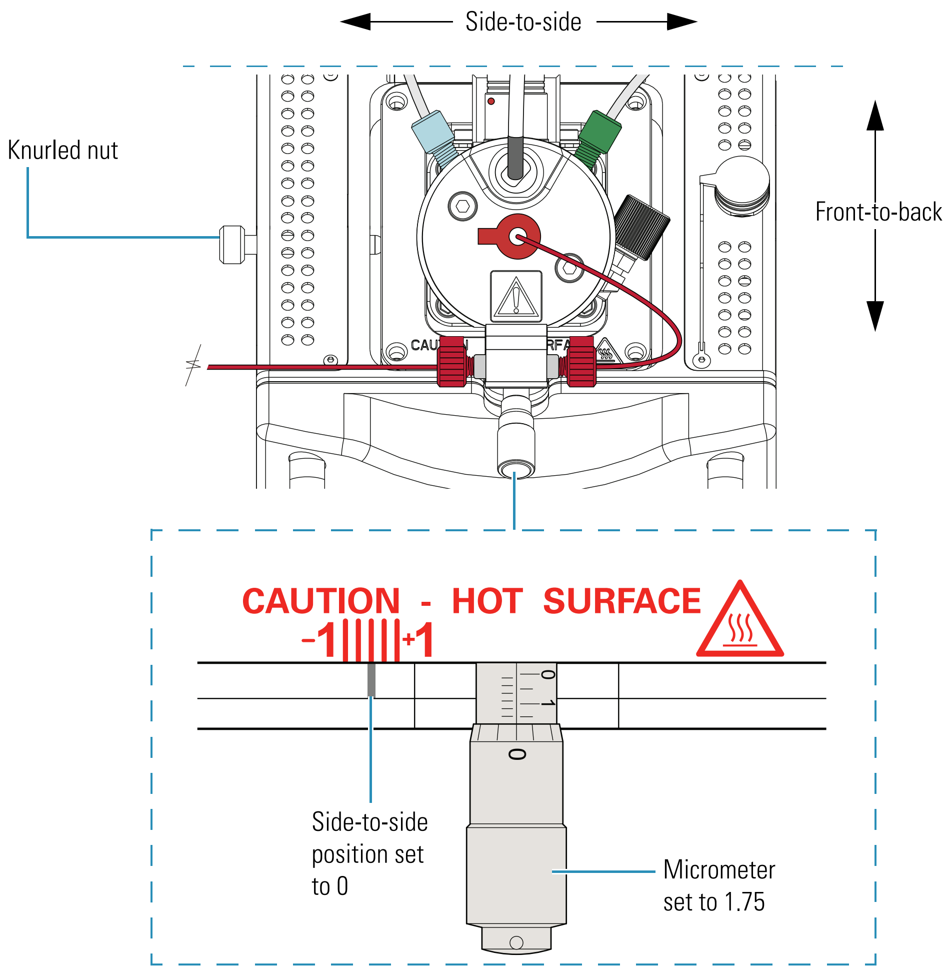
❖ **To adjust the probe position**

- Using the micrometer on the front of the Ion Max source housing, adjust the front-to-back probe position (see Figure 3).

CAUTION **Avoid burns.** When the system is in operation, the surface of the source housing can be hot enough to burn your skin. You can touch the micrometer and the knurled nut while the system is in operation, but avoid touching other parts of the source housing.

- Using the knurled nut on the left side and the +1 to -1 markers on the top front of the Ion Max source housing, adjust the side-to-side probe position (see Figure 3).
- Using the A, B, C, and D markers on the probe as a guide, adjust the probe depth.

Figure 3. Top view of the Ion Max source housing with a HESI-II probe



Removing the API Source Housing

You must remove the API source housing to access the APCI corona needle or the ion source interface.

❖ To remove the API source housing

1. Place the mass spectrometer in Standby mode.

> **Note** When the mass spectrometer is in Standby, the high voltage and nitrogen gas are off.

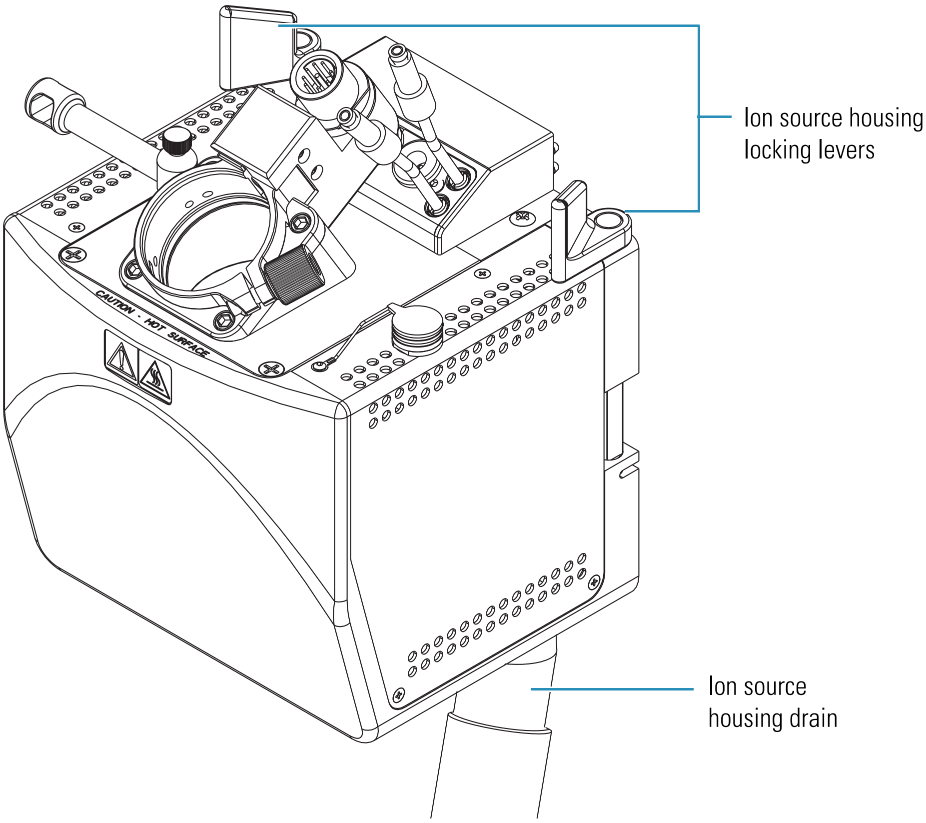
2. If the mass spectrometer was recently in operation, allow time for the ion source housing to cool to room temperature before you touch its external metal surface.

> **CAUTION** **Avoid burns**. During operation, the external surface of the ion source housing can become hot. Allow the ion source housing to cool to room temperature before touching it from the mass spectrometer.

3. If a probe is connected to the source housing, disconnect the external liquid lines before removing the source housing from the mass spectrometer.

4. Remove the drain tube from the ion source housing drain (see Figure 4).

Figure 4. View of the ion source housing locking levers and drain



5. Rotate the ion source housing locking levers 90 degrees to release the ion source housing from the ion source mount assembly (see Figure 4).

6. 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 API Source Housing

❖ **To install the API source housing**

1. Carefully align the two guide pin holes on the rear of the ion source housing (see Figure 5) with the ion source housing guide pins (see Figure 6) on the mass spectrometer. Carefully press the housing onto the ion source mount.

Figure 5. Rear view of the Ion Max-S ion source housing

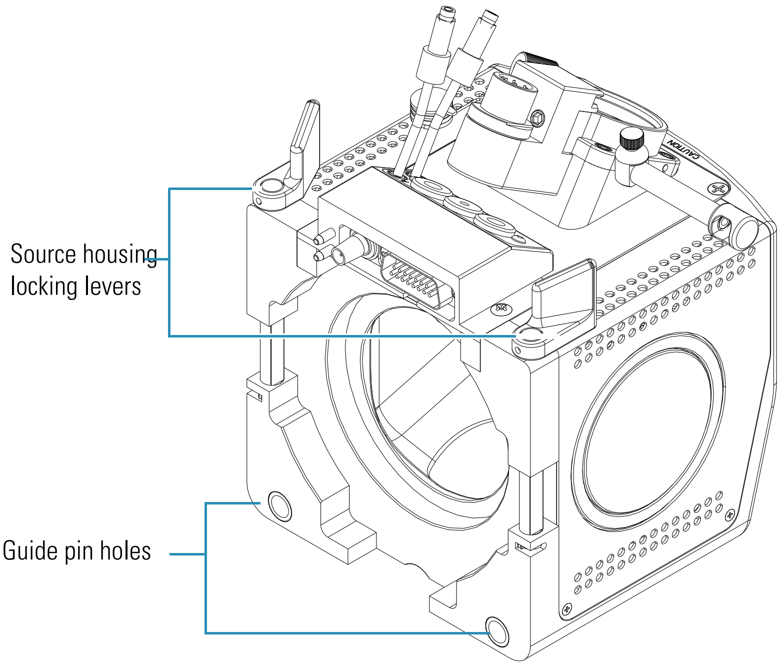
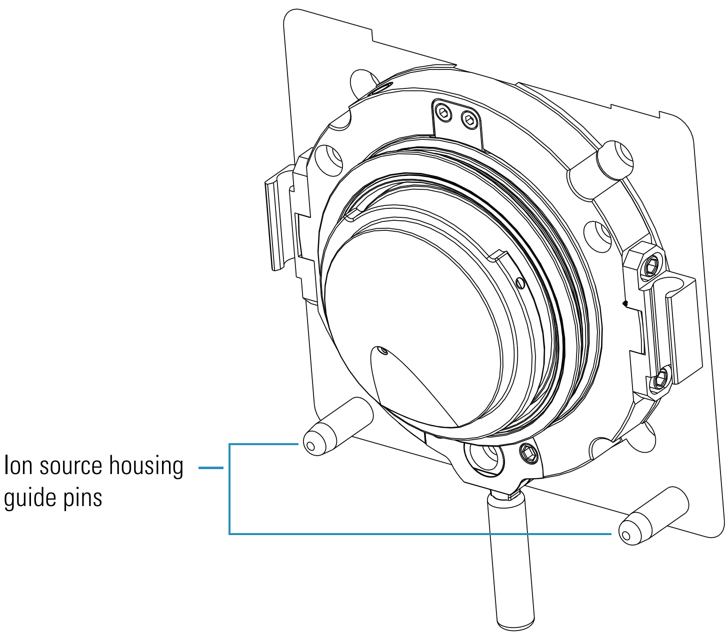


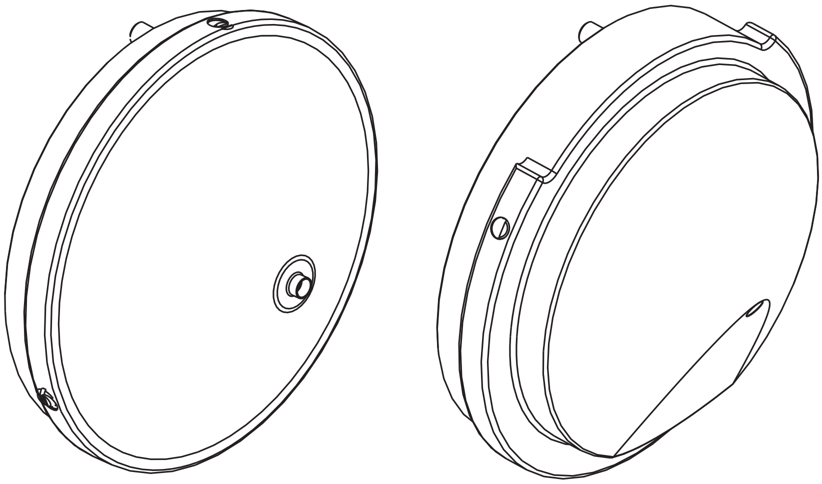
Figure 6. Ion source mount assembly with a view of the guide pins



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

Figure 6 shows the ion sweep cone that has an offset orifice. The ion sweep cone on your ion source mount assembly might look different from the one shown in Figure 6. Figure 7 shows the two types of ion sweep cones available on Thermo Scientific mass spectrometers.

Figure 7. Ion sweep cones



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.

3. Reinstall the source drain assembly as follows:

   a. Connect the source drain assembly to the ion source housing drain fitting (see "API Source Housing Drain" on page 9).

   b. Attach the free end of the hose to a waste container. Ideally, vent the waste container to a fume exhaust system.

API Source Housing Drain

When you install the Ion Max or Ion Max-S API source, you must reconnect the drain at the bottom of the source housing to the solvent waste container (see Figure 8).

Figure 8. Source drain assembly and waste container

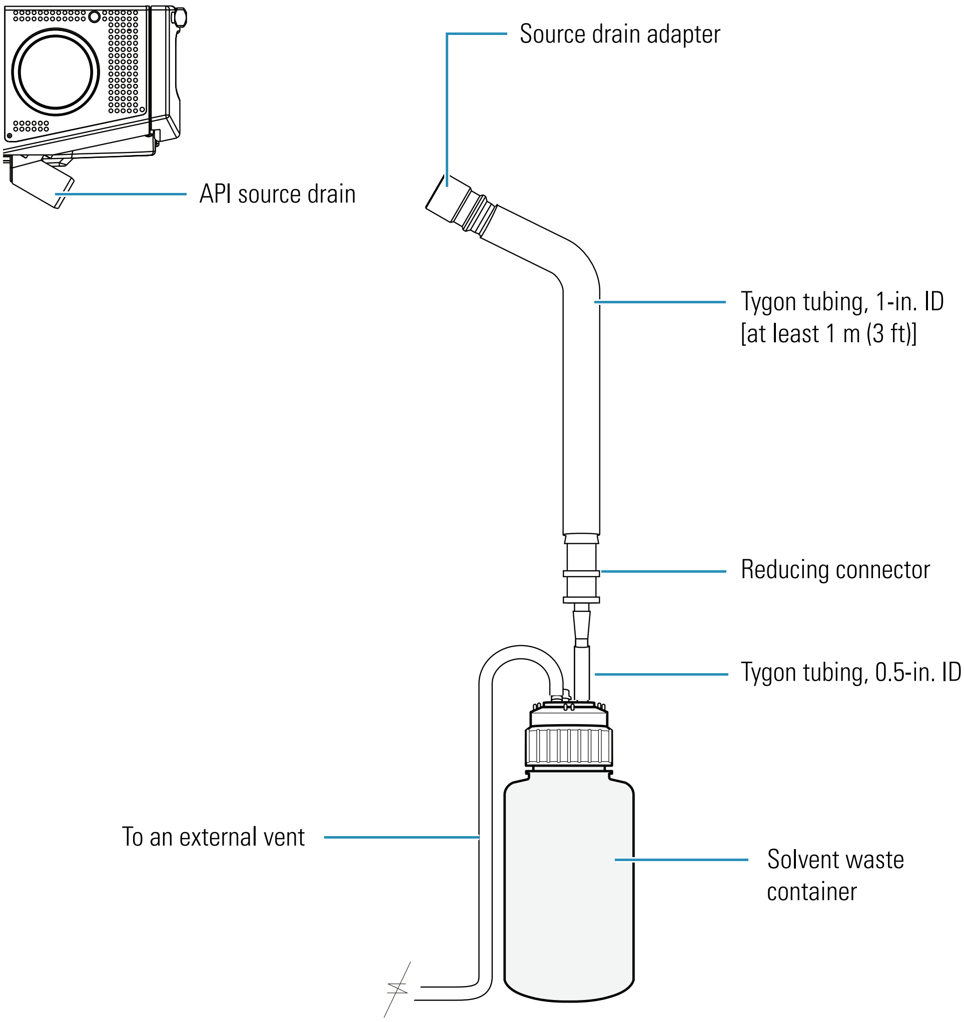


Table 2 lists the components of the solvent waste system. During the initial installation of the mass spectrometer, a Thermo Fisher Scientific field service engineer installs the solvent waste system.

Table 2. Solvent waste system parts

Part description	Part number	Kit location
Source drain adapter, Teflon®	70111-20971	MS Accessory Kit
Reducing connector, single barbed fitting, 1-in.×0.5-in.	00101-03-00001	MS Ship Kit
Tubing, Tygon® PVC, 1-in. ID×1.1875-in. OD	00301-22922	MS Ship Kit
Tubing, Tygon, 0.5-in. ID×0.75-in. OD	00301-22920	MS Ship Kit
Cap, filling/venting	00301-57022	MS Ship Kit
Heavy-duty, 4 L bottle	00301-57020	MS Ship Kit

When you reconnect the drain tubing to the drain at the bottom of the Ion Max or Ion Max-S API source, ensure that you connect the Teflon source drain adapter, which can withstand the high temperatures produced by the H-ESI or APCI source, to the source drain.

IMPORTANT Do **not** connect Tygon tubing directly to the source drain. At high temperatures, Tygon releases volatile contaminates.

CAUTION Prevent solvent waste from backing up into the API source and mass spectrometer. Always ensure that the PVC drain tubing is above the level of liquid in the waste container.

IMPORTANT Your laboratory must be equipped with at least two fume exhaust systems:

The analyzer optics can become contaminated if the API source drain tube and the (blue) exhaust tubing from the forepumps are connected to the same fume exhaust system. Route the (blue) exhaust tubing from the forepumps to a dedicated fume exhaust system.

Do **not** vent the PVC drain tube (or any vent tubing connected to the waste container) to the same fume exhaust system that you have connected the forepumps to. Vent the waste container to a dedicated fume exhaust system.

IMPORTANT Do not connect silicon tubing to the API source outlet drain. If silicone tubing is connected to the outlet drain, you might observe background ions at *m/z* 536, 610, and 684. Use the silicone tubing that is provided with the filling/venting cap to connect the waste container to a fume exhaust system.

2

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

The electrospray ionization (ESI) mode transforms ions in solution into ions in the gas phase.[1] 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 NH_4^+ 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 100 000 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).

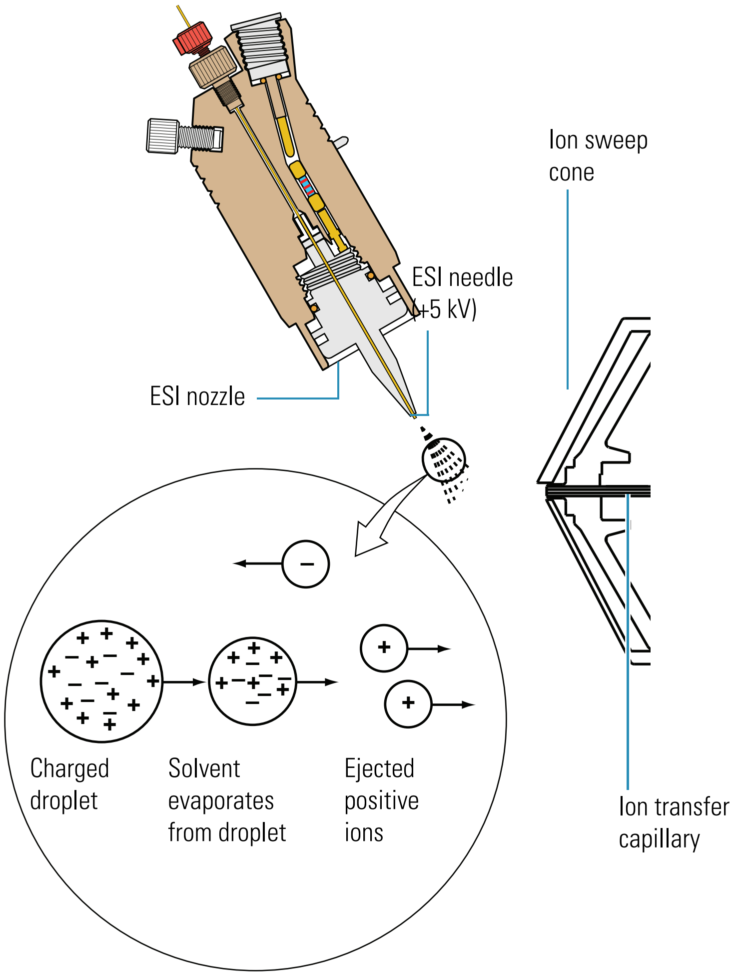
[1] Refer to the following papers for more information on the electrospray ionization process: Fenn, J. B.; Mann, M.; Meng, C. K.; Wong, S. F.; Whitehouse, C. M. *Mass Spectrom. Reviews* 1990, *9*, 37; Smith, R. D.; Loo, J. A.; Edmonds, C. G.; Barinaga, C. J.; Udseth, H. R. *Anal. Chem.* 1990, *62*,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

In ESI, preformed ions in solution are transferred to the gaseous phase and introduced into the mass spectrometer as follows:

4. The sample solution enters the ESI needle to which a high voltage is applied.
5. The ESI needle sprays the sample solution into a fine mist of droplets that are electrically charged at their surface.
6. The electrical charge density at the surface of the droplets increases as solvent evaporates from the droplets.
7. 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.
8. From the very small, highly charged droplets, sample ions are ejected into the gas phase by electrostatic repulsion.
9. The sample ions enter the mass spectrometer and are analyzed.

Figure 9 shows the steps in the formation of ions from highly charged droplets.

Figure 9. 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. Because the ejection of sample ions from droplets is facilitated if the ionic charge and surface charge of the droplet are of the same polarity, use the positive ion polarity mode to analyze positive ions and the negative ion polarity mode 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.

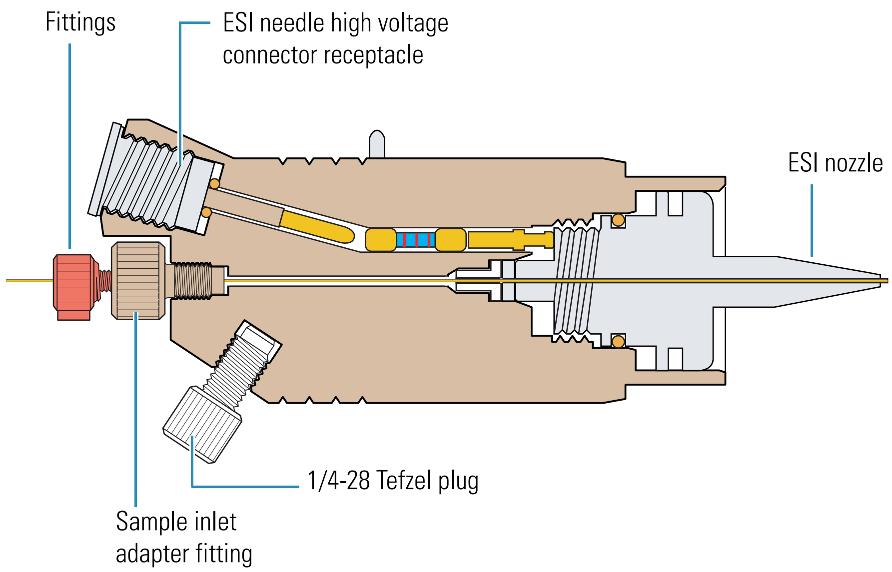
Functional Description of the ESI Probe

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

The ESI probe includes the ESI sample tube, needle, nozzle, and manifold. Sample and solvent enter the ESI probe through the sample tube. The sample tube is a short section of 0.1-mm ID×0.19 mm OD fused-silica tubing that extends from the stainless steel grounding to 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.

Figure 10 shows the solvent path through the ESI probe from the inlet at the back of the probe, through the nozzle at the front of the probe.

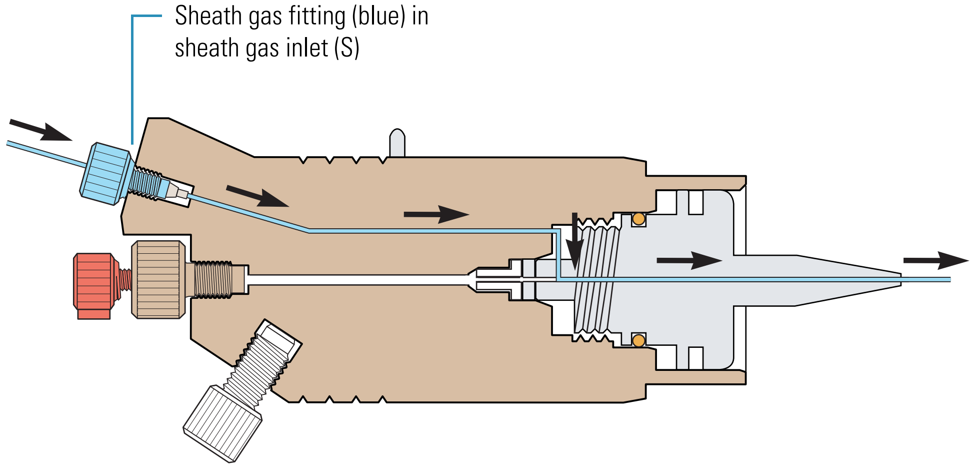
Figure 10. Internal view of the ESI probe



In addition to the sample inlet, the ESI probe has inlets for the introduction of 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 µL/min, and 30 to 60 units for sample flow rates greater than 400 µL/min. When you tune the mass spectrometer, you should adjust the sheath gas flow rate until the ion signal is stable.

Figure 11 shows a cross-sectional view of the sheath gas line. The sheath gas (inner coaxial nitrogen) enters the probe through the sheath gas inlet and exits the probe through the needle port in the front of the nozzle.

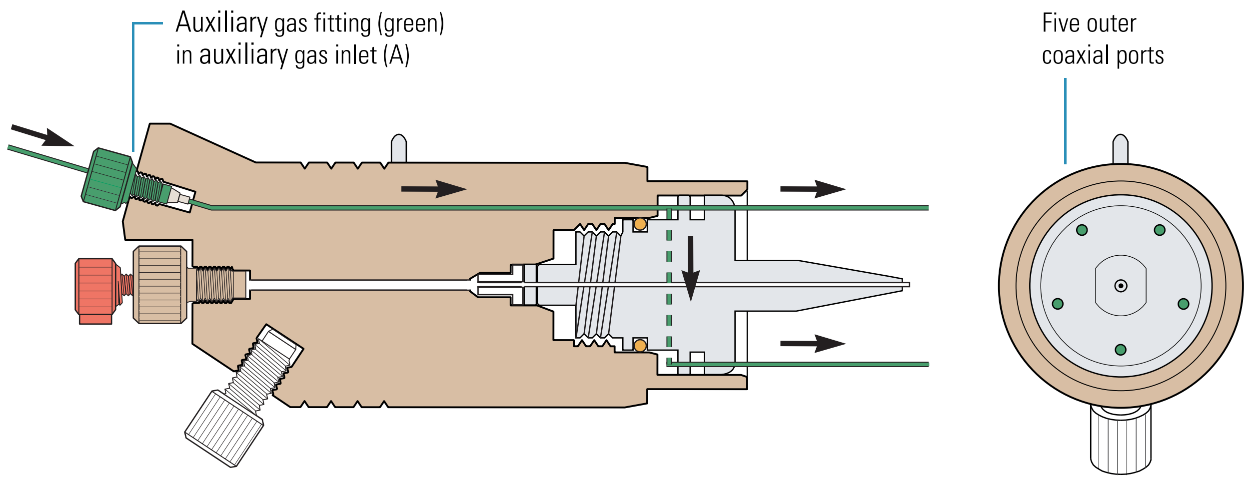
Figure 11. Cross-sectional view of the sheath gas path



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 µL/min.

Figure 12 shows a cross-sectional view of the auxiliary gas line. The auxiliary gas enters the probe through the auxiliary gas inlet and exits the probe through five ports in the nozzle.

Figure 12. Cross-sectional view of the auxiliary gas path



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

Unless you want to replace the fused-silica sample tube, do not disconnect the fused-silica sample tube from the probe sample inlet and the stainless steel grounding union when you remove the probe from the API source housing.

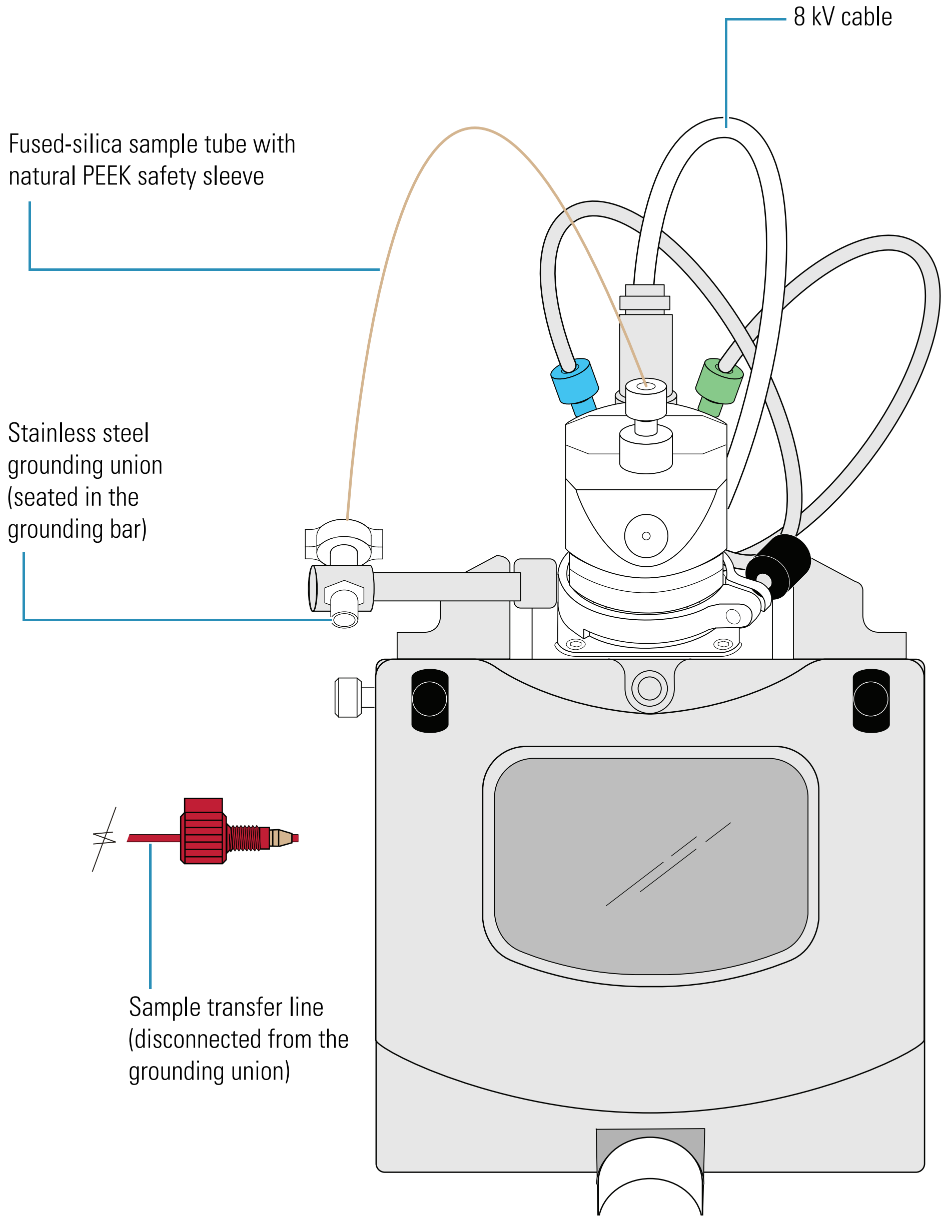
❖ **To remove the ESI probe from the API source housing**

1. Place the mass spectrometer in Standby mode.

> **Note** When the mass spectrometer is in Standby, the high voltage and nitrogen gas are off.

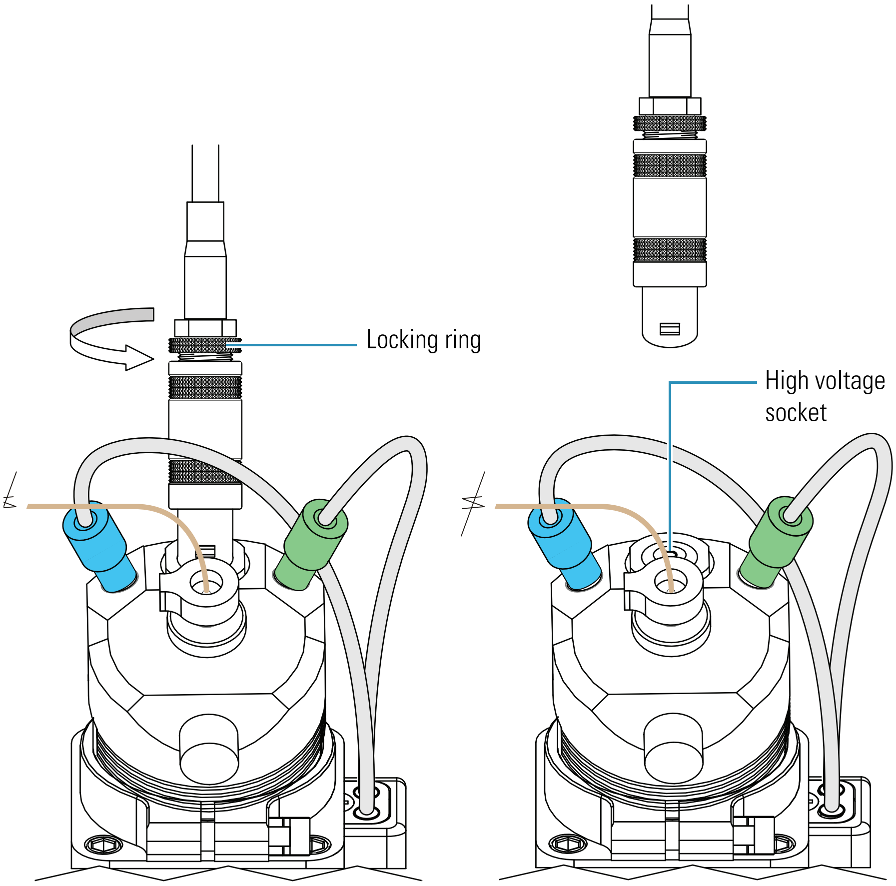
2. Disconnect the sample transfer line from the stainless steel grounding union. The grounding union is seated in the grounding bar (see Figure 13).

Figure 13. View of the sample transfer line disconnected from the grounding union



3. Remove the 8 kV cable from the ESI probe as follows (see Figure 14):
   a. Unlock the cable connector by twisting the locking ring counterclockwise.
   b. Unplug the 8 kV cable connector from the probe high voltage socket.

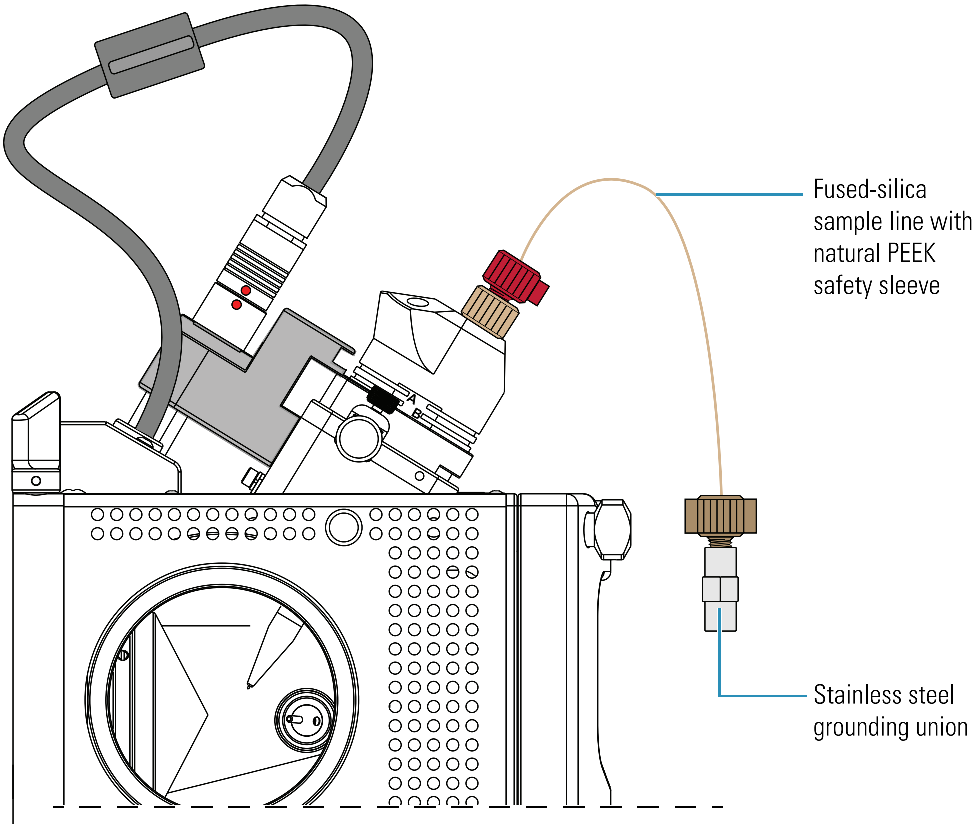
Figure 14. Removing the 8 kV cable connector from the ESI probe



4. Disconnect the nitrogen lines:
   - Disconnect the fitting (green) from the auxiliary gas inlet (A) on the probe manifold.
   - Disconnect the fitting (blue) from the sheath gas inlet (S) on the probe manifold.
5. Remove the stainless steel grounding union from the grounding bar.

   In Figure 15, the nitrogen lines and the 8 kV cable have been disconnected from the ESI probe. In addition, the stainless steel grounding union has been removed from the grounding union bar.

Figure 15. Left-side view of the API source housing



6. Unlock the probe locking ring by turning the probe locking knob counterclockwise.
7. To remove the probe from the port in the API source housing:
   a. Slowly pull the probe out of the port until you feel the resistance caused by the probe guide pin meeting the interlock block.
   b. Turn the probe counterclockwise until the guide pin is free of the interlock block.
   c. When the guide pin is free of the interlock block, pull the probe out of the port.
8. Store the ESI probe in its original shipping container.

Installing the ESI Probe

❖ **To install the ESI probe**

1. Place the mass spectrometer in Standby mode.

> **Note** When the mass spectrometer is in Standby, the high voltage and nitrogen gas are off.

2. If the MS detector is set up for the APCI mode, remove the APCI probe and the corona needle (see “Removing the APCI Probe and the Corona Needle” on page 62).

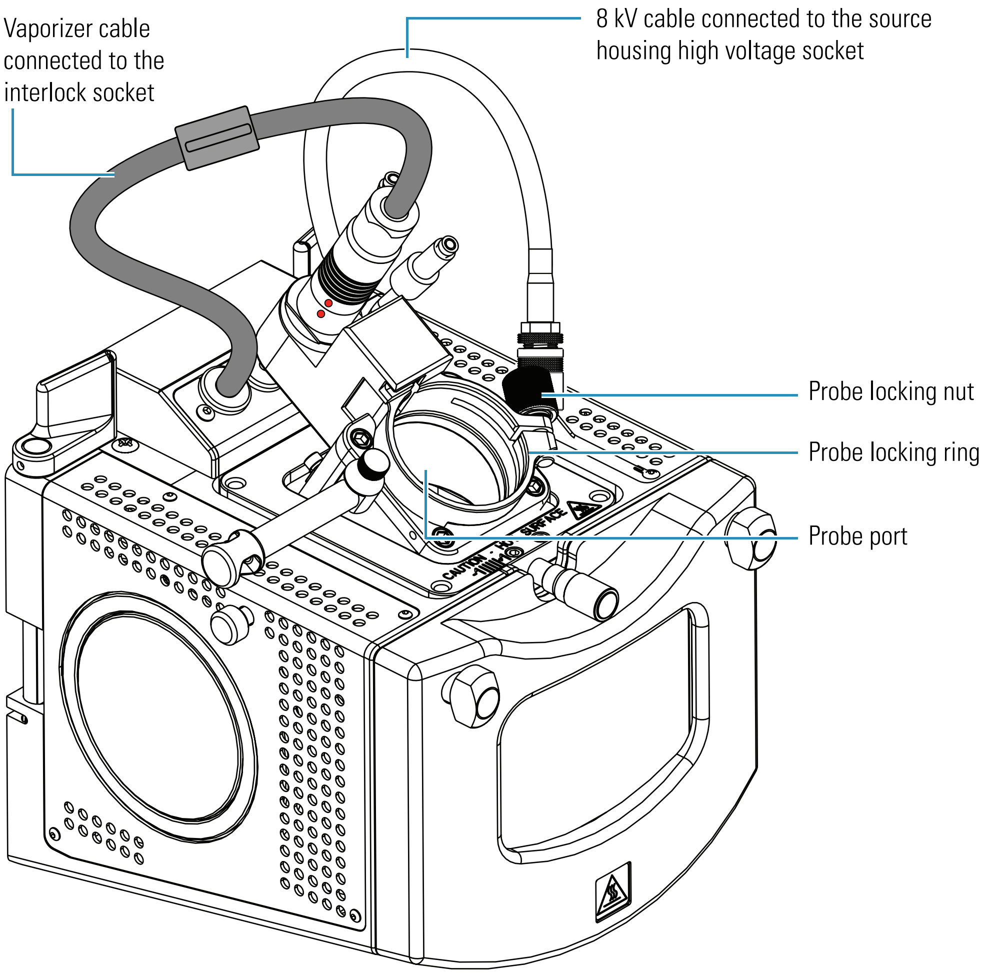
3. Remove the ESI probe from its storage container. Inspect and clean it if necessary.

4. If the ESI probe does not already have a sample tube (fused-silica capillary) and safety sleeve or a metal needle sample tube attached, see “Installing a Fused-Silica Sample Tube and PEEK Safety Sleeve” on page 28 or refer to the *Installation Guide for the 32-Gauge Metal Needle*, respectively.

   The installation guide for the metal needle (P/N 70001-97112) is provided in the Metal Needle Kit. You can also download the installation guide from the Thermo Fisher Scientific customer support Web site (see “Contacting Us” on page x).

5. Turn the probe locking knob counterclockwise until the probe locking ring is opened to its widest position (see Figure 16).

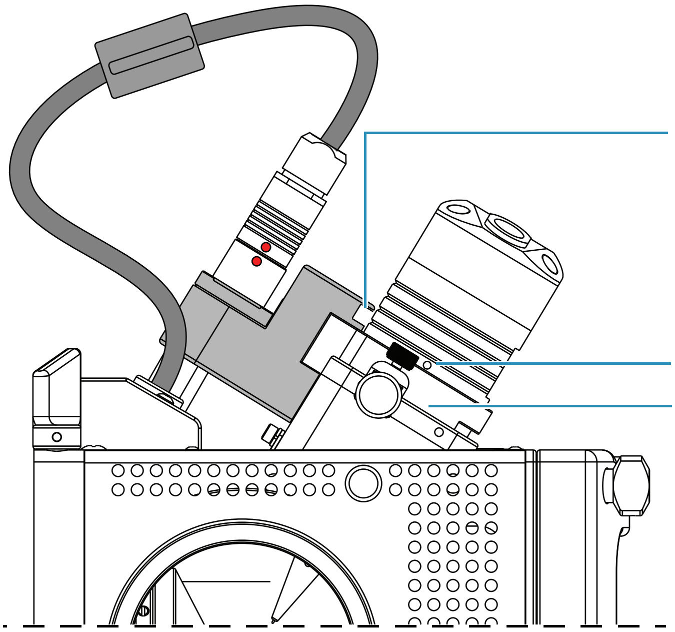
Figure 16. Top-left view of the Ion Max source housing



6. To seat the probe in the source housing probe port:

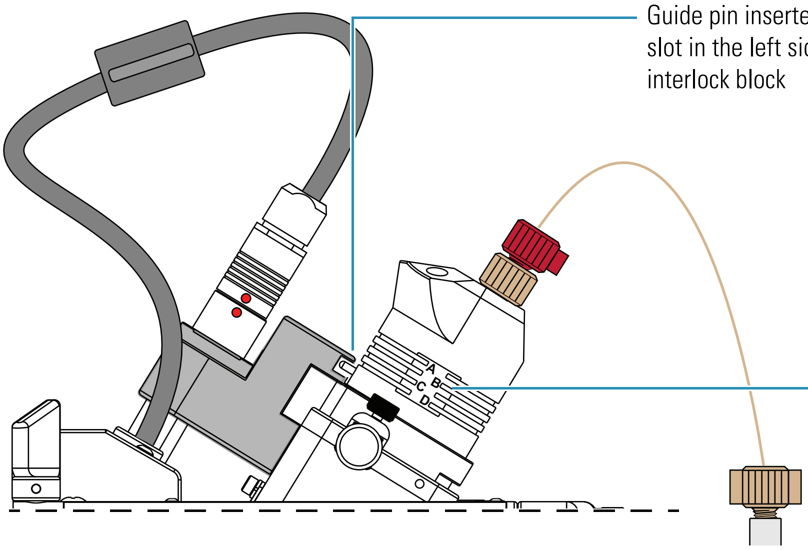
   a. Holding the probe with the nozzle facing downwards and the guide pin facing towards the left, slowly insert the probe into the port until the guide pin meets the locking ring on the API source housing (see Figure 17).

Figure 17. View of the ESI probe guide pin touching the locking ring



   b. Pull the probe slightly upward until the guide pin is level with the slot on the left side of the interlock block. Then turn the probe clockwise until the guide pin meets resistance from the interlock block (see Figure 18).

Figure 18. View of the probe guide pin inserted into the interlock block slot



   c. Push the probe further downward into the port to the appropriate depth indicated by the A, B, C, and D depth markers on the probe.

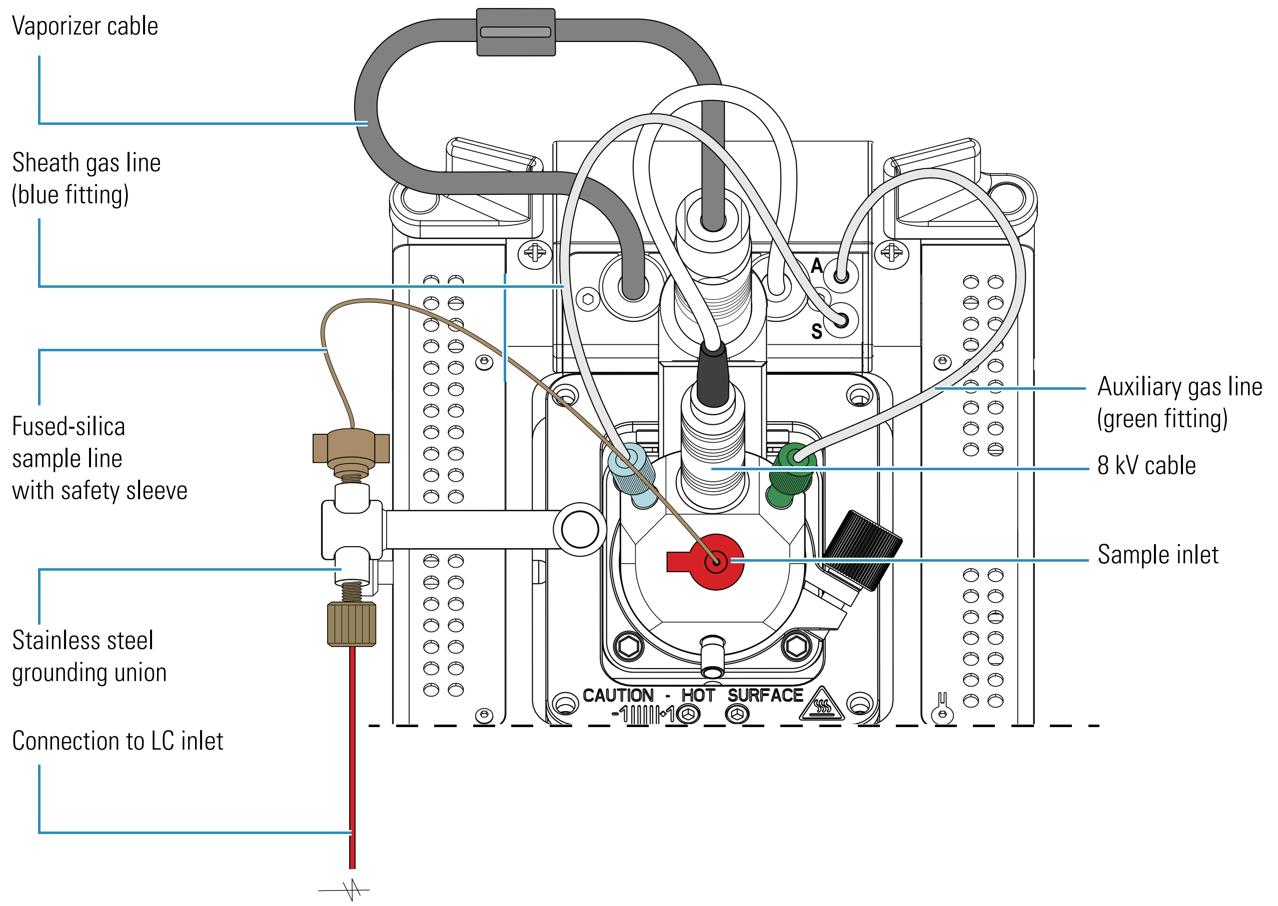
7. Lock the probe in place by turning the probe locking knob clockwise until you feel resistance.

8. Connect the nitrogen gas lines to the ESI probe as follows:
   a. Connect the sheath gas fitting (blue) to the sheath gas inlet (S).
   b. Connect the auxiliary gas fitting (green) to the auxiliary gas inlet (A).
9. Connect the 8 kV cable connector to the 8 kV cable socket on the probe. Tighten the locking ring on the 8 kV cable connector (see Figure 14 on page 17).
10. Ensure that the stainless steel grounding union is seated in the grounding union bar.
11. Using two fingertight fittings, connect a length of red PEEK tubing to the LC outlet and to the left side of the grounding union.

Figure 19 shows the ESI probe installed in the Ion Max API source housing.

> **Note** The LTQ and TSQ Series mass spectrometers can auto-sense the ESI probe. For the LCQ Advantage MAX and the Deca XP MAX, you must change the ionization mode in the tune application by choosing **Setup > Change to ESI**.

Figure 19. ESI probe connections



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 the nozzle bore or the interior surfaces of the manifold or to replace the electrospray needle or needle seal, you must disassemble the ESI probe.

To minimize cleaning of the probe components, flush the ESI probe at the end of each working day by pumping a 50:50 HPLC-grade methanol/distilled water solution through the ESI probe.

IMPORTANT Wear clean gloves when you handle ESI probe components.

To maintain the ESI probe, follow these procedures:

- Trimming the ESI Sample Tube
- Disassembling the ESI Probe
- Cleaning or Replacing the ESI Probe Components
- Assembling the ESI Probe

Trimming the ESI Sample Tube

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

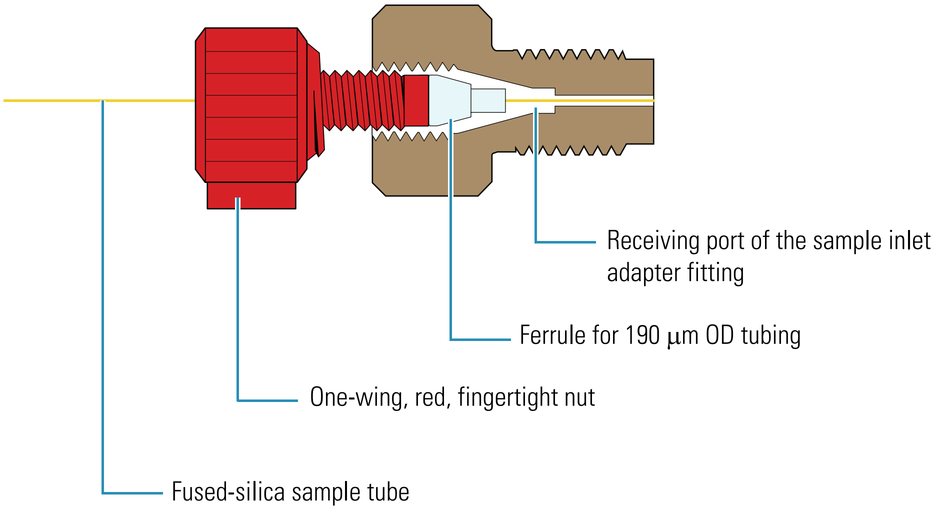
❖ **To cut and reposition the end of the fused-silica sample tube**

1. Remove the ESI probe from the Ion Max or Ion Max-S source housing (see “Removing the ESI Probe” on page 16).

2. Loosen the two-piece fingertight fitting that secures the position of the fused-silica sample tube and the PEEK safety sleeve at the ESI probe sample inlet (see Figure 20).

   Note When the nut and ferrule assembly are properly seated in the receiving port, the receiving port compresses the ferrule so that it fits snugly to the tubing. When you loosen the fitting, the receiving port no longer compresses the ferrule and the tubing is free to move.

Figure 20. Two-piece fingertight fitting loosened from the sample inlet adapter fitting



3. Gently pull back on the sample tube to free it from the ferrule.
4. Push the sample tube forward so that it extends beyond the end of the electrospray needle.
5. Using a fused-silica cutting tool, 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 flush with the ESI needle.

   The optimal sample tube protrusion depends on the solvent flow rate:

   - For flow rates below or equal to 100 µL/min, set the sample tube protrusion to 1 mm.
   - For flow rates above 100 µL/min, ensure that the sample tube is flush with the ESI needle or recessed inside the ESI needle by less than 1 mm.
7. Tighten the two-piece fingertight fitting securely to hold the sample tube in place.
8. Because the sample tube can move forward as you tighten the two-piece fingertight fitting, ensure that the sample tube is still set to the appropriate protrusion. If necessary, loosen the fitting and reposition the sample tube.
9. Reinstall the ESI probe in the API source housing (see "Installing the ESI Probe" on page 19).

Disassembling the ESI Probe

To replace or clean the ESI probe components, you must disassemble the ESI probe. To disassemble the ESI probe, you must have these tools:

- 5/16-in. wrench
- 1/2-in. wrench

❖ To disassemble the ESI probe

1. If you have not already done so, remove the ESI probe from the API source housing (see "Removing the ESI Probe" on page 16).

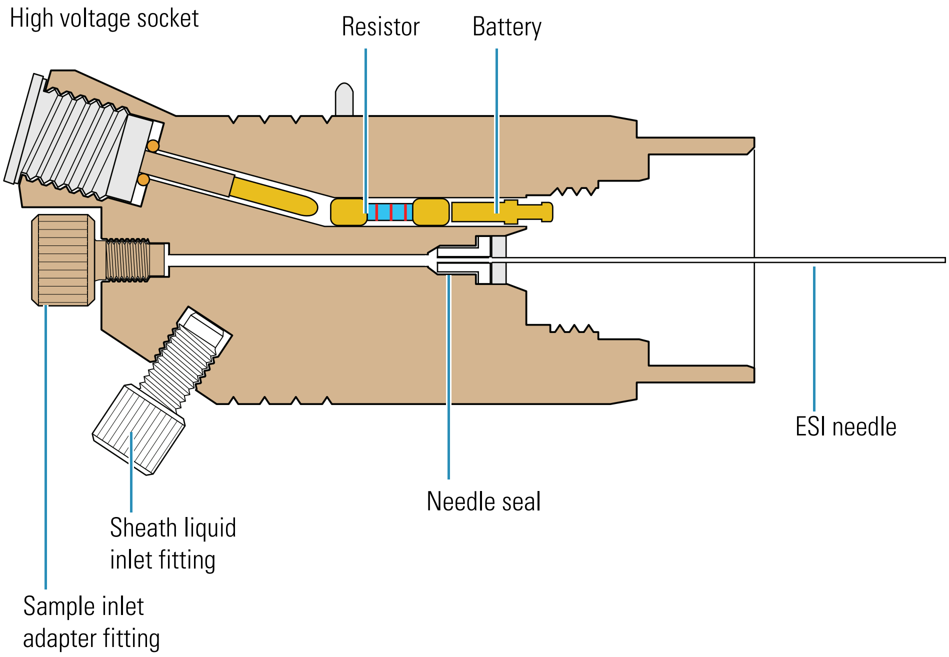
2. Unscrew the two-piece fingertight fitting from the sample inlet adapter fitting and remove the sample tube from the ESI probe.

3. Because the ESI manifold contains loose components (resistor and battery), hold the ESI probe with the nozzle facing upwards as you loosen and remove the ESI nozzle from the ESI manifold with a 5/16-in. open-end wrench.

4. If the nozzle requires cleaning, see “Cleaning the ESI Nozzle” on page 25.

5. Pull the ESI needle out of the ESI manifold (see Figure 21).

Figure 21. Cross-sectional view of the ESI probe with the nozzle removed



6. To dislodge the needle seal, gently tap the ESI manifold against a hard surface. If necessary, use the needle or another appropriate tool to push the needle seal out of the ESI manifold.

   Tapping the manifold against a hard surface also dislodges the resistor and contact battery.

7. Using a 1/2-in. wrench, disconnect the high voltage socket from the back of the ESI probe.

8. Unscrew the fitting from the sheath liquid inlet.

9. If the ESI manifold needs cleaning, see “Cleaning the ESI Manifold” on page 26.

10. To reassemble the ESI probe, see “Assembling the ESI Probe” on page 27.

Cleaning or Replacing the ESI Probe Components

Maintaining the ESI probe requires occasional replacement of the 26-gauge needle, the needle seal, the ESI nozzle and the high voltage socket O-rings. In addition, the ESI nozzle and manifold occasionally require cleaning.

To clean or replace the ESI probe components, follow these procedures:

- Cleaning the ESI Nozzle
- Replacing the Needle, Needle Seal, or Both
- Cleaning the ESI Manifold

Cleaning the ESI Nozzle

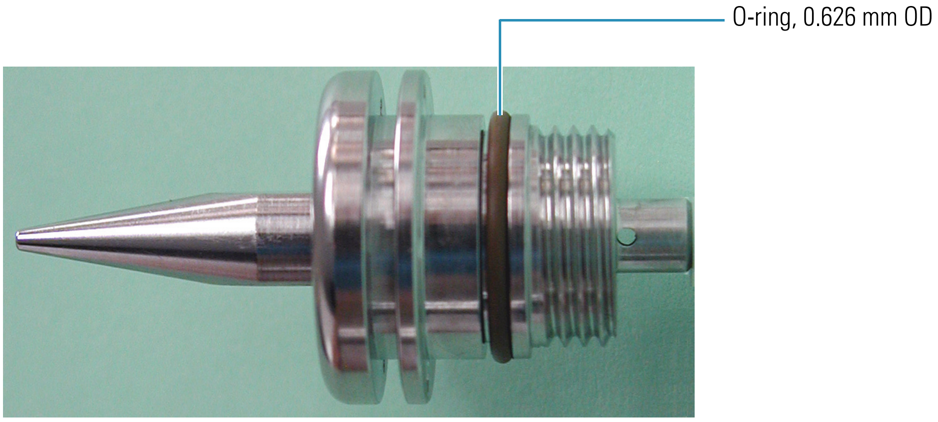
❖ To clean the ESI nozzle

1. Remove the ESI probe from the API source housing (see "Removing the ESI Probe" on page 16).
2. Disconnect the ESI nozzle from the ESI manifold (see "Disassembling the ESI Probe" on page 23, step 2 through step 3).
3. Clean the bore of the ESI nozzle with an appropriate solvent.

   The choice of solvent depends on the solubility of the chemical deposits.
4. Rinse the nozzle with methanol and dry with nitrogen gas.
5. Inspect the ESI nozzle O-ring and replace it if necessary (see Figure 22).

Part	Part number
ESI nozzle O-ring	00950-00990

Figure 22. ESI nozzle



6. Reconnect the ESI nozzle to the ESI manifold (see "Assembling the ESI Probe" on page 27, step 4 through step 6).

Replacing the Needle, Needle Seal, or Both

If the needle is damaged, replace it. If the sheath gas is leaking at the interface between the needle seal and the needle, replace the needle seal.

❖ To replace the needle, needle seal or both

1. Remove the ESI probe from the API source housing (see "Removing the ESI Probe" on page 16).
2. Disconnect the ESI nozzle from the ESI manifold (see "Disassembling the ESI Probe" on page 23, step 2 through step 3).
3. Pull the ESI needle out of the ESI manifold.
4. To dislodge the needle seal, gently tap the ESI manifold against a hard surface. If necessary, use the needle or another appropriate tool to push the needle seal out of the ESI manifold.

   Tapping the manifold against a hard surface also dislodges the resistor and contact battery.
5. If necessary, replace one or both of the following parts:

Part	Part number
Needle seal	00950-00990
ESI needle	00950-00952

6. Reassemble the ESI probe (see "Assembling the ESI Probe" on page 27, step 1 through step 6).

Cleaning the ESI Manifold

❖ To clean and dry the ESI manifold

1. If you have not already done so, remove the ESI probe from the API source housing (see "Removing the ESI Probe" on page 16).
2. Disassemble the ESI probe (see "Disassembling the ESI Probe" on page 23).
3. Rinse the ESI manifold with distilled water and then with HPLC-grade methanol. Remove excess methanol from the ESI manifold with a lint free tissue.
4. Dry the ESI manifold with nitrogen gas.
5. Inspect the following parts:

Part	Part number
ESI nozzle O-ring	00107-05710
High voltage socket O-ring	00107-02550
Teflon™ needle seal	00950-00952
26-gauge spray needle	00950-00990

6. Replace damaged parts.
7. Reassemble the ESI probe.

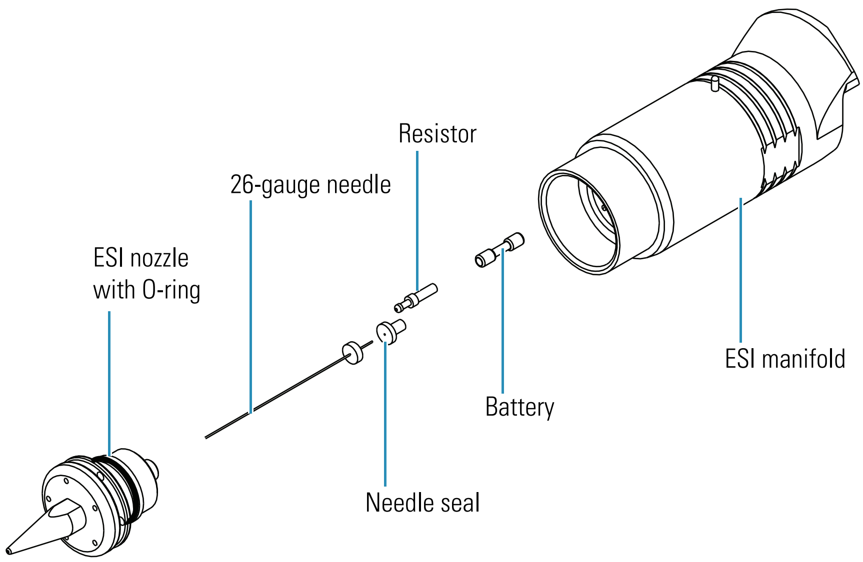
Assembling the ESI Probe

❖ To assemble the ESI probe

1. Insert the resistor and contact battery into the ESI manifold.
2. Insert the entrance end of the ESI needle into the needle seal.
3. Seat the ESI needle and needle seal in the ESI manifold.
4. Ensure that the 0.676-in. ID O-ring is placed into the precut groove on the ESI nozzle. See Figure 22 on page 25.
5. Thread the ESI nozzle over the needle and into the ESI manifold. Slightly wet the nozzle threads with HPLC-grade methanol for lubrication.
6. 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 23 shows an exploded view of front end of the ESI probe.

   Figure 23. Exploded view of the front end of the ESI probe



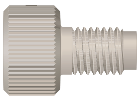
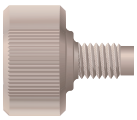
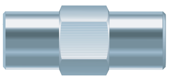
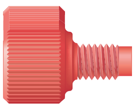
7. Insert the high voltage socket into the back of the ESI manifold. Using a 1/2-in. wrench, tighten the socket.
8. Reconnect the sheath liquid fitting.
9. Install a new sample tube (see "Installing a Fused-Silica Sample Tube and PEEK Safety Sleeve" on page 28).

Installing a Fused-Silica Sample Tube and PEEK Safety Sleeve

When you use a fused-silica sample tube with the ESI probe, you must cover the exposed portion of the sample tube with a PEEK safety sleeve.

CAUTION AVOID ELECTRICAL SHOCK. Cover the fused-silica capillary tube with the PEEK safety sleeve and associated PEEK ferrules provided in the Safety Sleeve Kit (P/N 70005-62015) before you operate the instrument. The PEEK tubing acts as a second level of protection against accidental electrical discharge.

Installing a fused-silica sample tube with the PEEK safety sleeve requires these parts:

Part	Description	Part number
	Tubing, fused-silica, 0.1-mm ID × 0.190-mm OD	00106-10499
	Tubing, red PEEK, 0.005-in. ID × 1/16-in. OD	00301-22912
	PEEK safety sleeve, pre-cut 25 cm (10 in.) length of natural PEEK tubing 0.23-mm ID × 0.61-mm OD (0.009-in. ID × 0.0240-in. OD)	00301-22806
	Fitting, adapter, 10-32 to 1/4-28, natural PEEK, 0.040-in. (1.0-mm) thru-hole (for the ESI probe sample inlet) (Upchurch P-669)	00101-18080
	Fitting, fingertight, natural PEEK, two-wings, for 1/16-in. OD high-pressure tubing (Upchurch F-300)	00101-18081
	Ferrule, 0.027-in. ID, natural PEEK (for use with the 0.024-in. OD PEEK safety sleeve)	00101-18119
	Fitting, grounding union, stainless steel, 0.010-in. thru-hole (Upchurch U-435)	00101-18182
	Fitting, fingertight, for 1/16-in. OD high pressure tubing (Upchurch F-200)	00101-18195

To install a fused-silica sample tube, you have to thread the 0.19-mm OD fused-silica sample tube through the ESI needle that protrudes from the ESI probe nozzle and the 0.23-mm ID safety sleeve. Because you have to thread the fused-silica tubing through these small orifices, you might find a microscope useful.

❖ To install the new sample tube and PEEK safety sleeve

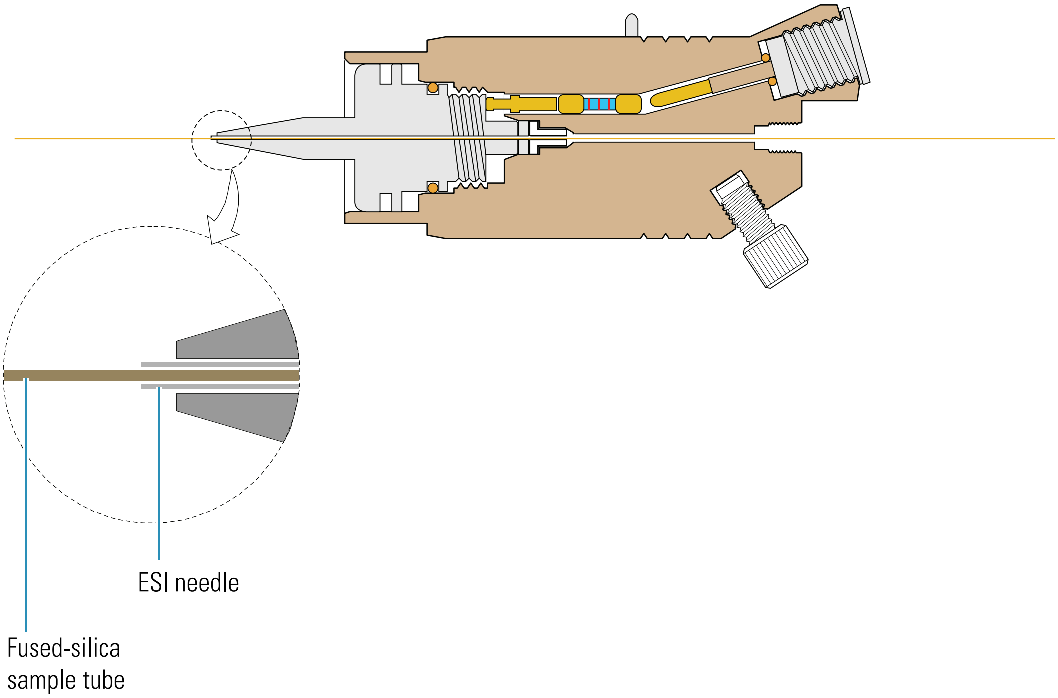
1. Using a fused-silica cutting tool to ensure square cuts, cut a length of fused-silica tubing approximately 38 cm (15 inch) long.

   The piece of fused-silica tubing must be long enough to extend through the ESI probe and the natural PEEK safety sleeve.

2. Remove the sample inlet adapter from the ESI probe sample inlet.

3. Insert the fused-silica sample tube through the ESI needle that protrudes from the ESI nozzle, and then push the sample tube through the ESI probe until approximately 3.5 cm (1.5 in.) of the sample tube protrudes from the front of the probe. The remaining length of sample tube protrudes from the ESI probe sample inlet at the back of the probe (see Figure 24).

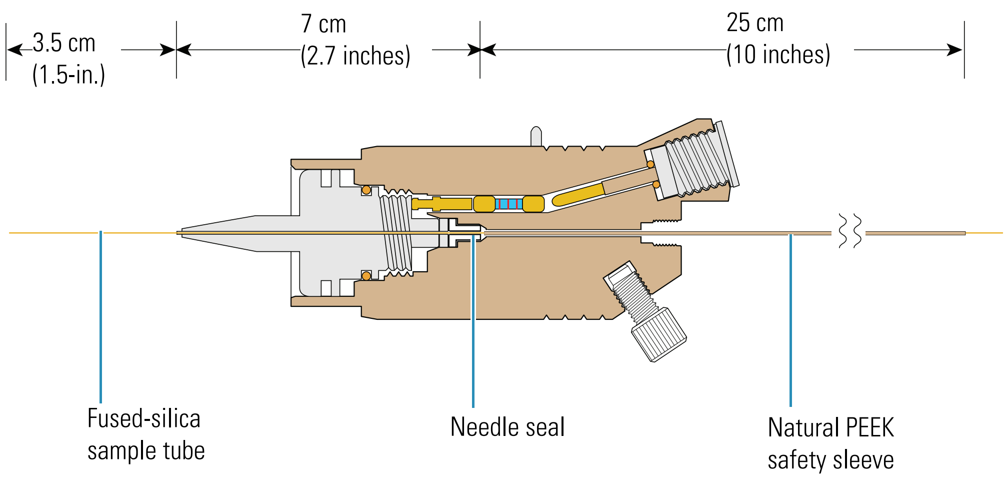
Figure 24. Inserting the fused-silica sample tube through the front of the ESI probe



4. Slide the safety sleeve over the end of the fused-silica sample tube that protrudes from the probe sample inlet, and then push the safety sleeve into the probe until it meets resistance (see Figure 25).

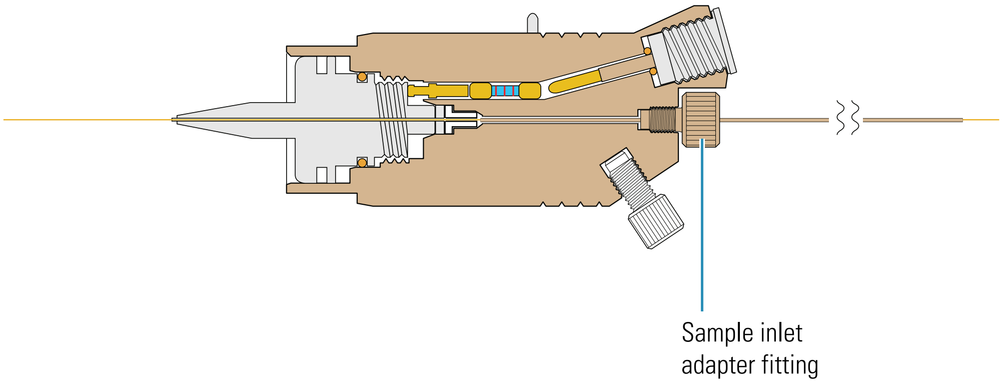
   The safety sleeve is a a pre-cut length of natural PEEK tubing that acts as a second level of protection against accidental electrical discharge.

Figure 25. Installing the natural PEEK adapter into the probe sample inlet



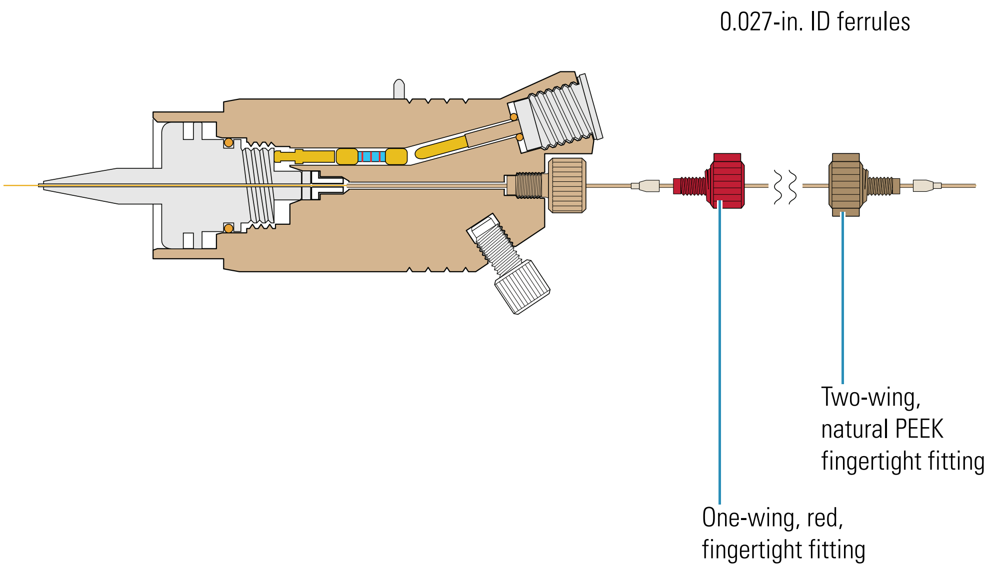
5. With the external threads facing the ESI probe sample inlet, slide the 10-32 × 1/4-28 natural PEEK fitting adapter over the safety sleeve, and then fingertighten the fitting into the ESI probe sample inlet (see Figure 26).

Figure 26. Inserting the safety sleeve into the probe sample inlet



6. Slide the fingertight fittings onto the PEEK sleeve (see Figure 27) as follows:
   a. Slide the 0.027 in. ID PEEK ferrule with the tapered end facing the sample inlet onto the PEEK safety sleeve.
   b. Slide the red, one-wing fingertight fitting with the threaded end facing the sample inlet onto the PEEK safety sleeve.
   c. Slide the natural PEEK, two-wing fingertight fitting with the threaded end facing away from the sample inlet onto the PEEK safety sleeve.
   d. Slide the 0.027 in. ID PEEK ferrule with the tapered end facing away from the sample inlet onto the PEEK safety sleeve.

Figure 27. Sliding the two-piece fingertight fitting over the safety sleeve and into the adapter

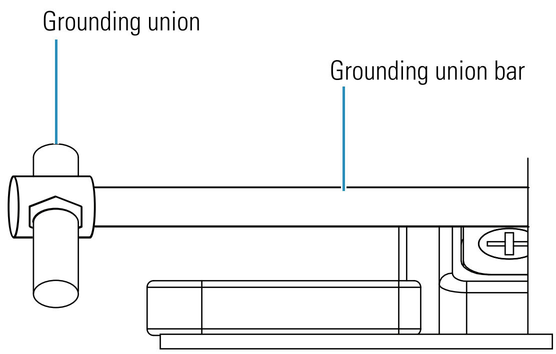


7. Connect the safety sleeve and fused-silica assembly to the grounding union as follows:

   a. Adjust the position of the fused-silica sample tube so that it is flush with the end of the safety sleeve that protrudes from the back of the probe.

   b. To provide leverage when you tighten the fitting to the union, seat the grounding union in the grounding union bar of the ion source housing (see Figure d).

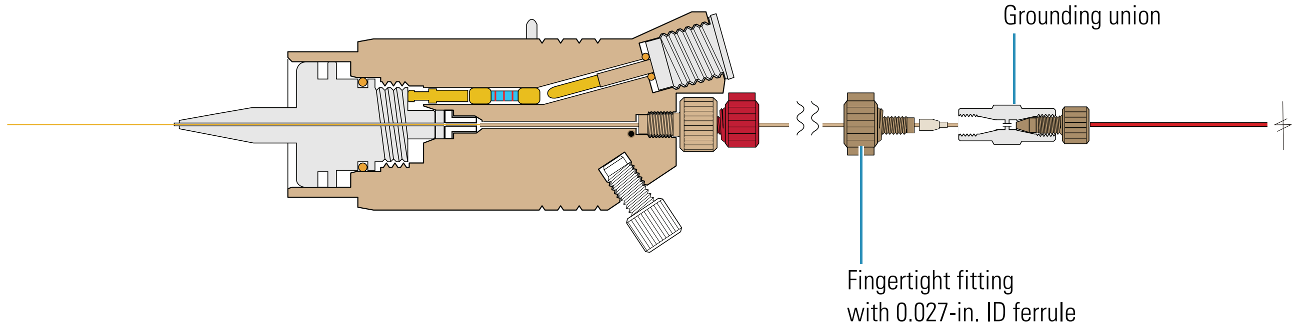
   Figure 28. Grounding union seated in grounding bar

   c. To prevent the 0.190 mm OD fused-silica sample tube from slipping through the 0.010 inch (0.25 mm) grounding union thru-hole and out the other end of the grounding union, connect a fingertight fitting and red PEEK tubing to the other end of the grounding union (see Figure 29).

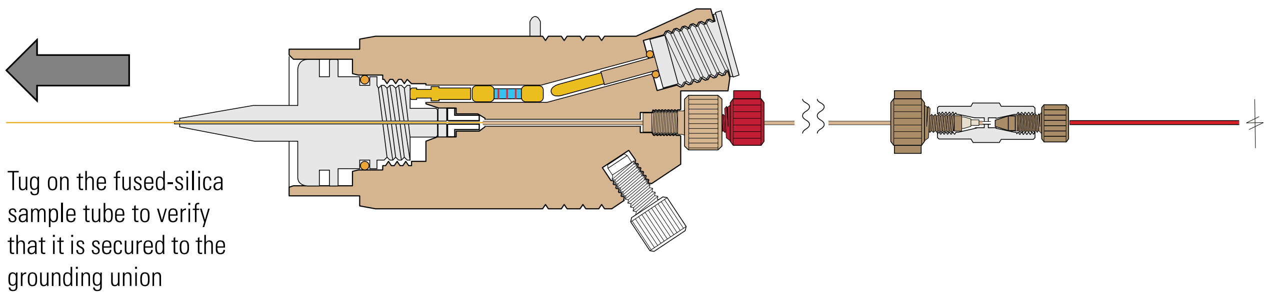
   d. While holding the safety sleeve and fused-silica sample tube firmly against the grounding union receiving port, tighten the fitting as tight as you can with your fingers (see Figure 29 and Figure 30).

Figure 29. Connecting the safety sleeve to the grounding union



   e. Ensure that the fused-silica sample tube is held tightly in the grounded union by gently pulling the sample tube from the exit end of the ESI needle (see Figure 30).

Figure 30. Checking that the fused-silica sample tube is securely tightened to the grounding union



8. Adjust the position of the fused-silica sample tube (see Figure 31 and Figure 32) as follows:

   a. Using a fused-silica cutting tool, cut the fused-silica sample tube so that 2.5 cm (1-in.) of the sample tube protrudes from the ESI needle.

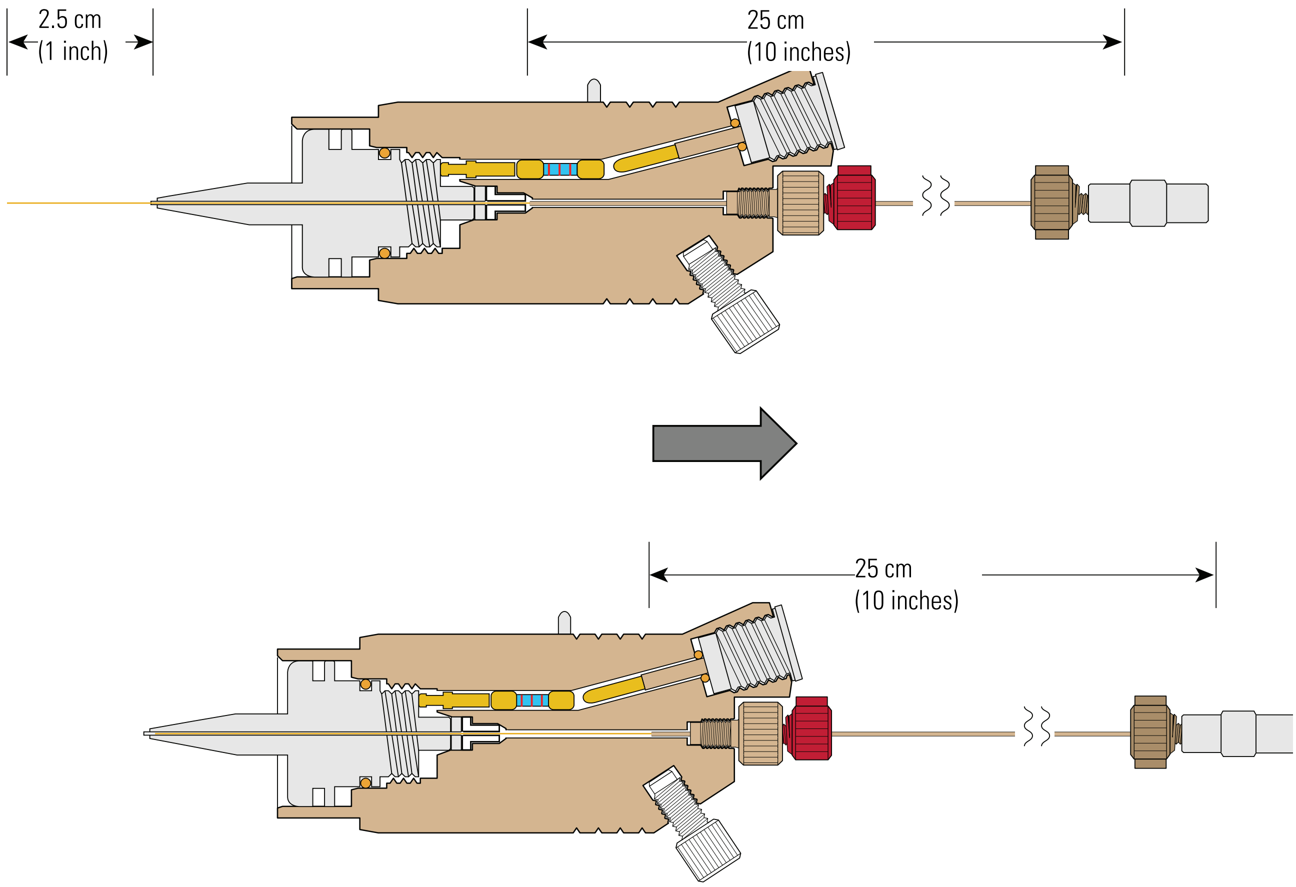
   b. From the ESI sample inlet, loosen the red PEEK fitting, and then pull the PEEK safety sleeve backwards so that the fused-silica sample tube is positioned appropriately within the ESI needle.

      The optimal position of the sample tube depends on the solvent flow rate:

      - For flow rates below or equal to 100 µL/min, set the sample tube protrusion to 1 mm from the ESI needle tip.

      - For flow rates above 100 µL/min, ensure that the sample tube is flush with the ESI needle tip or recessed inside the ESI needle by no more than 1 mm.

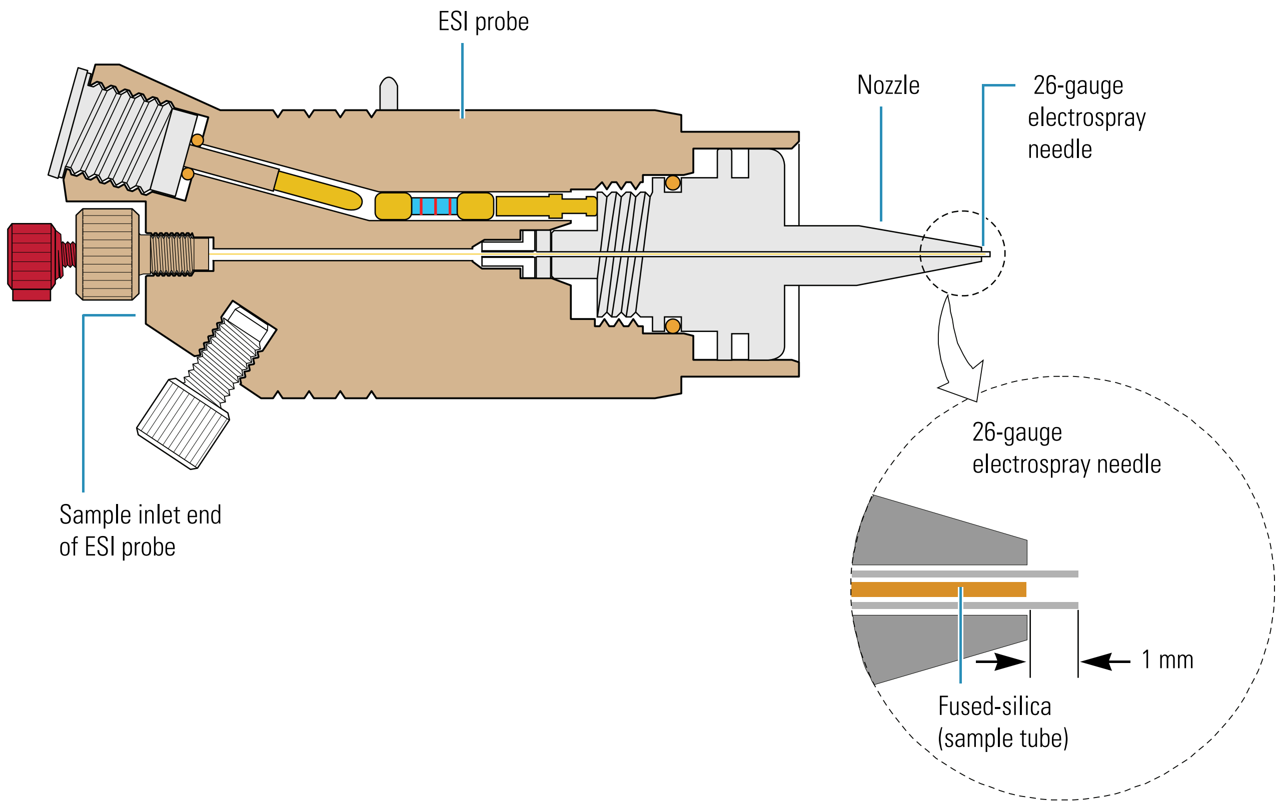
Figure 31. Adjusting the position of the fused-silica sample tube



   c. Tighten the fingertight fitting securely to hold the PEEK safety sleeve and sample tube in place.

   d. Because the sample tube can move forward when you tighten the sample inlet fitting, ensure that the sample tube is recessed within the ESI needle. If necessary, loosen the red PEEK fitting and reposition the sample tube.

Figure 32. Installing the ESI fused-silica sample tube



Installing an Optional Metal Needle Sample Tube

You can configure the 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 for the ESI Probe

Figure 33 and Figure 34 show these parts.

Probe, ESI OPTON-20011

Body-probe manifold 97055-20300
Nozzle-ESI probe 3-port 97055-20146
Fitting, union, 1/4-28, PEEK. 00109-00304
Battery, contact, Be Cu, 0.598-mm length, 0.02 ohm, 4A 00004-21402
Seal, standard needle, 5000 series 00950-00952
Needle, D point, 26 gauge, 2.7-in. length, 0.24-in. OD washer 00950-00990
Connector receptacle, high voltage, shielded 00004-89626
Ferrule, 0.012-in. ID, KEL-F HPLC 00101-18116
Fitting, Fingertight 2 Upchurch™ 00101-18195
Fitting, plug, 1/4-28, Tefzel®, HPLC 00101-18075
O-ring, 0.676-in. ID × 1/16-in. THK, Viton™ 00107-05710
O-ring, 0.125-in. ID × 1/16-in. THK, Viton 00107-02550
Assembly-resistor contact-ESI probe. 97055-60058
Resistor FXD CC 1/4W 22M 5%, ROHS 00015-02-00032
Fitting, HPLC adapter, 10-32 × 1/4-28, natural PEEK 00101-18080
Tubing, fused-silica, 0.10 mm ID × 0.19 mm OD. 00106-10499
Ferrule, 0.008-in. ID, KEL-F HPLC 00101-18114

Safety Sleeve Kit 70005-62015

Ferrule, 0.027-in. ID, PEEK HPLC. 00101-18119
Tube, 0.009-in. ID × 0.024-in. OD, 10-in. length, natural PEEK. 00301-22806
Fitting, one-wing, red nut with natural PEEK ferrule, Fingertight 2 (Upchurch F-200) 00101-18195
Fitting, natural PEEK, 10-32, two-wing nut, fingertight, HPLC (Upchurch F-300) 00101-18081

Stainless steel needle kit, 32 gauge OPTON-53003

Stainless steel needle kit, 34 gauge OPTON-30004

Figure 33. Exploded view of the ESI probe

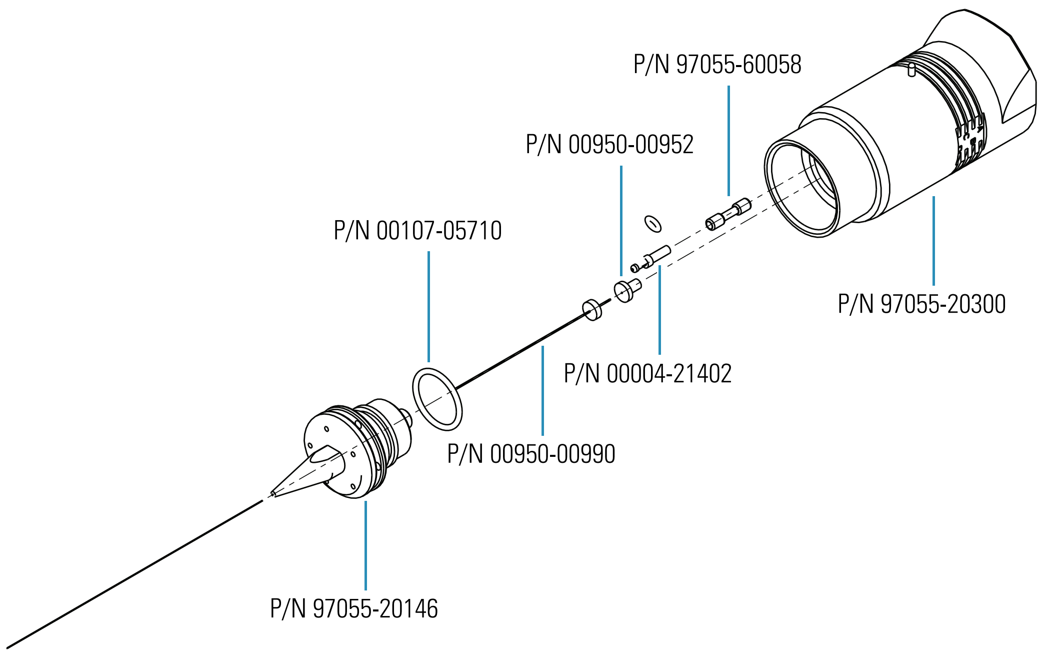
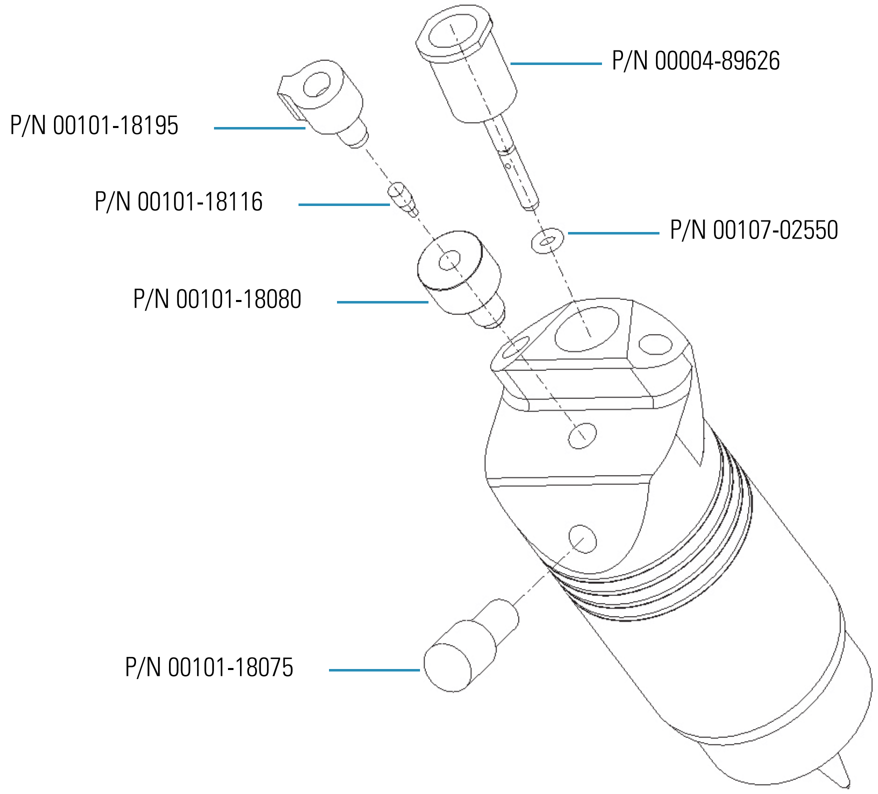


Figure 34. Replaceable parts for the ESI probe



Heated-Electrospray Ionization

This chapter describes how to install and maintain the HESI-II 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 HESI-II probe.

Contents

Introduction to the HESI-II Probe

You can perform heated electrospray ionization to introduce gas phase ions into the mass spectrometer with the HESI-II probe (see Figure 35) and a Thermo Scientific mass spectrometer.

Figure 35. HESI-II probe

Table 3 shows initial H-ESI settings for different liquid flow rates for a 50 percent aqueous solution. These initial settings provide a starting point for optimizing system performance. Refer to the getting started guide for your mass spectrometer for information on optimizing the system performance for your compound.

For best results, avoid operating the HESI-II probe at elevated temperatures without solvent flow from the LC system or the syringe pump. Allowing the HESI-II probe to run dry at elevated temperatures can cause blockage of the replaceable metal needle insert (see "Replacing the Needle Insert" on page 52).

Table 3. Initial H-ESI settings for the HESI-II probe

Liquid flow rate (µL/min)	Ion transfer tube temperature (°C	H-ESI vaporizer temperature (°C)	Sheath gas pressure (psi)	Auxiliary gas flow (arbitrary units)	Spray voltage (V)	Typical nitrogen gas consumption (L/min)
5	240	Off to 50	5	0	+3000 (-2500)*	<1
200	350	250 to 350	35	10	+3000 (-2500)	8
500	380	300 to 500	60	20	+3000 (-2500)	13
1000	400	500	75	30	+3000 (-2500)	17

*Negative ion mode

Functional Description of the HESI-II Probe

The HESI-II probe produces charged aerosol droplets that contain sample ions. The HESI-II probe accommodates liquid flows of 1 µL/min to 1 mL/min without splitting.

The removable components of the HESI-II probe are the end cover that is secured to the probe body with socket head screws and the metal needle insert that screws into the probe body (see Figure 36). The end cover includes the high voltage feedthrough and the sample inlet port. The external components of the probe body include the grounding union holder, vaporizer cable connector socket, probe sleeve with depth markers, and probe nozzle (see Figure 36 and Figure 37).

Figure 36. HESI-II probe removable components

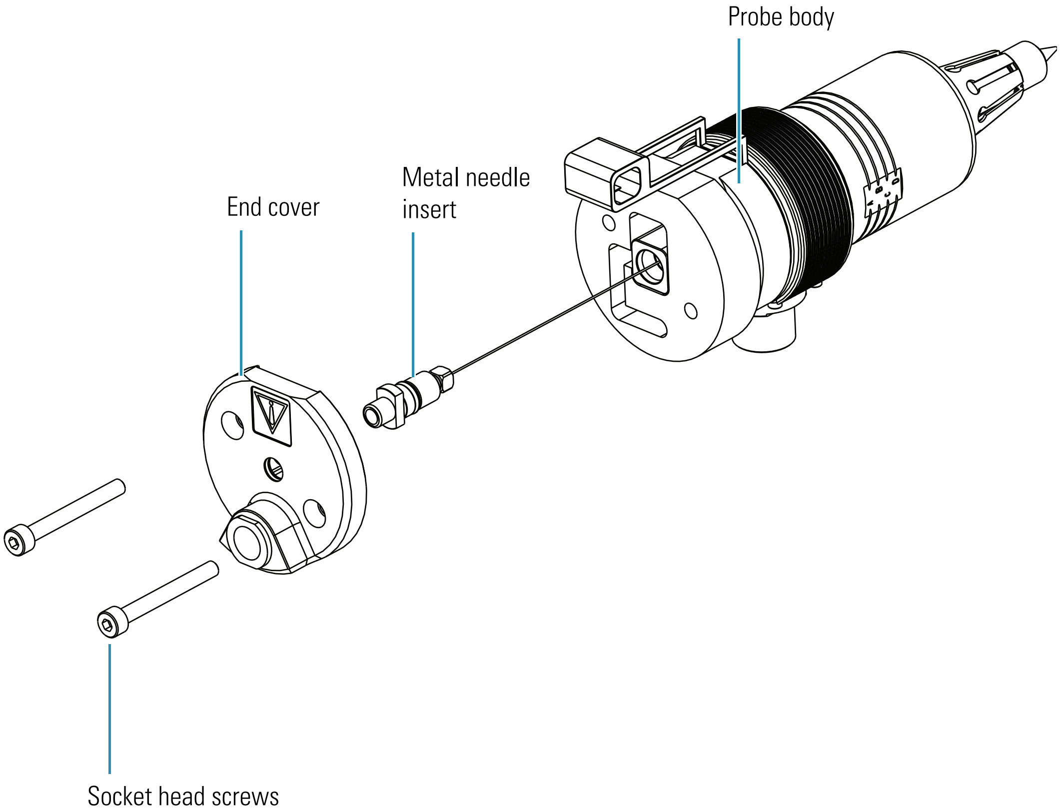
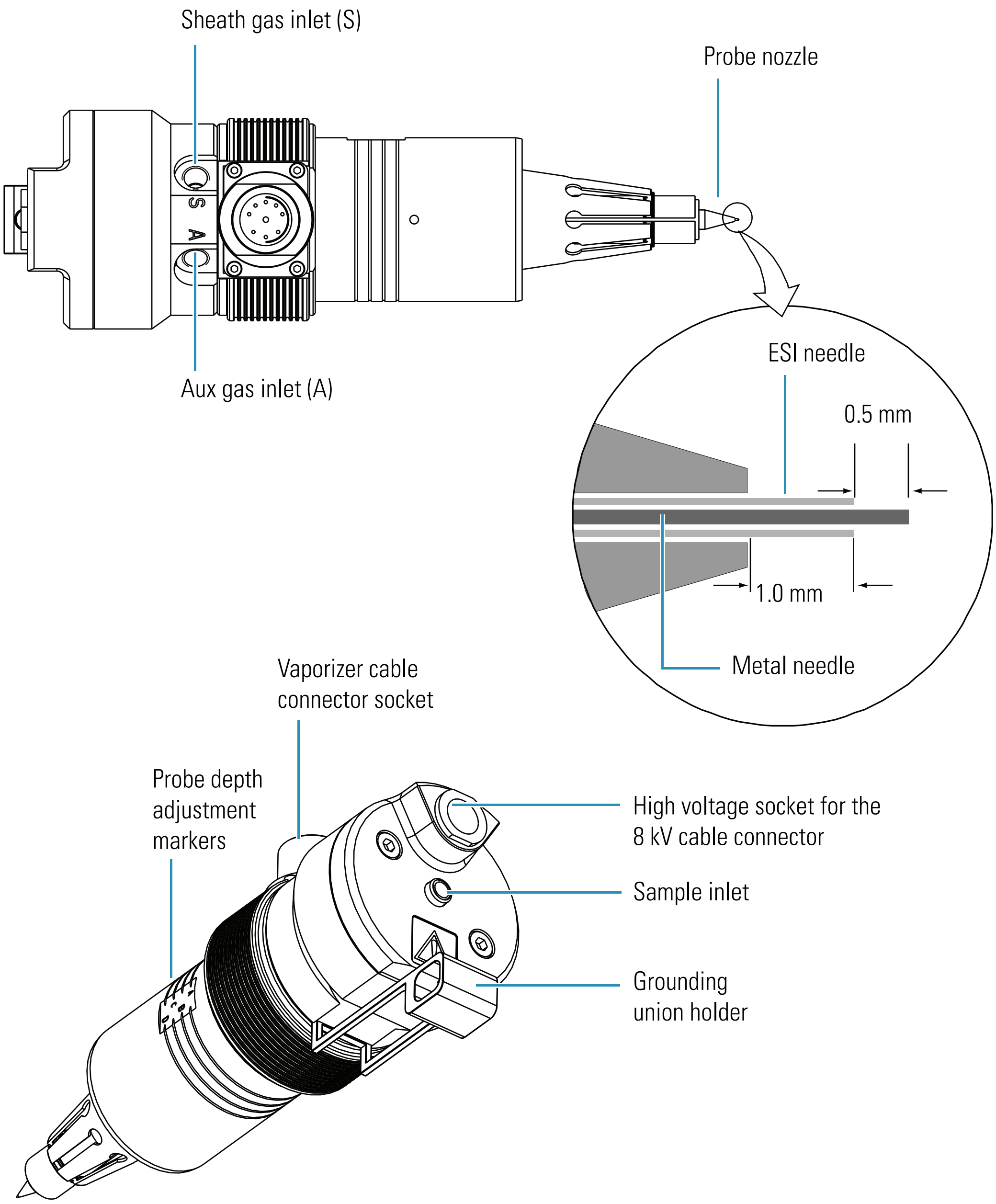


Figure 37. HESI-II probe views



Sample and solvent enter and exit the HESI-II probe through the metal needle insert, which protrudes from the sample inlet port at the back of the probe and from the nozzle at the front of the probe (see Figure 37). The metal needle insert (see Figure 36 on page 39) includes an adjustable union, a needle guide fitting and ferrule, an ESI needle, and a metal needle (see Figure 37). The adjustable union is used to adjust protrusion of the insert tip from the probe nozzle.

A large negative or positive voltage is applied to the ESI needle (typically ±3 to ±5 kV), which sprays the sample solution into a fine mist of charged droplets. The probe nozzle directs the flow of sheath gas and auxiliary gas at the droplets. The probe body houses the probe nozzle and metal needle insert, and includes the sheath gas and auxiliary gas plumbing.

The probe body has inlets for the sheath and auxiliary gases. The sheath gas is the inner coaxial nitrogen gas that sprays (nebulizes) the sample solution into a fine mist as it exits the metal needle sample tube (see Figure 38). The heated auxiliary gas is the outer coaxial nitrogen gas that assists the sheath gas in the desolvation of sample solutions (see Figure 39). The auxiliary gas heats as it passes through a vaporizer. The vaporizer is thermally insulated from the sample tube to prevent direct heating of the sample solution. You can control the vaporizer temperature from the Xcalibur™ data system. The temperature range is from ambient room temperature to 600 °C. For recommended operating temperatures and gas flow settings, see Table 3 on page 38.

Figure 38. Sheath gas plumbing

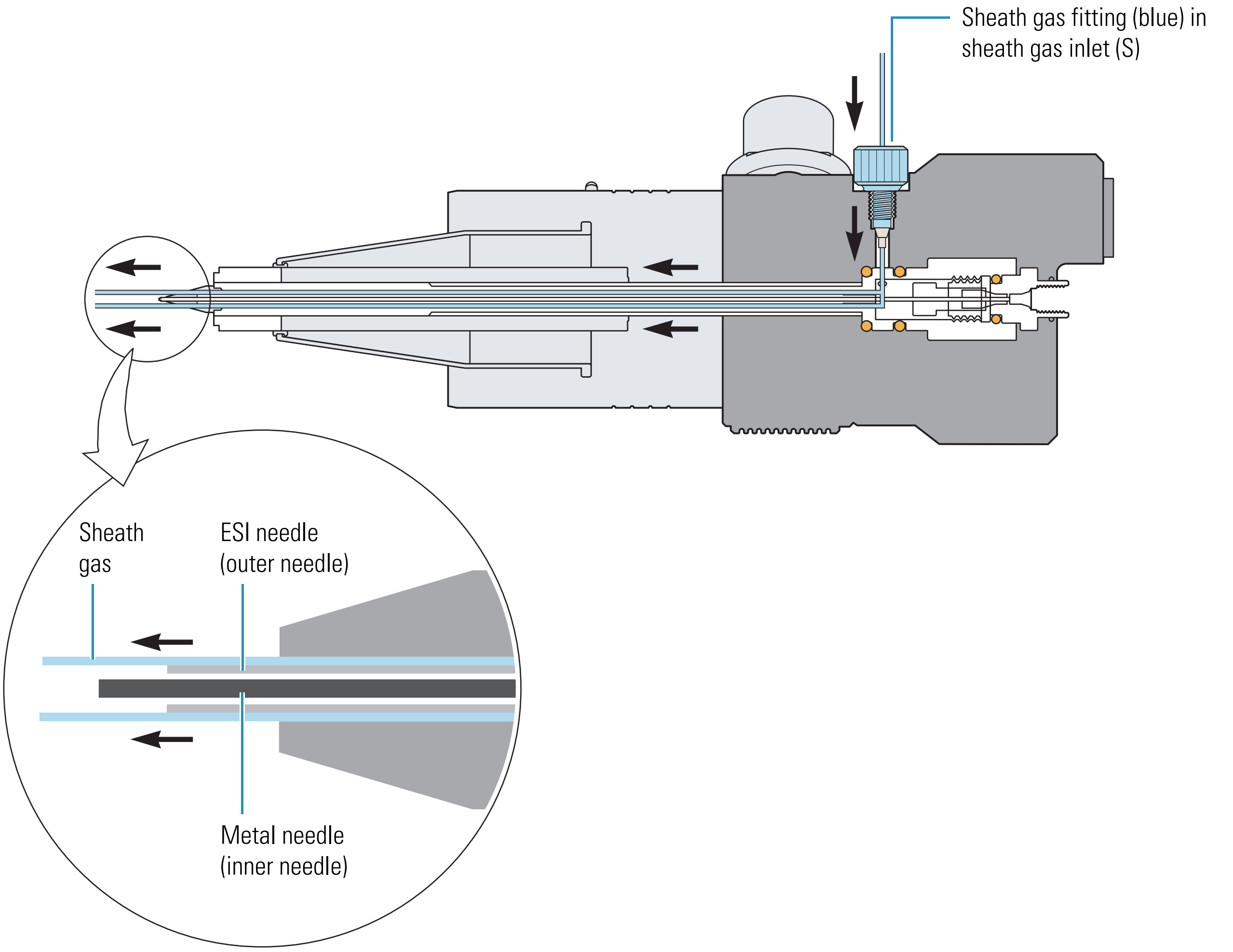
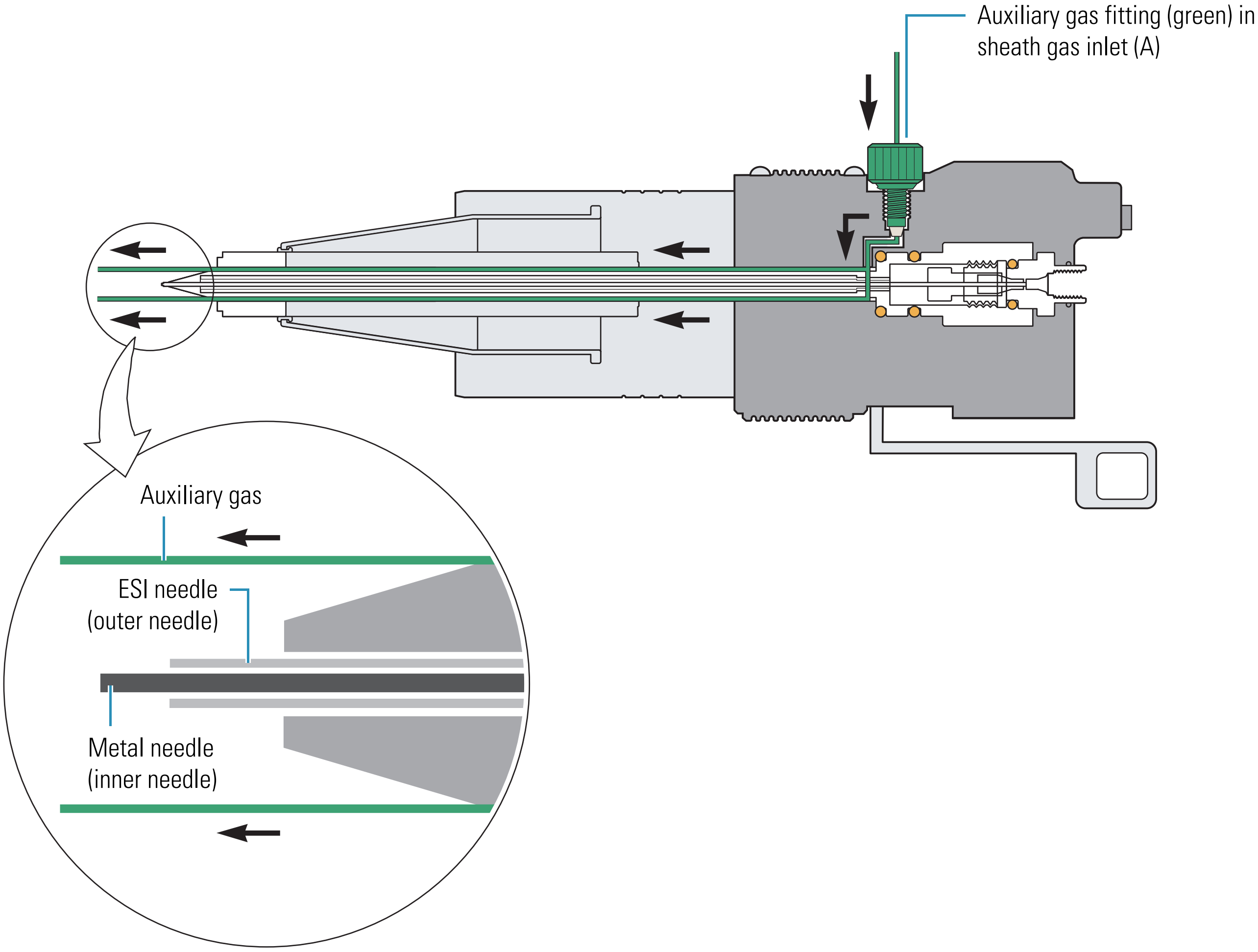


Figure 39. Auxiliary gas plumbing



The angle of the HESI-II probe is fixed at approximately 60 degrees. To optimize the spray stability, use the A, B, C, and D markers on the probe body as a reference to adjust the probe depth and the adjustment screws on the Ion Max source housing to make small changes to the probe position. 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 HESI-II Probe

CAUTION AVOID BURNS. At typical operating temperatures (350 to 450 °C), the HESI-II probe can severely burn you. Before removing the probe from the API source housing, allow the probe to cool to room temperature (for approximately 20 minutes) before you touch it.

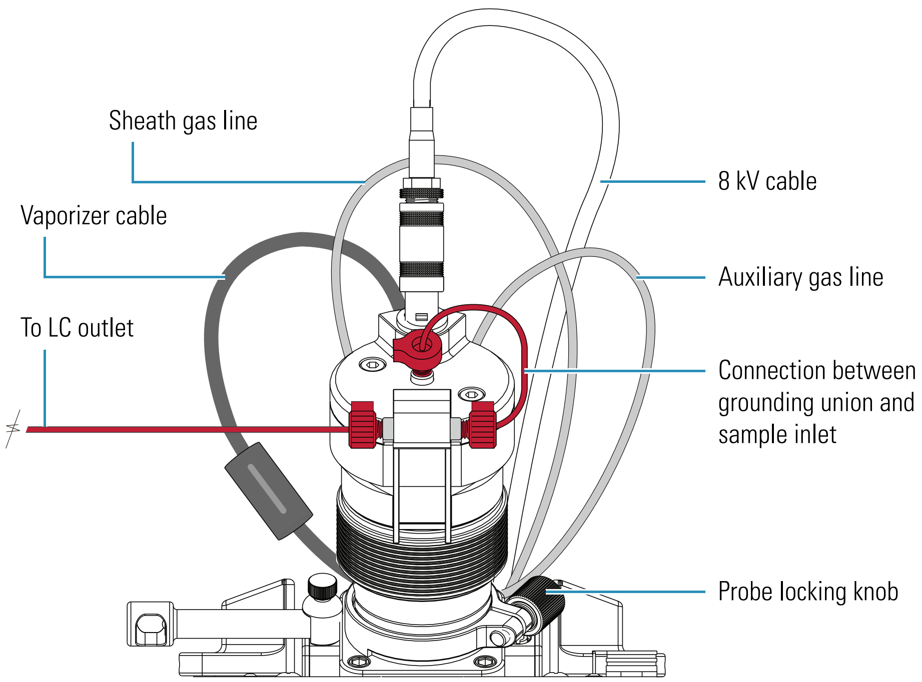
❖ **To remove the HESI-II probe from the API source housing**

1. Place the mass spectrometer in Standby mode, and allow time for the probe to cool to room temperature.

   Wait approximately 20 minutes for the probe to reach room temperature when it has been operating at high temperature. If the mass spectrometer is connected to an LC system, leave the solvent flow from the LC pump on while the probe is cooling to room temperature.

2. If the mass spectrometer is connected to an LC system, turn off the solvent flow from the LC pump, and then disconnect the tubing from the left side of the HESI-II probe grounding union (see Figure 40).

Figure 40. Front view of the connections to the HESI-II probe

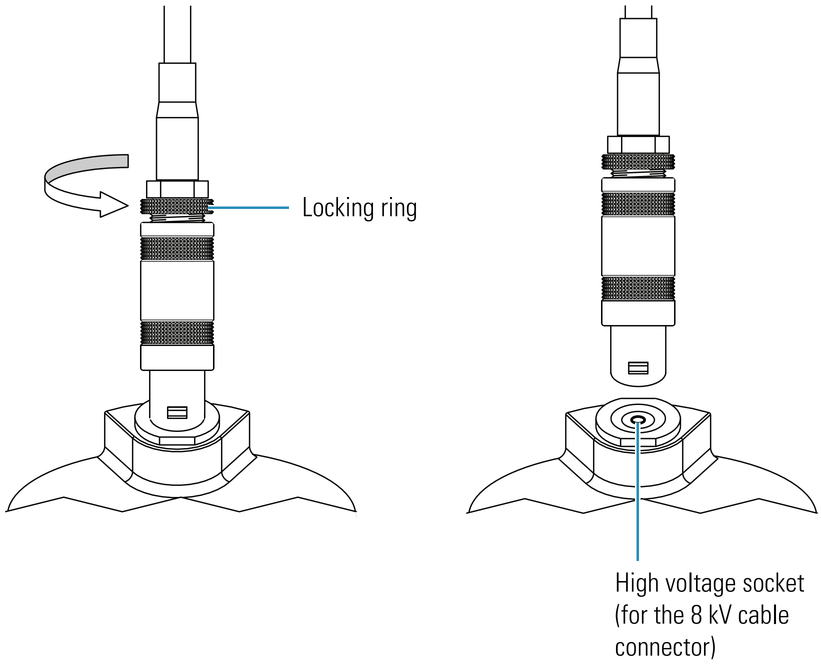


3. Disconnect the 8 kV cable from the probe high voltage receptacle as follows (see Figure 41 and Figure 42):

   a. Unlock the cable by twisting the locking ring counterclockwise.

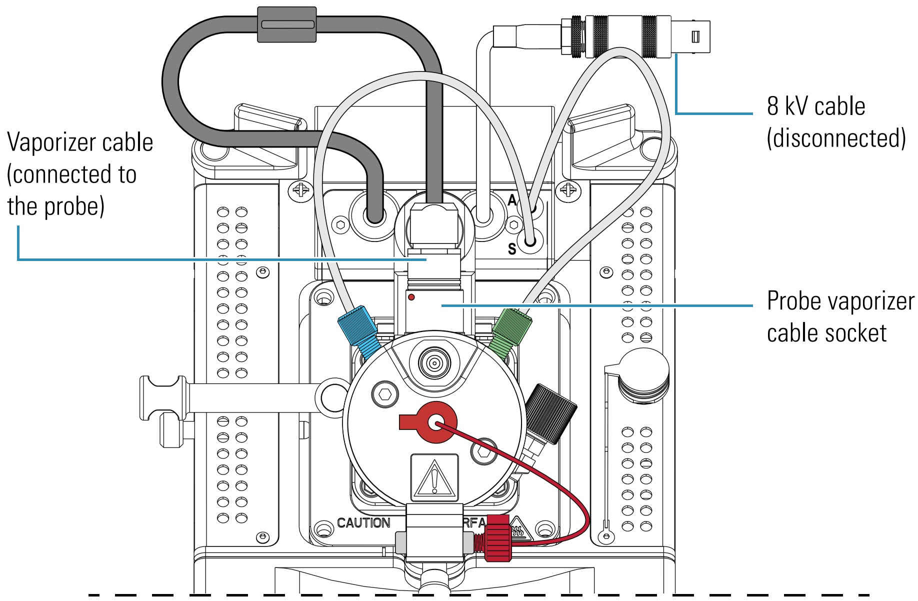
   b. Unplug the 8 kV cable from the probe high voltage socket.

Figure 41. Enlarged view of the 8 kV cable connector



4. Unplug the vaporizer cable from the probe vaporizer cable socket (see Figure 42 and Figure 43).

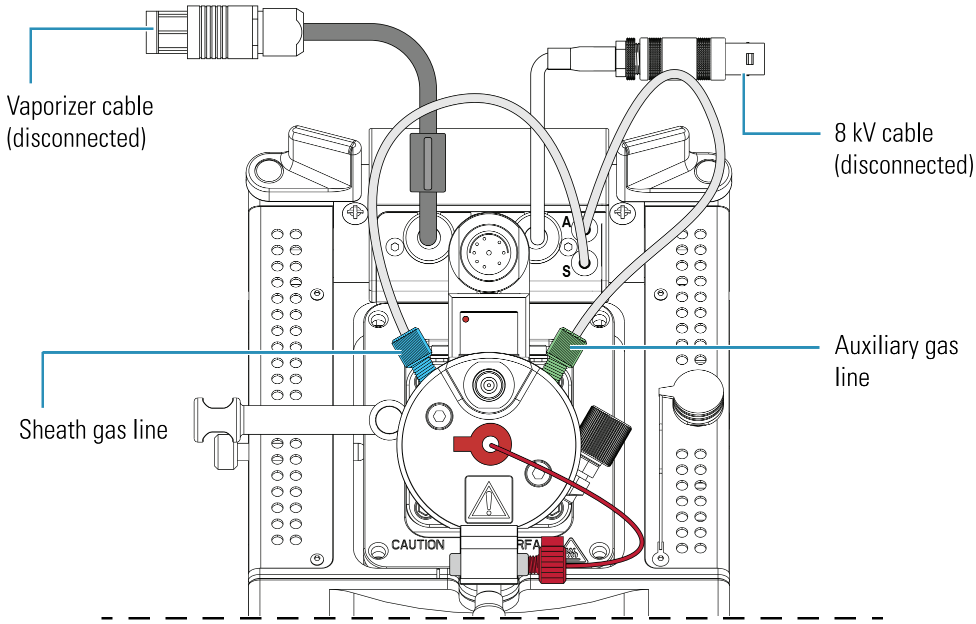
Figure 42. Top view of the API source housing and the HESI-II probe



5. Disconnect the nitrogen gas lines (see Figure 43):

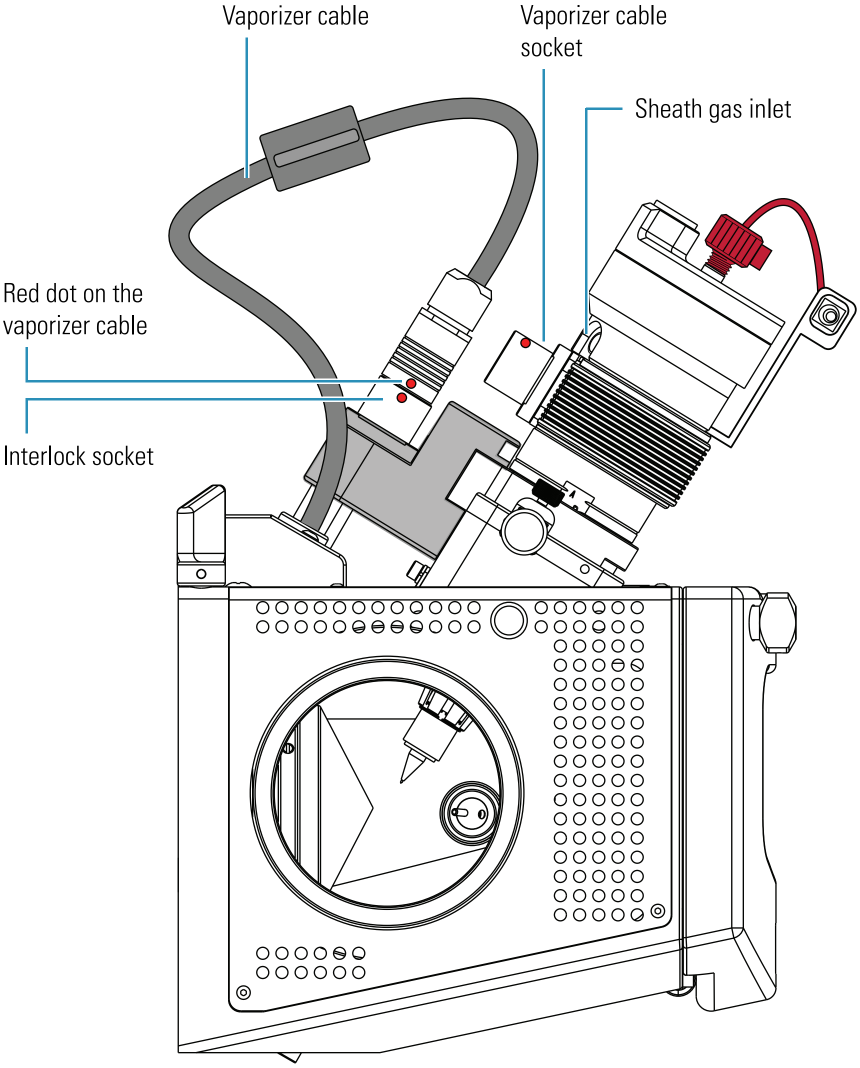
   - Disconnect the auxiliary gas fitting (green) from the auxiliary gas inlet (A) on the probe.
   - Disconnect the sheath gas fitting (blue) from the sheath gas inlet (S) on the probe.

Figure 43. Top view showing the sheath and auxiliary gas lines connected to the probe



6. Connect the vaporizer cable to the interlock socket on the interlock block. To align the socket pins, align the red dot on the vaporizer cable connector with the red dot on the interlock socket (see Figure 44).

Figure 44. View of the left side of the API source housing



7. Unlock the probe locking ring by turning the probe locking knob (see Figure 40 on page 43) counterclockwise.

8. To remove the probe from the port in the API source housing:

   a. Slowly pull the probe out of the port until you feel the resistance caused by the probe guide pin meeting the interlock block.

   b. Turn the probe counterclockwise until the guide pin is free of the interlock block.

   c. When the guide pin is free of the interlock block, pull the probe out of the port.

9. Store the HESI-II probe in its original shipping container.

Installing the HESI-II Probe

Note To remove an APCI or ESI probe, refer to the appropriate chapter in this manual.

❖ To install the HESI-II probe

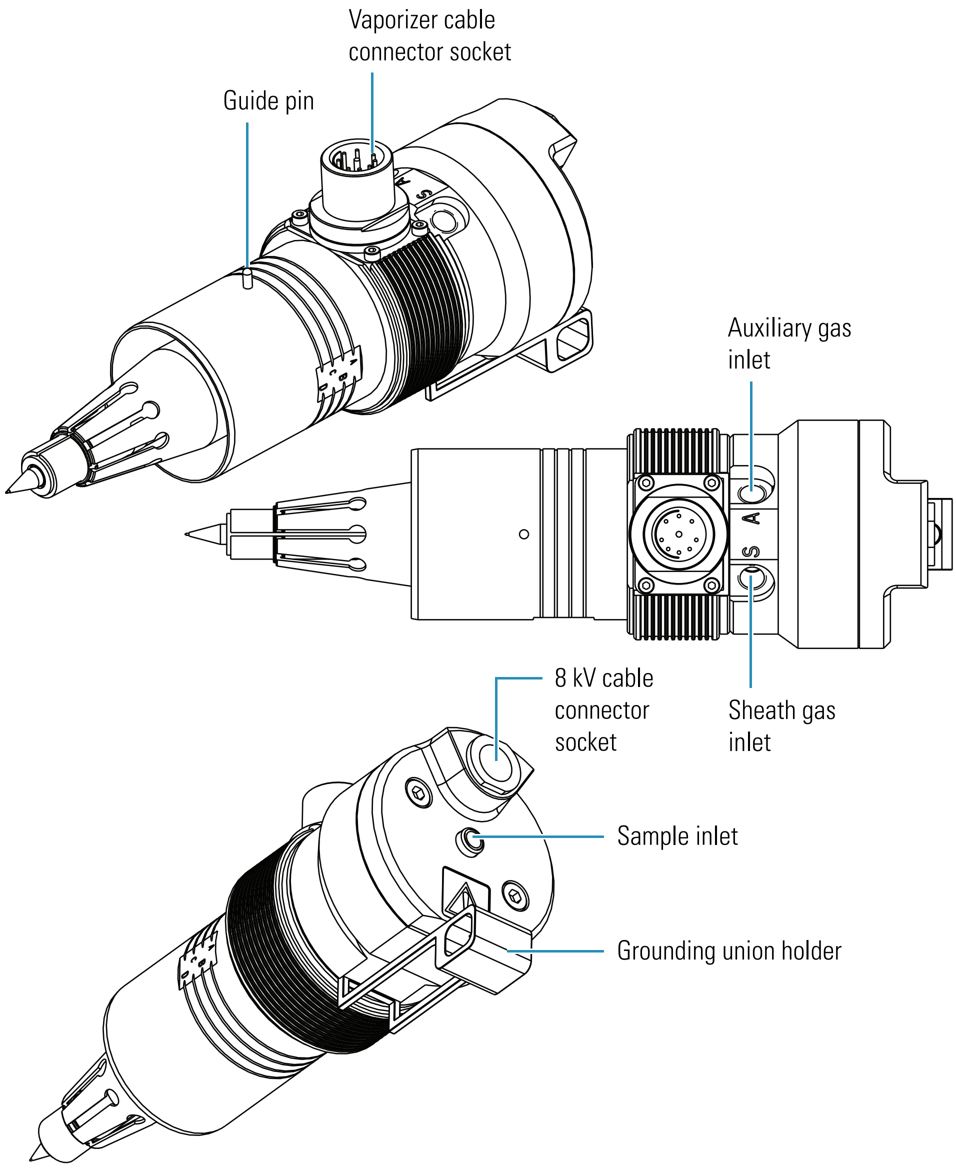
1. Place the mass spectrometer in Standby mode.

   Note When the mass spectrometer is in Standby, the high voltage and nitrogen gas are off.

2. Remove the HESI-II probe from its storage container. Inspect and clean it if necessary.

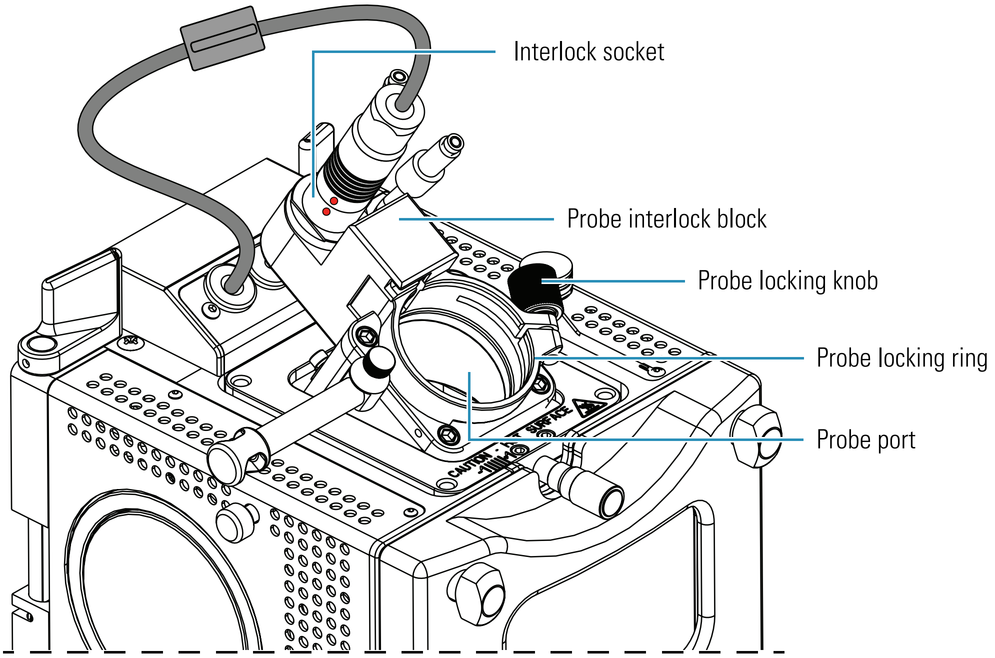
   Figure 45 shows the location of probe guide pin, vaporizer cable connector socket, sheath and auxiliary gas inlets, 8 kV cable connector socket, sample inlet, and grounding union holder.

   Figure 45. HESI-II probe views



3. Turn the probe locking knob counterclockwise until the probe locking ring is opened to its widest position (see Figure 46).

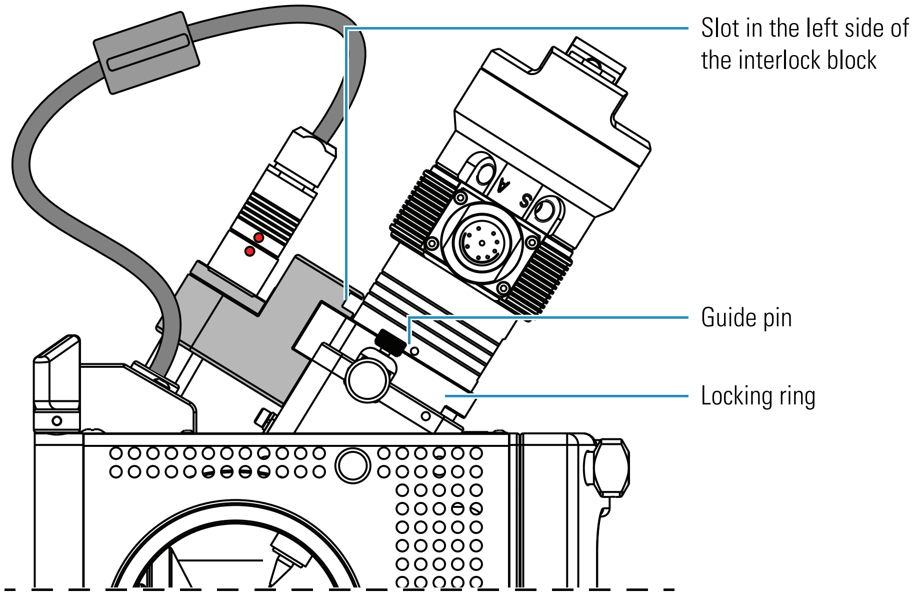
Figure 46. Ion Max API source housing without a probe



4. To seat the probe in the probe port:

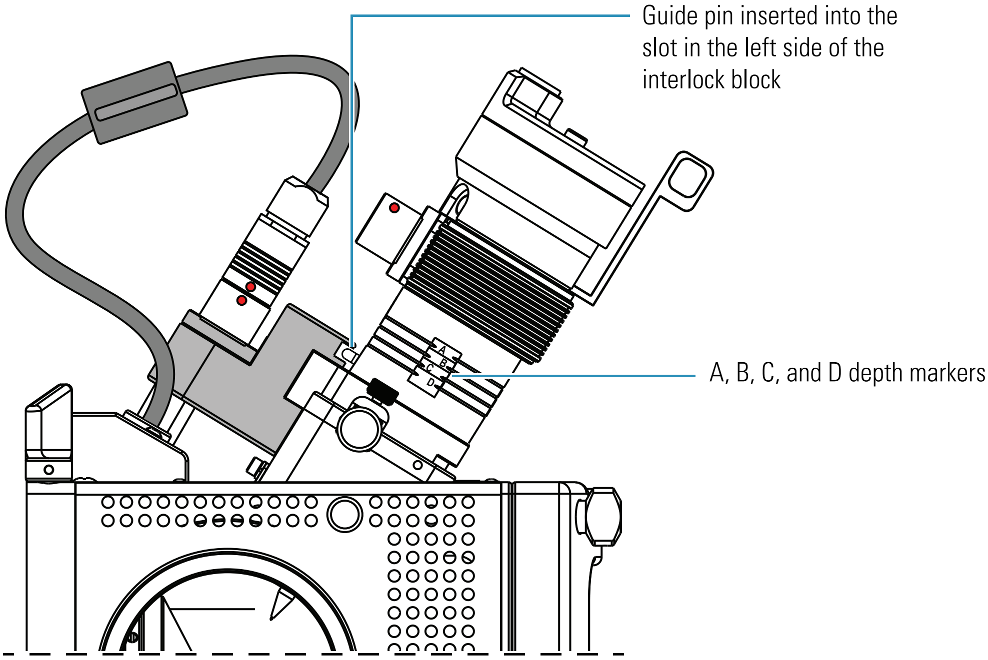
   a. Holding the probe with the nozzle facing downwards and the guide pin facing toward the left, slowly insert the probe into the port until the guide pin meets the locking ring on the API source housing (see Figure 47).

   Figure 47. View of the guide pin touching the locking ring



b. Pull the probe slightly upward until the guide pin is level with the slot on the left side of the interlock block. Then turn the probe clockwise until the guide pin meets resistance from the interlock block (see Figure 48).

Figure 48. View of the guide pin inserted into the slot



c. Push the probe further downward into the port to the appropriate depth indicated by the A, B, C, and D depth markers on the probe.

In the H-ESI mode, insert the probe to a depth of B, C, or D. For high solvent flow rates, adjust the probe depth so that the nozzle is farther away from the ion interface (depth C or D). Conversely, for low solvent flow rates, adjust the probe depth so that the nozzle is closer to the ion interface (depth B or C).

5. Lock the probe in place by turning the probe locking knob (see Figure 46 on page 48) clockwise until you feel resistance.

6. Connect the nitrogen gas lines to the HESI-II probe as follows:

- Connect the sheath gas fitting (blue) to the sheath gas inlet (S).
- Connect the auxiliary gas fitting (green) to the auxiliary gas inlet (A).

7. Unplug the vaporizer cable connector from the interlock socket and connect it to the vaporizer cable connector socket on the probe. To align the connector pins with the socket, align the red dot on the connector with the red dot on the socket. Figure 44 on page 46 shows the red alignment dots on the connector and the socket.

8. Connect the 8 kV cable connector to the 8 kV cable connector socket on the probe. Tighten the locking ring (see Figure 41 on page 44) on the 8 kV cable connector.

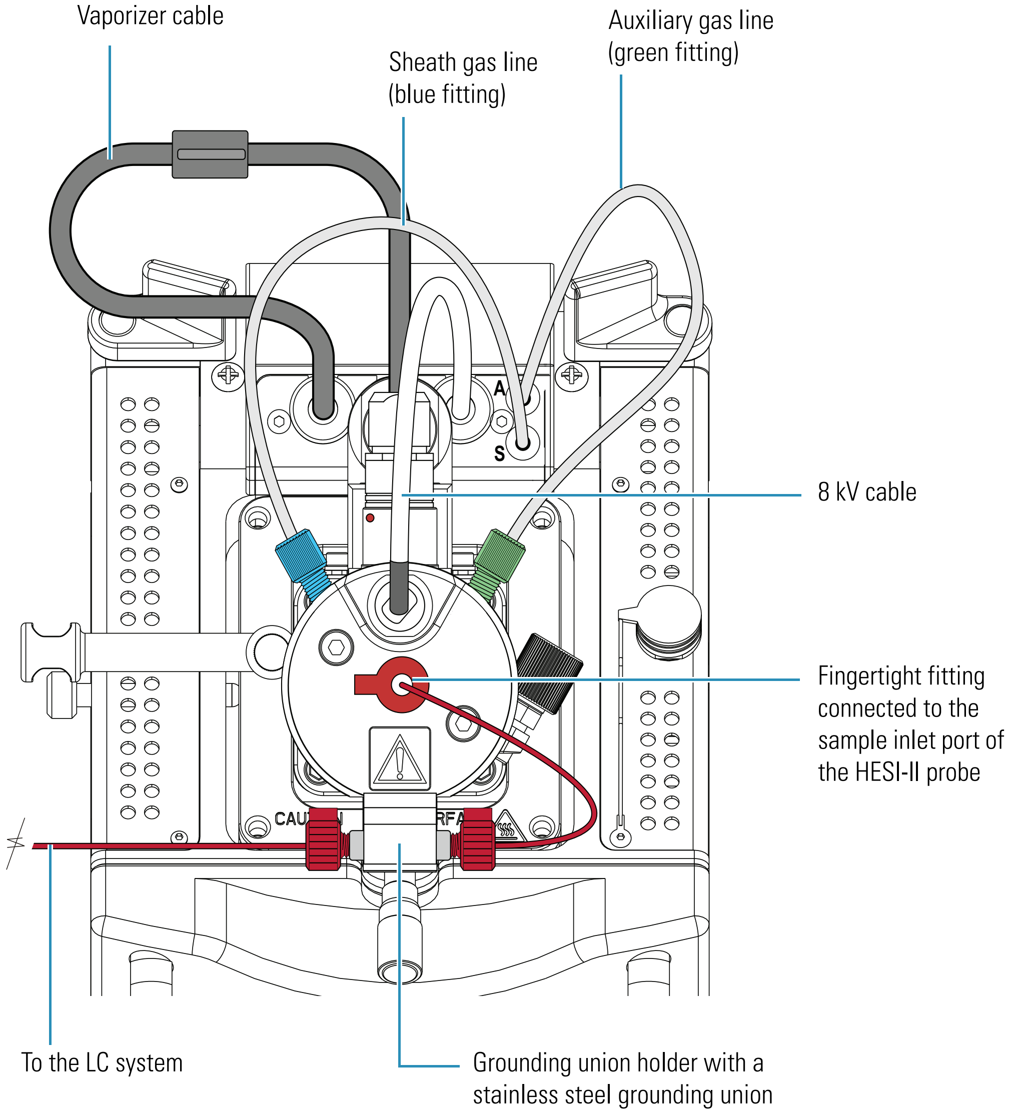
9. Ensure that the grounding union (stainless steel ZDV fitting) is seated in the grounding union holder on the probe.

10. Using two fingertight fittings, connect a short length of red PEEK tubing to the right side of the grounding union and to the probe sample inlet.

11. Using two fingertight fittings, connect a length of red PEEK tubing to the LC outlet and to the left side of the grounding union.

Figure 49 shows the HESI-II probe installed in the Ion Max API source.

Figure 49. HESI-II probe installed in the Ion Max API source housing



Maintaining the HESI-II Probe

The HESI-II probe requires minimum maintenance. If the metal needle sample tube is plugged, you must replace it. Replacing the metal needle requires a partial disassembly of the probe.

This section contains the following maintenance procedures:

- Flushing the Sample Transfer Line and Sample Tube
- Replacing the Needle Insert

Note For best results, flush the HESI-II probe at the end of each working day, using a 50:50 HPLC-grade methanol/distilled water solution from the LC pump through the HESI-II probe.

IMPORTANT For best results, avoid operating the HESI-II probe at elevated temperatures without solvent flow. Allowing the HESI-II probe to run dry at elevated temperatures can cause blockage of the replaceable metal needle.

IMPORTANT For best results, wear clean gloves when you handle HESI-II probe components.

CAUTION **AVOID BURNS.** At operating temperatures, the vaporizer can severely burn you. The vaporizer typically operates between 350 and 450 °C. Always allow the heated vaporizer to cool to room temperature (for approximately 20 min) before you remove or touch the HESI-II probe.

Flushing the Sample Transfer Line and Sample Tube

For best results flush the sample transfer line, sample tube, and HESI-II probe for 15 minutes at the end of each working day (or more often if you suspect they are contaminated). Use a 50:50 methanol/distilled water solution from the LC system through the API source.
After 15 minutes, turn off the flow of liquid from the LC to the API source, but keep the API source on (including the sheath gas and auxiliary gas) for an additional 5 minutes. Refer to the daily operations chapter in the hardware manual for your mass spectrometer.

Replacing the Needle Insert

If the metal needle is plugged, you can replace the needle insert. The needle insert is assembled at the factory and consists of an adjustable union, a needle guide fitting, a ferrule, an O-ring, an ESI needle, and a metal needle (see Figure 50). The ferrule is swaged onto the ESI needle. Factory adjusted, the metal needle protrudes 0.5 mm from the end of the ESI needle (see Figure 51).

Figure 50. Needle insert components

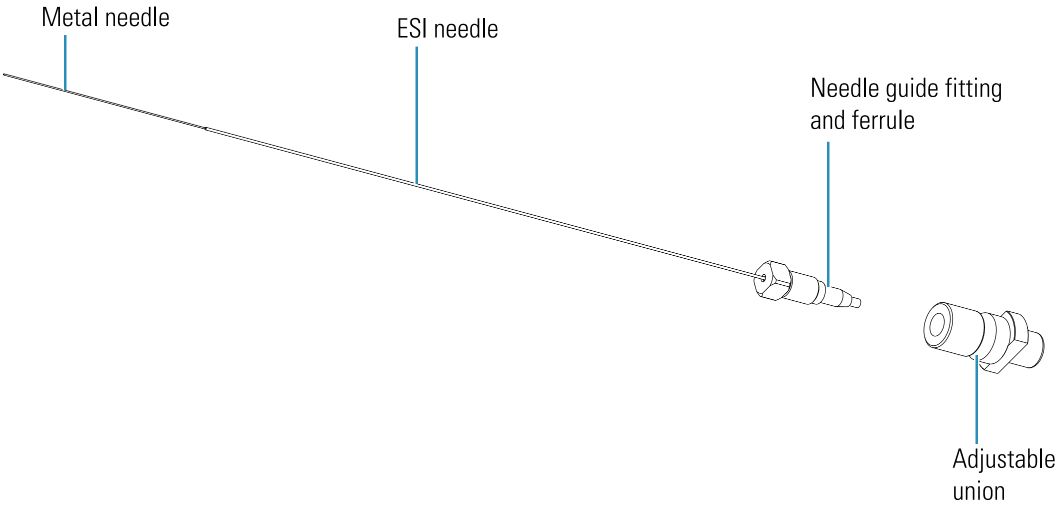
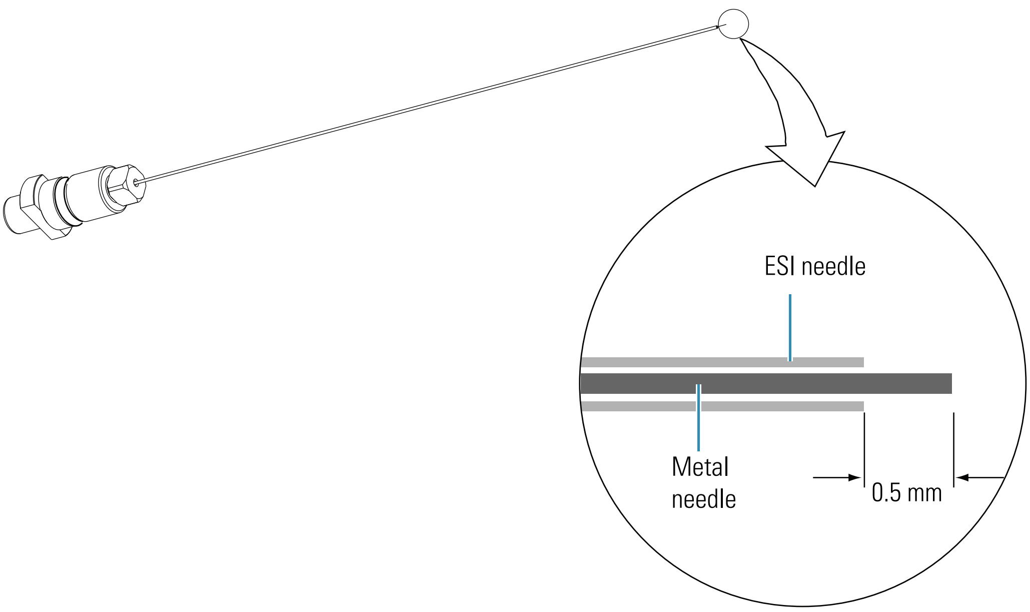


Figure 51. Metal needle insert assembly with an enlarged view of the stainless steel needle tip



To support flow rates from 5 to 2000 µL/minute, Thermo Fisher Scientific provides two metal needle inserts for the HESI-II probe. The difference between the two inserts is the size of the metal needle and supporting ferrule (Table 4).

Table 4. Metal needle inserts

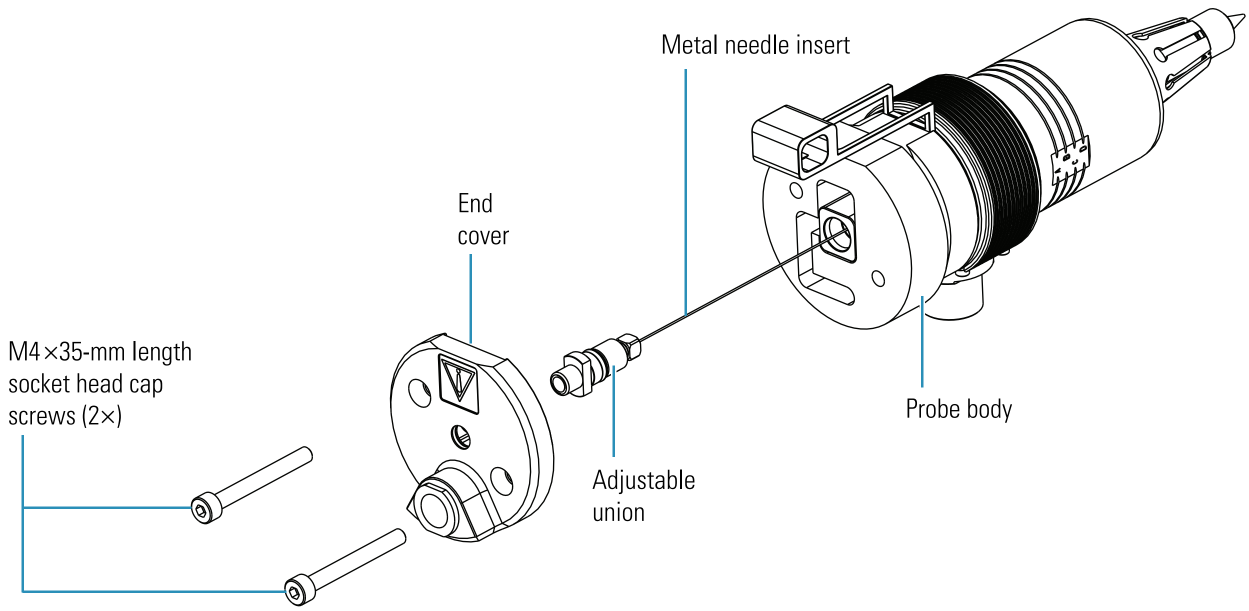
Metal needle insert description	Metal needle	Ferrule	Flow rate range	Part number
32-gauge needle insert, HESI-II probe	0.004-in. ID 0.009-in. OD	0.4 mm thru-hole	5 to 2000 µL/min	70005-60155
34-gauge needle insert, HESI-II probe	0.003-in. ID 0.007-in. OD	0.2 mm thru-hole	1 to 10 µL/min	70005-60180

To replace the metal needle insert, you must have a 3-mm (7/64-in.) hex wrench or ball driver.

❖ To replace the metal needle insert

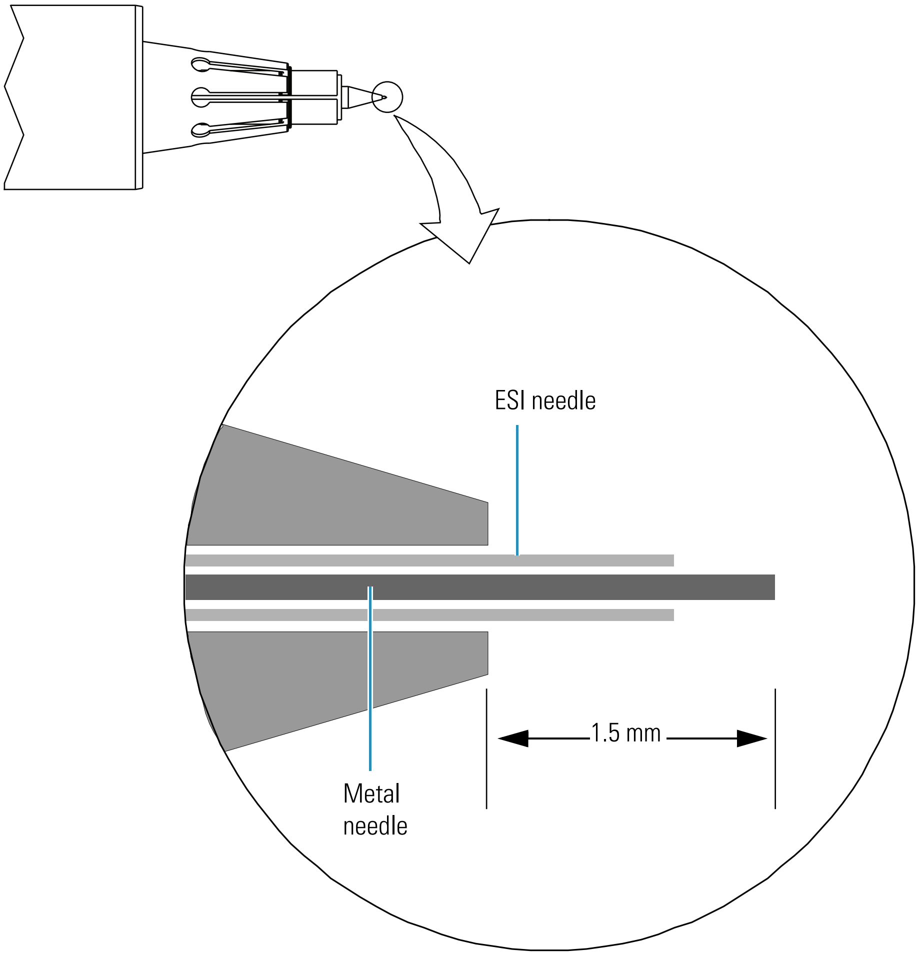
1. Remove the HESI-II probe from the Ion Max API source (see “Removing the HESI-II Probe” on page 43).
2. Unscrew the fingertight fitting from the sample inlet port (see Figure 49 on page 50).
3. Remove the metal needle insert from the probe as follows (see Figure 52):
   a. Using a 3-mm (7/64-in.) hex wrench or ball driver, remove the two M4×35-mm length, socket head cap screws.
   b. Pull the end cover off of the probe.
   c. Unscrew the metal needle insert, and then pull it out of the probe body.

Figure 52. Exploded view of the HESI-II probe



4. Insert a new metal needle insert into the probe body.
5. Hand tighten the adjustable union fitting until the tip of the needle insert protrudes from the probe nozzle by 1.5 mm (see Figure 53).

Figure 53. Enlarged view of the probe nozzle



6. Position the end cover on the probe body.
7. Insert the two M4×35-mm length, socket head cap screws into the end cover, and then tighten them with a 3-mm (7/64-in.) hex wrench or ball driver.
8. Reinstall the H-ESI probe (see "Installing the HESI-II Probe" on page 47).

Replaceable Parts for the HESI-II Probe

Use the following part numbers when yo need to order the replaceable and consumable parts for the HESI-II probe.

You can order these parts directly from Thermo Fisher Scientific.

HESI-II Probe Kit . . . . **OPTON 20037**
High-flow needle insert assembly **OPTON-53010**
Low-flow needle insert assembly **OPTON-53011**

You can order these parts from Upchurch Scientific; however, these part numbers are subject to change.

Fitting, fingertight, one-piece, for 1/16-in. OD high pressure tubing F-120
Fitting, fingertight with one wing, two-piece,
red PEEK, for 1/16-in. OD high pressure tubing F-200
Union, stainless steel, for 1/16-in. OD high pressure tubing U-435

4

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

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

In APCI, ions are produced and introduced into the mass spectrometer 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 54 shows the APCI process for a positive adduct ion formation.

Because APCI is a gas phase ionization technique, 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

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

- Secondary ion formation

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

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

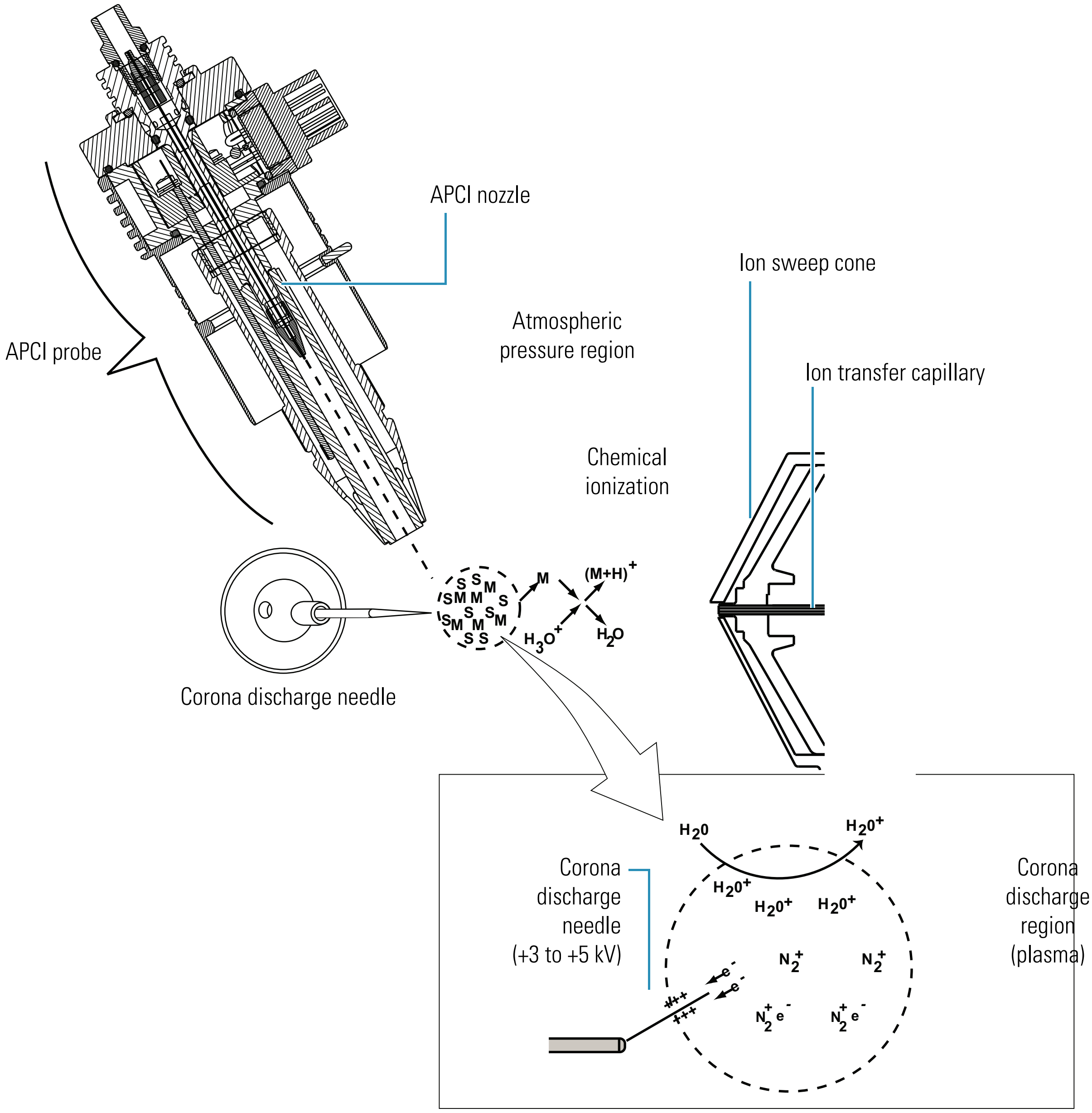
- Proton transfer

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

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 54. 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 and accommodates liquid flows of 100 µL/min to 2 mL/min without splitting.

The APCI probe includes the APCI sample tube, nozzle, sheath gas and auxiliary gas plumbing, and vaporizer (see Figure 55 and Figure 56).

Figure 55. Cross-sectional view of the APCI probe

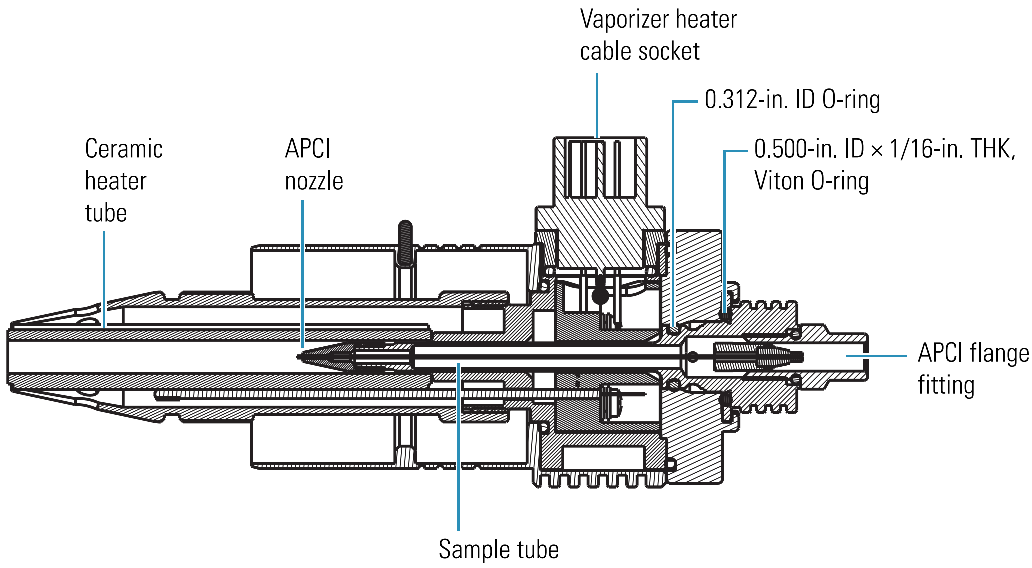
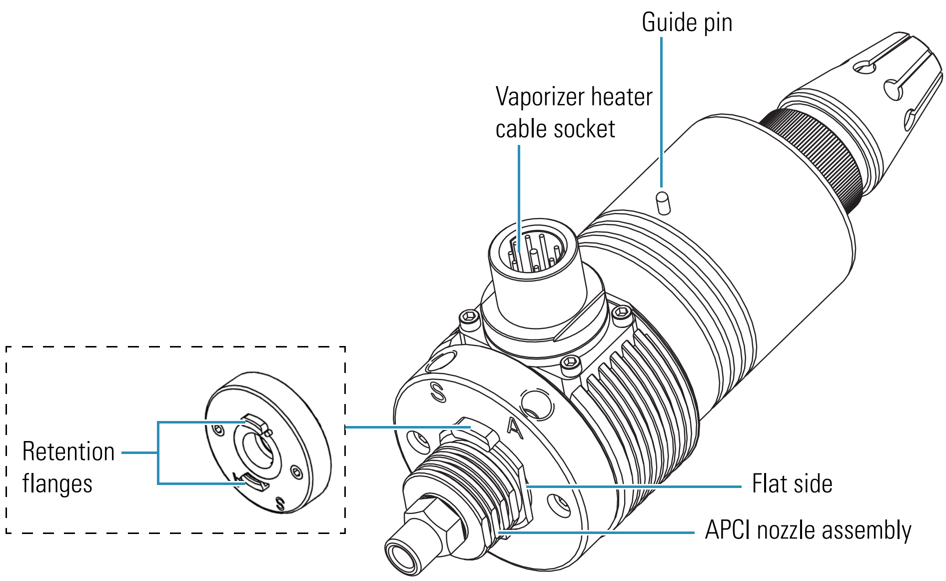


Figure 56. Exterior view of the APCI probe



Sample and solvent enter the APCI nozzle through the sample tube. The sample tube is a short section of 0.15-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 µA. (A typical value of the corona discharge current is 5 µA.) 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 and the Corona Needle

This section describes how to remove the APCI probe and the corona needle from the API source housing.

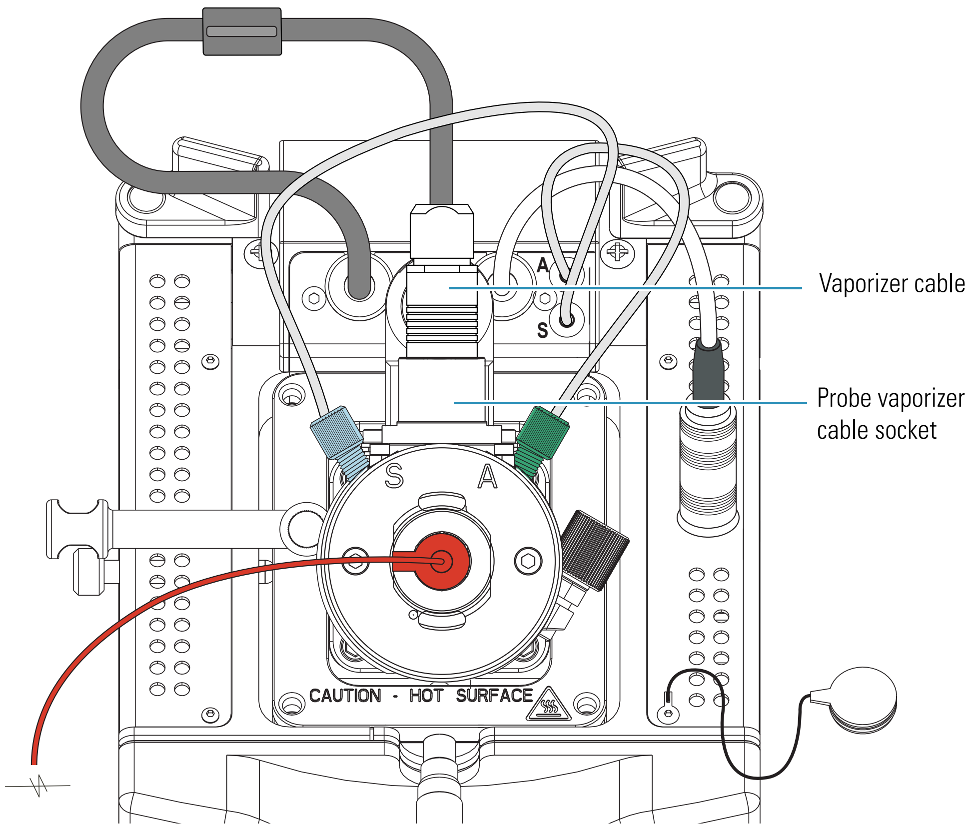
❖ To remove the APCI probe and the corona needle

1. Place the mass spectrometer in Standby mode. For information on placing the mass spectrometer in Standby mode, refer to the Help available from the instrument control software.
2. If necessary, turn off the solvent flow from the optional LC pump.
3. Allow time for the API source housing to cool to ambient room temperature.

CAUTION AVOID BURNS. Allow heated surfaces to cool. Both the ion source housing and the APCI probe can become too hot to touch when the mass spectrometer is operated in the APCI mode.

4. Unplug the vaporizer cable from the probe vaporizer cable socket (see Figure 57).

Figure 57. APCI probe connections



5. Disconnect the auxiliary gas fitting (green) from the auxiliary gas inlet (A) on the probe.
6. Disconnect the sheath gas fitting (blue) from the sheath gas inlet (S) on the probe.
7. Connect the vaporizer cable to the interlock socket on the interlock block. To align the socket pins, align the red dot on the vaporizer cable connector with the red dot on the interlock socket. Figure 60 on page 65 shows the vaporizer cable connected to the interlock socket.

8. Unlock the probe locking ring by turning the probe locking knob counterclockwise.
9. To remove the probe from the probe port in the API source housing:
   a. Slowly pull the probe out of the port until you feel the resistance caused by the probe guide pin meeting the interlock block.
   b. Turn the probe counterclockwise until the guide pin is free of the interlock block.
   c. When the guide pin is free of the interlock block, pull the probe out of the port.
10. Store the APCI probe in its original shipping container.
11. If you are changing the ionization mode from the APCI mode to the ESI or H-ESI modes, do the following:
    a. Remove the 8 kV cable from the high voltage socket on the source housing:
       i. Unlock the cable connector by twisting the locking ring counterclockwise.
       ii. Unplug the 8 kV cable from the corona needle high voltage socket.
       iii. Insert the high voltage socket cover into the high voltage socket on the source housing.

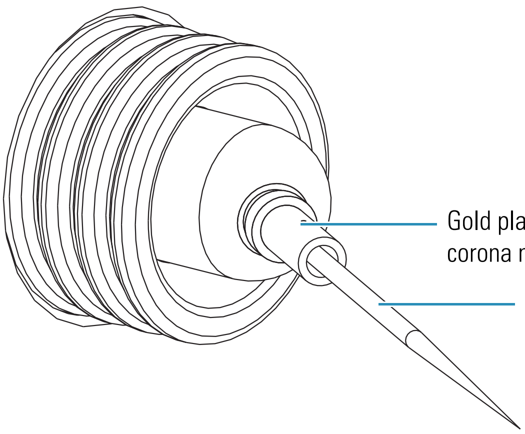
CAUTION AVOID INJURY. The corona discharge needle is very sharp and can puncture your skin. Handle it with care.

    b. Remove the corona needle:
       i. Remove the source housing (see "Removing the API Source Housing" on page 6).

       The corona needle is in the corona assembly inside of the ion source housing across from the window.

       ii. Using pliers, grasp the needle and pull it straight out of the corona needle contact (see Figure 58).

Figure 58. Corona needle, view from inside the Ion Max housing



    c. Remount the ion source housing or place it in a safe location for temporary storage.
12. Store the APCI probe and the corona needle in the original shipping container.

Installing the APCI Probe and the Corona Needle

To operate the system in the APCI mode, you must install the APCI probe and the corona needle.

❖ To install the APCI probe and the corona needle

1. Place the mass spectrometer in Standby mode.

   Note When the mass spectrometer is in Standby, the high voltage and nitrogen gas are off.

2. Install the corona needle as follows:

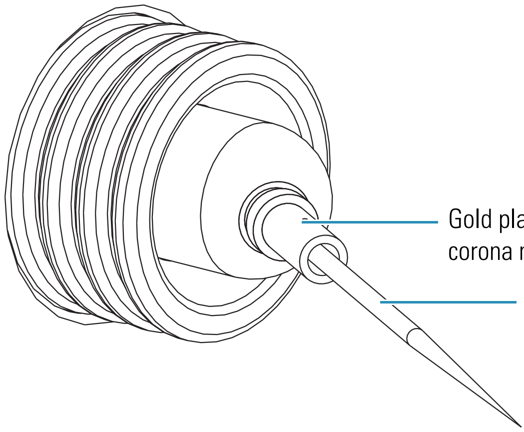
   a. Unlock the source housing by turning the ion source locking levers 90 degrees.

   b. Remove the source housing by pulling the housing straight off of the ion source assembly.

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

   c. Using pliers, grasp the corona needle and push it straight into the corona needle contact (see Figure 59).

Figure 59. Corona needle, view from inside the Ion 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.

3. Connect the 8 kV cable to the high voltage socket on the source housing as follows:

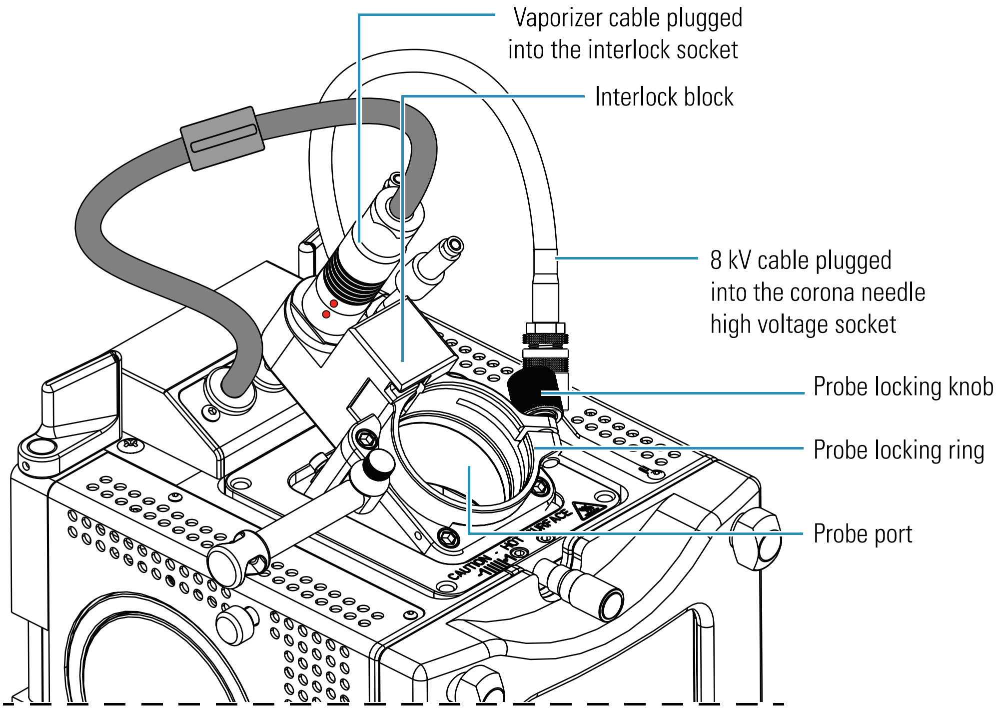
   a. Remove the cover from the high voltage socket on the right side of the top of the ion source housing.

   b. Plug the 8 kV cable connector into the high voltage socket on the source housing (see Figure 60.

   c. Lock the cable by twisting the locking ring clockwise.

4. Turn the probe locking knob counterclockwise until the probe locking ring is opened to its widest position (see Figure 60).

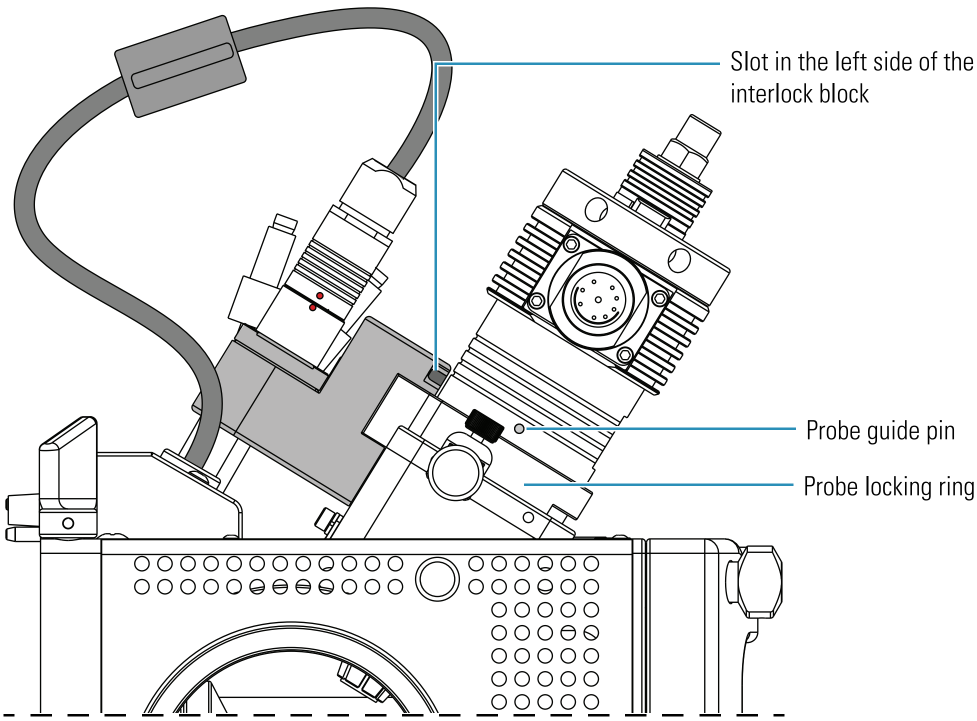
Figure 60. Ion Max API source housing



5. To seat the probe in the probe port in the API source housing:

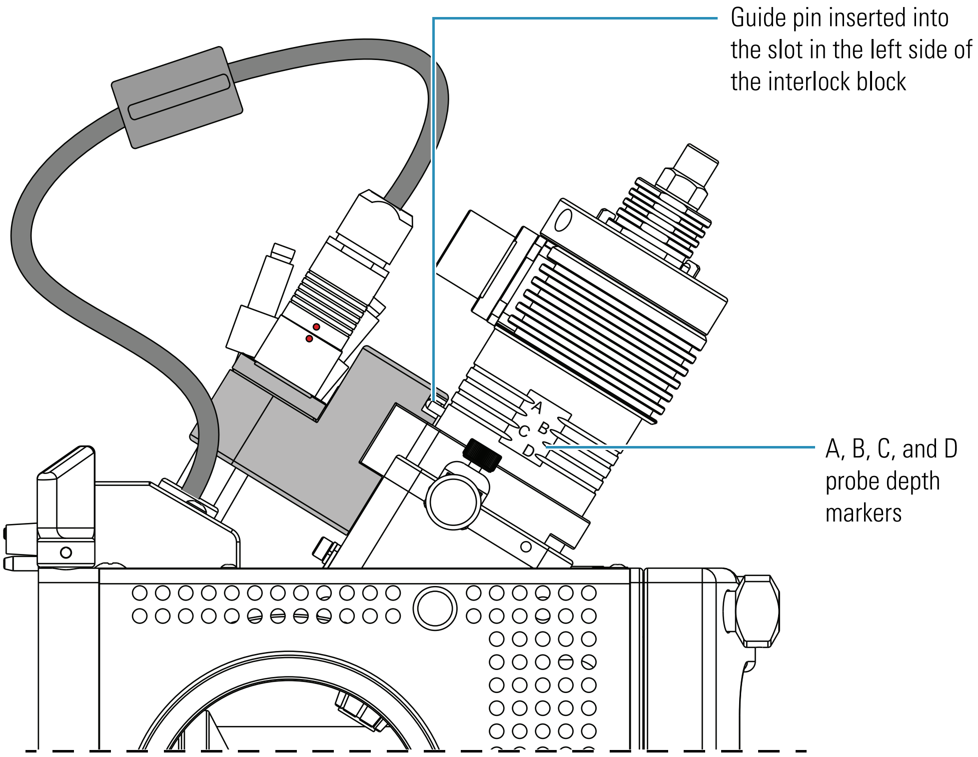
   a. Holding the probe with the nozzle facing downward and the guide pin facing toward the left, slowly insert the probe into the port until the guide pin meets the locking ring on the API source housing (see Figure 61).

   Figure 61. View of the slot in the left side of the interlock block



b. Pull the probe slightly upward until the guide pin is level with the slot on the left side of the interlock block. Then turn the probe clockwise until the guide pin meets resistance from the interlock block (see Figure 62).

Figure 62. View of the probe guide pin inserted into slot on the left side of the interlock block



c. Push the probe further downward into the port to the appropriate depth indicated by the A, B, C, and D depth markers on the probe.

Insert the probe to a depth of B, C, or D. For high solvent flow rates, adjust the probe depth so that the nozzle is farther away from the ion interface (depth C or D). Conversely, for low solvent flow rates, adjust the probe depth so that the nozzle is closer to the ion interface (depth B or C).

6. Lock the probe in place by turning the probe locking knob (see Figure 60) clockwise until you feel resistance.

7. Connect the nitrogen gas lines to the APCI probe as follows:

   a. Connect the sheath gas fitting (blue) to the sheath gas inlet (S).

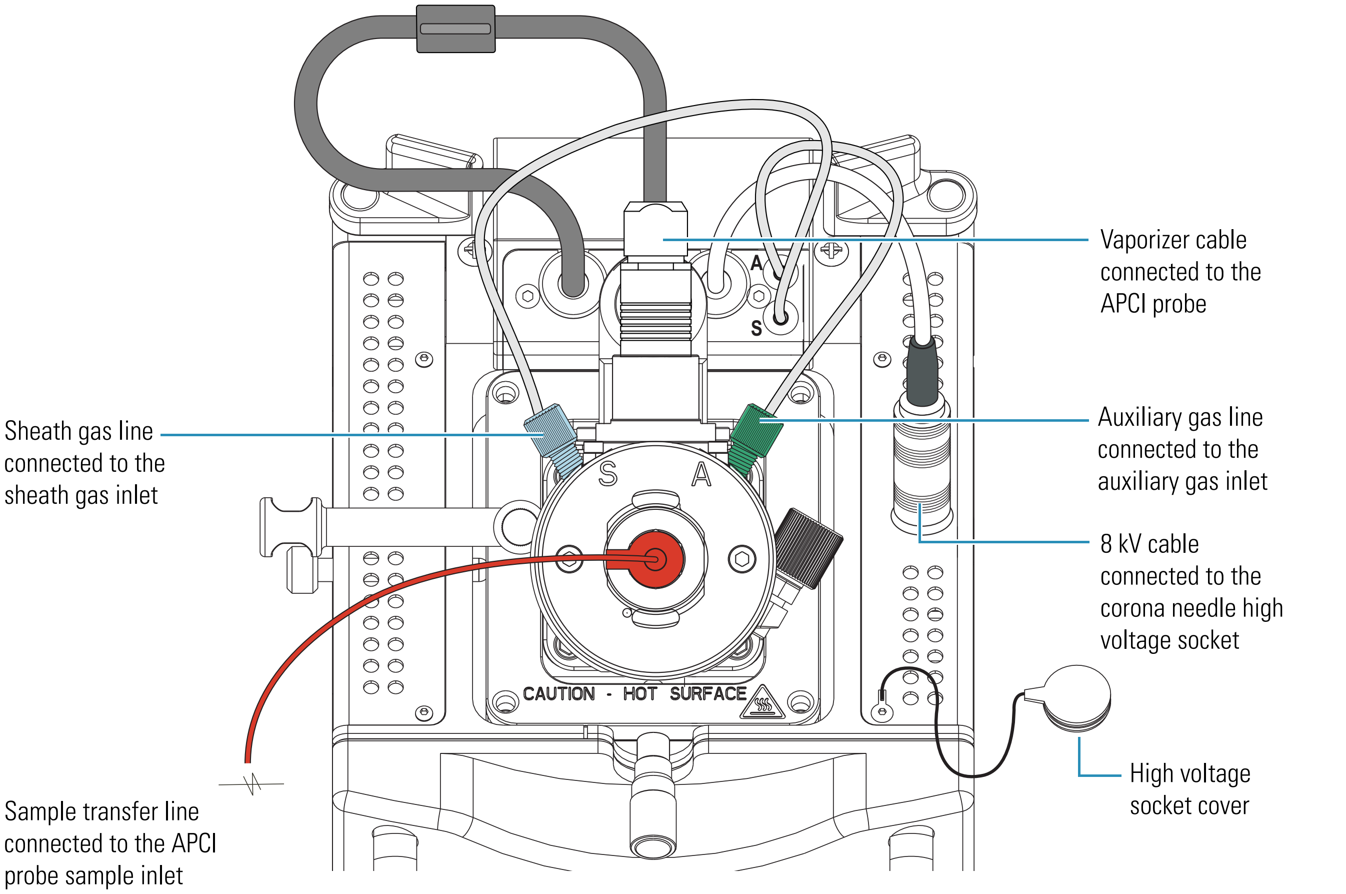
   b. Connect the auxiliary gas fitting (green) to the auxiliary gas inlet (A).

8. Unplug the vaporizer cable connector from the interlock socket and connect it to the vaporizer cable connector socket on the APCI probe. To align the connector pins with the socket, align the red dot on the connector with the red dot on the socket.

9. Connect the sample transfer line to the probe sample inlet.

The APCI source is now properly installed on the mass spectrometer (see Figure 63).

Figure 63. APCI probe connections



Maintaining the APCI Probe

The APCI probe requires a minimum of maintenance. Occasionally, you must replace the APCI sample tube (150-µm ID×390-µm OD) fused-silica tubing) and clean the APCI nozzle.

Figure 55 and Figure 56 on page 60 show the major components of the APCI probe.

Note For best results, flush the APCI probe at the end of each work day by pumping a 50:50 methanol/water solution through the APCI source.

IMPORTANT Wear clean gloves when you handle APCI probe components.

To maintain the APCI probe, follow these procedures:

- Removing the APCI Nozzle
- Cleaning the APCI Nozzle
- Removing the APCI Sample Tube from the APCI Nozzle
- Installing the APCI Sample Tube
- Reassembling the APCI Probe

Removing the APCI Nozzle

❖ To remove the APCI nozzle from the APCI probe

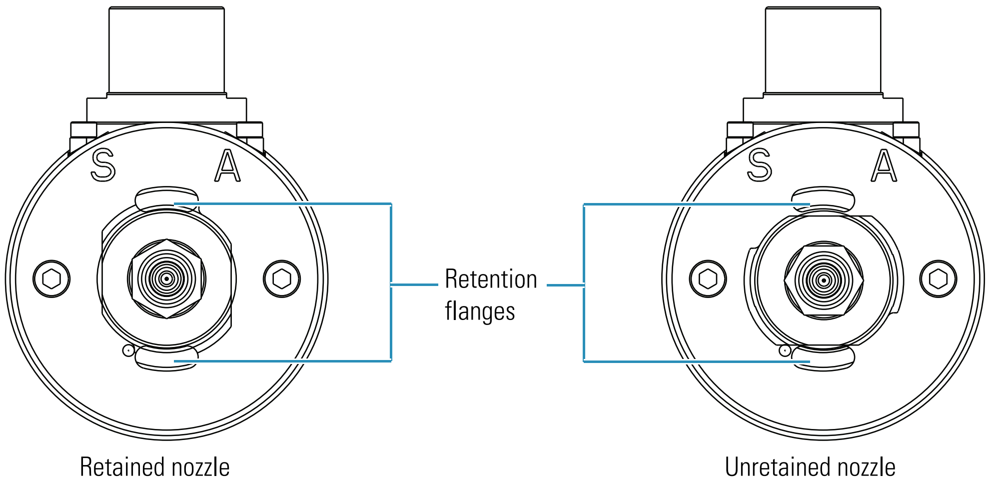
1. Place the instrument in Standby mode.

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.**

3. Allow the heated components to cool to room temperature.
4. While holding the APCI probe body with one hand, grasp the head of the APCI nozzle assembly and rotate the head of the nozzle assembly counterclockwise until the flat sides of the head are facing towards the retention flanges (see Figure 56 on page 60 and Figure 64).

Figure 64. View of the sample inlet end of the APCI probe



5. Carefully pull the nozzle assembly straight out the back of the APCI probe.

CAUTION If the sample tube hits the sides of the vaporizer, it can break. To prevent breakage, carefully pull the APCI nozzle straight back from the APCI probe.

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

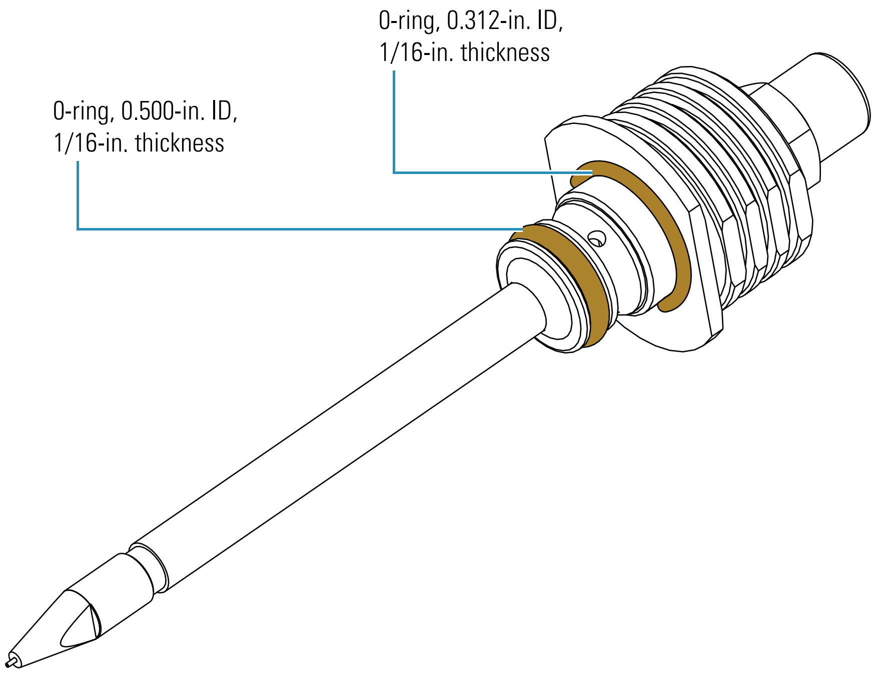
Cleaning the APCI Nozzle

❖ **To clean the APCI nozzle**

1. Remove the APCI nozzle from the probe body (see “Removing the APCI Nozzle” on page 68).
2. Check the condition of the O-rings on the APCI nozzle (see Figure 65).

Part	Part number
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

Figure 65. 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 removed while cleaning.

5. Do one of the following:

   - If you do not want to replace the APCI sample tube, reinstall the APCI nozzle (see "Reassembling the APCI Probe" on page 73).

   - If you want to replace the sample tube, go to the next procedure "Removing the APCI Sample Tube from the APCI Nozzle."

Removing the APCI Sample Tube from the APCI Nozzle

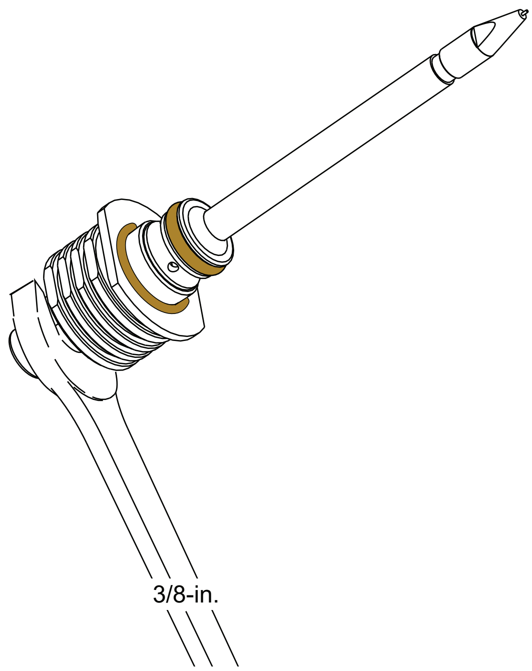
The sample tube for the APCI probe is an 8.6 cm length of 390 µm OD fused-silica tubing.

❖ **To remove the APCI sample tube from the APCI Nozzle**

1. If you have not already done so, remove the APCI nozzle from the APCI probe (see "Removing the APCI Nozzle" on page 68).

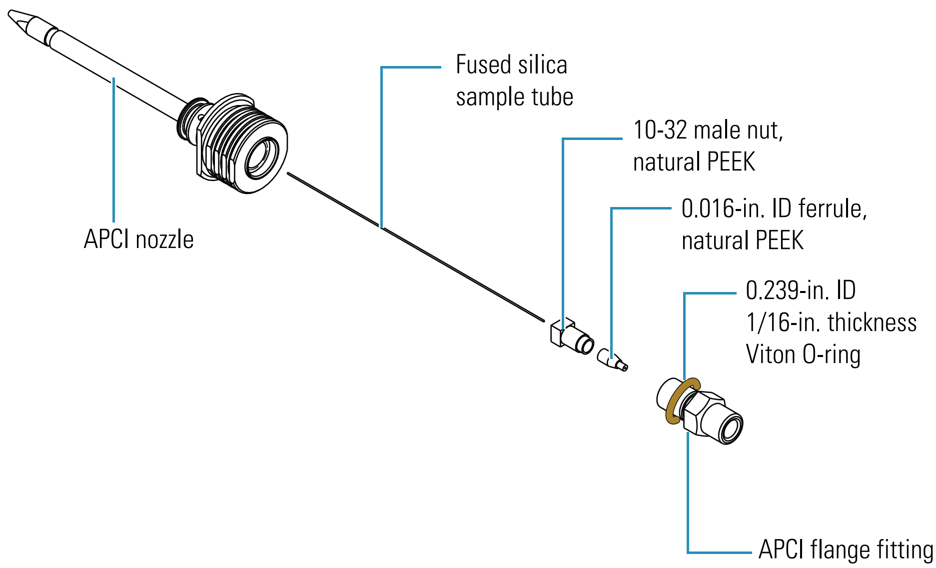
2. Using a 3/8-in. open-end wrench (see Figure 66), loosen the APCI flange fitting, and then pull the sample inlet fitting, exit nut, PEEK ferrule, and sample tube assembly from the APCI nozzle (see Figure 67).

Figure 66. Loosening the APCI flange fitting



3. Remove the fused-silica sample tube, nut, and ferrule assembly from the APCI flange fitting (see Figure 67).

Figure 67. APCI sample tube connection



4. Discard the used fused-silica sample tube.

Installing the APCI Sample Tube

The APCI sample tube is an 8.6 cm length of 150 µm ID × 390 µm OD fused-silica tubing.

❖ To install a new APCI sample tube

1. Check the condition of the 0.239-in. ID O-ring (see Figure 68) on the APCI flange fitting. Replace it if necessary.

Part	Part number
O-ring, 0.239-in. ID × 1/16-in. THK	00107-04000

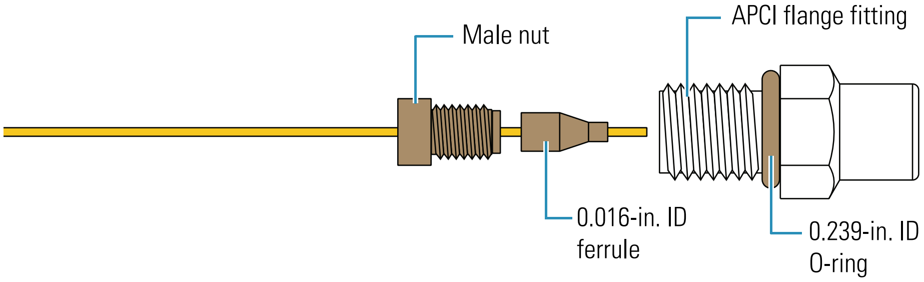
2. Using a fused-silica cutting tool, cut a piece of fused-silica tubing to a length of approximately 13 cm (5 inch). Ensure that you make square cuts to the ends of the fused-silica tubing.

3. Connect the fused-silica tubing to the APCI flange fitting:

   a. Slide the nut and the 0.016-in. ID ferrule onto the fused-silica tubing (see Figure 68).

   b. While pressing the fused-silica tubing into the externally threaded end of the APCI flange fitting, fingertighten the two-piece fitting (see Figure 68).

   Figure 68. Connecting the fused-silica tubing to the APCI flange fitting

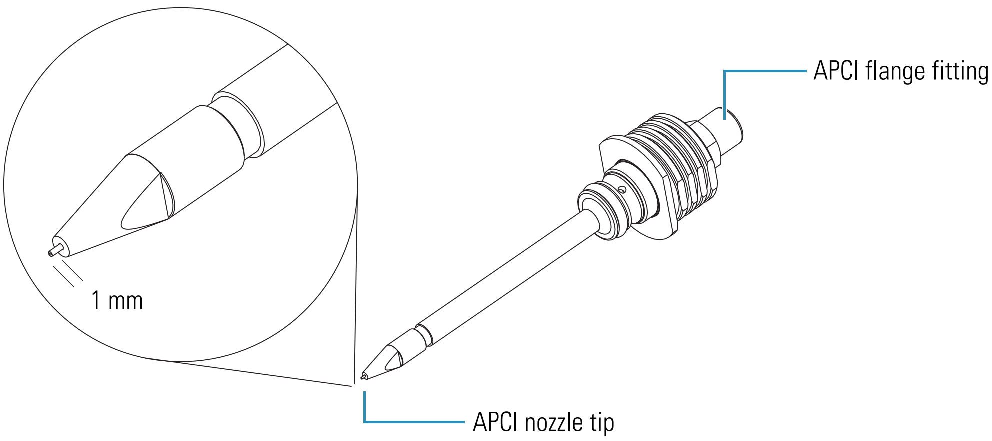


4. Carefully insert the free end of the fused-silica tubing into the back of the APCI nozzle and out the nozzle tip. Then fingertighten the APCI flange fitting.

5. Using a 3/8-in. open-end wrench, tighten the APCI flange fitting an additional quarter turn (see Figure 66 on page 71).

6. Using a fused-silica cutting tool, cut the fused-silica sample tube so that approximately 1 mm protrudes past the tip of the APCI nozzle (see Figure 69).

Figure 69. Proper position of the exit end of the APCI sample tube

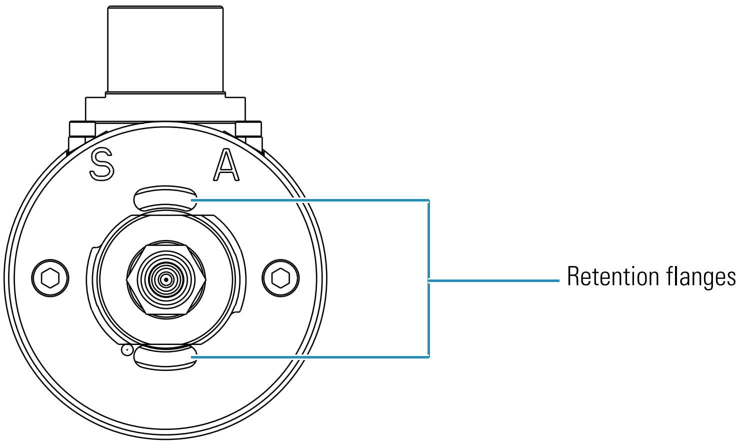


Reassembling the APCI Probe

❖ **To reassemble the APCI probe**

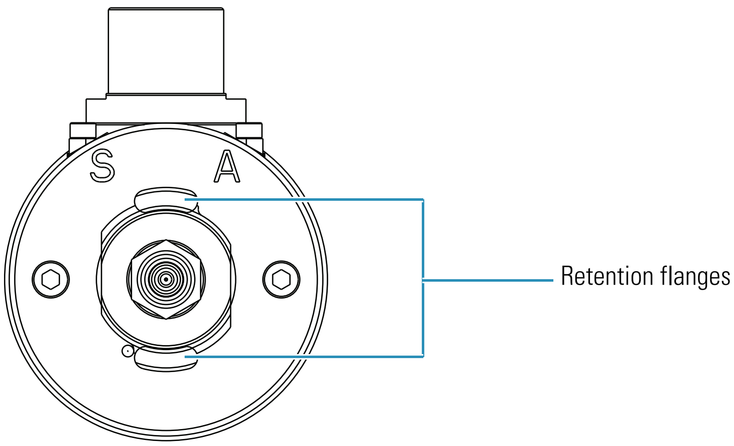
1. With one hand holding the APCI probe body to keep the probe from turning, carefully insert the APCI nozzle into the APCI probe.
2. With the flat sides of the APCI nozzle head facing the retention flanges on the probe body, seat the nozzle head into the APCI probe. See Figure 70.

Figure 70. View of the unsecured APCI nozzle seated in the APCI probe



3. To secure the APCI nozzle in the probe, rotate the head of the nozzle 90 degrees clockwise to secure the rounded sides of the nozzle head in the retention flanges.

Figure 71. View of the APCI nozzle secured by the retention flanges



To reinstall the probe in the Ion Max API source housing, see "Installing the APCI Probe and the Corona Needle" on page 64.

Replaceable Parts for the APCI Probe

Figure 72 shows the APCI probe, and Figure 73 shows the APCI probe nozzle assembly.

APCI probe OPTON-20012

APCI probe nozzle assembly. 97055-60089
Ferrule, 0.016-in. ID, natural PEEK 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, natural PEEK. 70005-20220
Fitting, APCI flange 70005-20250
Nozzle, APCI probe 97055-20221

Figure 72. APCI Probe Assembly (OPTON-20012)

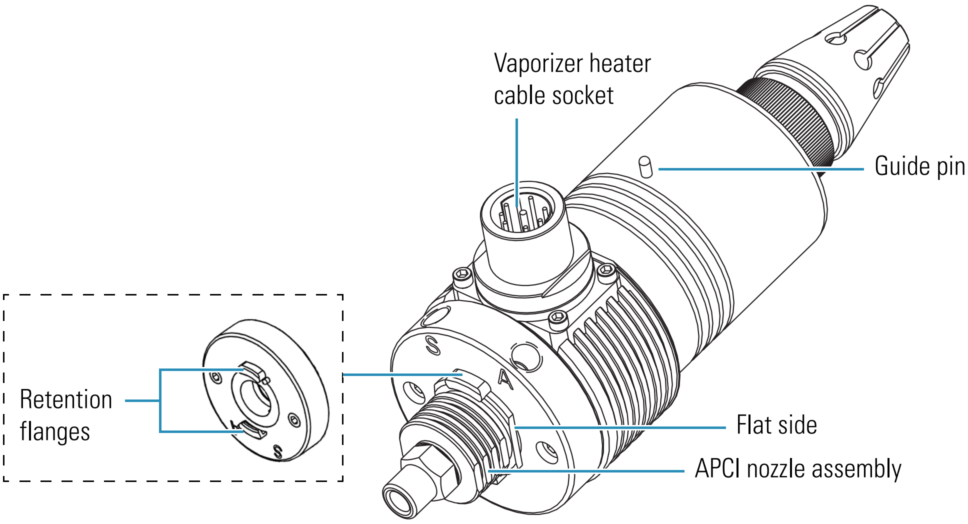
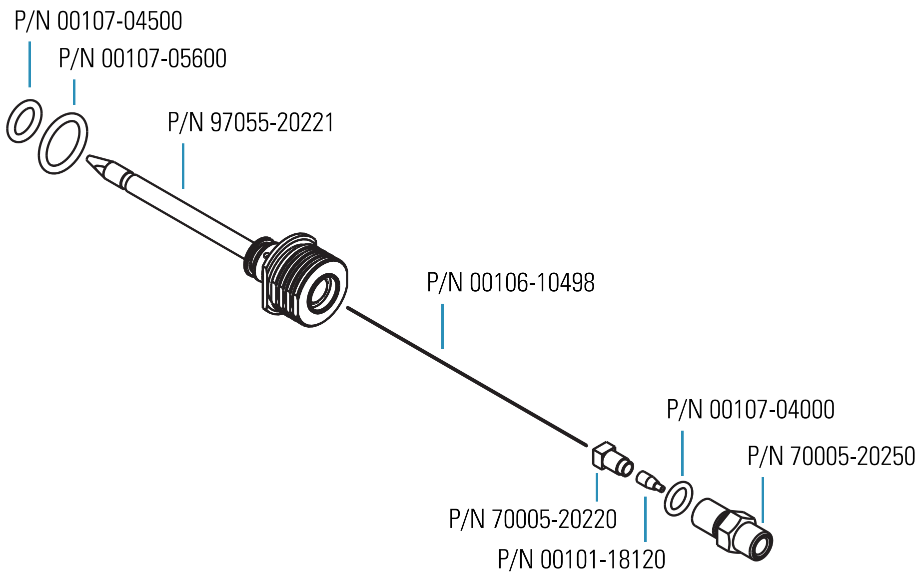


Figure 73. APCI probe nozzle assembly (P/N 97055-60089)



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