Tips on Tips - Online PicoTips®

Thank you for ordering from New Objective's line of PicoTip® emitters for online nanospray. Consisting of SilicaTips™, TaperTips™, and PicoFrits™, they represent the most advanced precision emitters available for nanospray.

Given the wide variety of electrospray ionization (ESI) sources produced by different manufacturers, the exact implementation of PicoTip emitters on your system may affect utility and performance. This "tip sheet" gives a few pointers on the successful use of PicoTips. Please observe all manufacturer safety recommendations and read the safety statement at the end of this document.

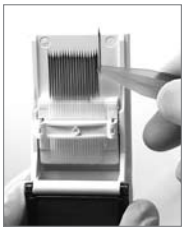
Unpacking and handling your PicoTips®

NOTE: Please wear ANSI-approved safety glasses when handling PicoTip® emitters.

Coated PicoTips® have a special enhanced conductive multilayer coating (U.S. Patent 5,788,166) that provides for excellent electrochemical stability and durability against ESI solvent exposure and arcing. Coated tips should be handled with care, as mechanical abrasion can remove the coatings. No attempt should ever be made to handle emitters with bare hands. Non-serrated forceps are recommended for handling all varieties of emitters. A rubber-tipped disposable pair is included with each package of PicoTips. New Objective carries an accessory kit containing a complete assortment of high-quality tools (cleaver, special forceps, ruler, etc.) needed to properly handle PicoTips. Please see our web site for a full description of our accessory kit (stock number TIP-KIT).

Open package by grasping lid sides

Inside the box, the PicoTips are held in place by a padded pressure bar. When ready to use, pull the PicoTip out from behind the holding bar with a pair of forceps, taking care not to touch the tip or scrape off the conductive coating, as the coating can be ruined by improper or rough handling. Grasp the shaft towards the tip end of the emitter and pull the emitter directly through the holding bar being particularly careful not to bend the emitter. Bending may cause damage to the emitter. The end of the tip must not make physical contact with any surface.



Pull emitter directly up to remove

Cleaving fused silica

Proper cleaving of fused-silica tubing is a critical but often overlooked operation in the preparation of PicoTip® emitters for use. A flat, smooth cleave is critical for maintaining low dead-volume connections with other sections of fused-silica tubing. It is also critical that cleaving does not generate flow-stopping particulate matter. Cleaving is best accomplished with a high-quality diamond chip or sapphire cleaving tool. New Objective's 1 mm wide diamond-blade cleaving tool has been selected to provide a consistent, flat cleave with a minimum of particulate generation. Inexpensive carbide scribing tools are not recommended since they generally result in poor-quality (i.e., ragged) cleaved end faces that generate many fine particles.

1) Place the tubing to be cut on a flat, clean surface and position the cleaving tool perpendicular to the tubing surface, as shown in Figure 1. The long axis of the blade should be perpendicular to the tubing bore.
2) Press down gently; DO NOT use a sawing motion when pressing the blade. You only need to nick the surface of the polyimide coating. Be careful not to force the blade through the tubing, which will generate a ragged end and many particles.

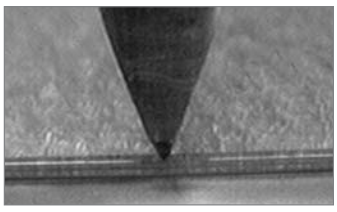


FIGURE 1 Cleaving tool in proper position

3) Pull gently on the tubing along its axis; it should easily separate at the point of contact. If it does not, repeat the procedure with a little more force.

Inspection of the distal end of the tip for particle contamination with a light microscope at 100x magnification is highly recommended.

Mounting PicoTips®

Fused-silica PicoTips® can be easily coupled to the "outside world" using any variety of zero or low dead volume unions available from a number of manufacturers. We recommend the use of Upchurch components (MicroTight® series) specifically designed for connecting different diameters of fused-silica tubing. Standard 1/16" HPLC hardware can be used for connecting fused-silica tubing by using 1/16" outer diameter PEEK™ sleeves with an inner diameter appropriate for the silica tubing outer diameter. Avoid the use of graphite ferrules, which can generate particulate matter when handled. New Objective offers a fittings kit (stock number FSFK-1) of assorted Upchurch components. Please see our catalog or Web site for a full description.

Coating style

While many users obtain suitable performance with our standard coated PicoTips® (-CE- in the stock number), some find that emitter performance can be compromised by excessive arcing during tuning due to an overvoltage condition. Although great improvements have been made with emitter coatings, constant arcing may still damage a coated tip, reducing or preventing stable operation. A good solution for those experiencing tuning problems is to use emitters with a distal coating (-D- in the stock number). The high-voltage contact is made through a junction-style contact inside the PEEK™ union. Since the distal coating is only applied to the non-tip end of the emitter, it is immune to arcing.

Spraying

CAUTION: Make certain that all electrical voltages are at ground potential before attempting to insert or remove a PicoTip® on your inlet system.

Before use, emitters should be properly and safely mounted on your ESI emitter mounting system. Make sure there is robust electrical contact between the conductive coating on the coated PicoTip® and your applied voltage "contact point." The final position of the tip should be 1-5 mm from the mass spectrometer inlet.

Applying high voltage

Starting from zero (ground) potential, slowly increase the voltage of the ESI system while monitoring ion or spray current, if your system provides a monitoring point. Although it varies greatly depending upon the exact geometry of your ESI system, spray should initiate at a potential difference between 1000 and 1500 volts. To optimize the applied voltage, monitor ion current while increasing the ESI potential(s). With most systems, a plateau in current is obtainable. The optimal set point is generally found at a voltage just before the onset of the plateau. Occasionally, and especially when spraying solutions that carry no organic solvent, the voltage required to initiate ESI current is quite high (greater than 2.0 kV); such a high voltage generally wastes sample. The voltage can usually be lowered after initiation of stable spray with no expense in ion current and a concurrent reduction in sample flow rate.

In general, the maximum voltage the tips can handle before a stable corona occurs is 3.0-3.5 kV. The fine wall structure of the tip cannot withstand prolonged arcing between the tip and inlet. Potentials that cause arcing should be avoiding when using PicoTips®. Excessive potentials result in higher required flow rates with little gain in total ion current.

Flow rates

Performance varies greatly from instrument to instrument and is highly dependent upon solution characteristics. The most significant influences on flow rate performance are solvent composition, electric field strength, and backing pressure. For operation at lower flow rates, choose smaller diameter PicoTips®. Consult our product literature or Web site for a listing of tip sizes. PicoTips can generally support stable ESI over a range of flow rates. For example, a 5 um tip can operate at rates from less than 25 nL/min to nearly 100 nL/min.

Approximate flow characteristics of the most common sizes of SilicaTips™ are:

Stock Number	Flow Range (nL/min)
FS360-75-30	300-1000
FS360-75-15	200-500
FS360-50-8	50-300
FS360-20-5	20-100

See our website (www.newobjective.com) for more information on flow ranges.

Troubleshooting

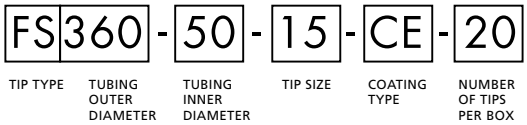
An erratic drop in ion intensity caused by gas bubbles in the system
Gas bubbles can wreak havoc with spray stability. Small bubbles can originate from trapped air pockets within a coupling union, electrolysis at a high-voltage contact, or dissolved gasses in the solvent. Bubbles can be minimized by making certain all fittings are sufficiently gas- and liquid-tight. Allow time for any residual gas to bleed out of the system. If air bubbles persist, try using a PicoTip® with a smaller inner diameter than that of the transfer line. This can create sufficient back-pressure and reduce or eliminate outgassing from solvents and electrolysis.

A droplet forms on the emitter tip
Droplets will form on the tip of an emitter when the applied voltage is not sufficient to maintain a stable spray. Droplets commonly form during the aqueous portion of an LC gradient, as the optimal potential is highest under aqueous conditions. Because most analytes do not elute under highly aqueous conditions, this should not degrade the performance of your system. If increasing the voltage does not help, it may mean that the flow rate is too high. Many customers report fewer problems with droplet formation when using smaller PicoTips®.

Ion signal is diminished
Ion signal can also be diminished with the deterioration of the conductive coating. As a rule of thumb, we suggest you change your tip when this occurs. If using a distal-coated PicoTip®, electrical contact can be reestablished by cleaving a small piece of fused silica from the back end of the tip. The cleaving removes the segment of tubing with an uneven coating and exposes a fresh piece of coating within the union to establish a spray.

Unable to see any flow through the emitter
The primary cause of tip failure is clogging due to particulates. Particles can be seen using a light microscope at 100x magnification. Inline filtration can effectively reduce clogging and extend emitter lifetime. We have obtained best results using HPLC-grade bottled water that has been distilled in glass.

Product Specifications



If you ordered the SilicaKit-360, you have been supplied with nine tips:

Order Number	Tubing OD	Tubing ID	Nominal Tip ID	Quantity
FS360-75-15	360 um	75 um	15 +/-1.5 um	3
FS360-50-8	360 um	50 um	8 +/-1 um	3
FS360-20-5	360 um	20 um	5 +/-1 um	3

Safety Precautions

CAUTION: Electrospray ionization involves the use of potentially lethal high-voltage electrical current. Observe all manufacturers' safety recommendations in the use of such equipment. No equipment modifications should be made except as authorized by the manufacturer in accordance with all safety requirements. Never use this product in defective, damaged, or faulty equipment. Serious personal injury or death could result.

Installation of such equipment should be performed by a qualified contractor in accordance with all applicable electrical codes. This product should be used only by experienced personnel.

Provide a safe workplace and all necessary safety equipment. Follow all safety recommendations of the equipment manufacturer(s). Inspect all equipment and ionization emitters carefully prior to use. Any damaged, chipped, or cracked emitters should not be used. Handling of fused-silica tubing and emitters can result in serious personal injury, including skin and eye injury. Use safety glasses or goggles meeting ANSI Z87.1-1989 requirements or the equivalent. Puncture- and chemical-resistant gloves should be worn at all times.