

## Using Pre-Cut SilicaTips™

### *Safety notes and precautions*

- 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.
- Make certain that all electrical voltages are at ground potential before attempting to insert or remove a SilicaTip on your inlet system.

### *Unpacking and handling SilicaTips™*

Any nanospray emitter should be handled with care, as mechanical abrasion can damage the tubing and any contact to the tip will damage the fragile tip opening. No attempt should ever be made to handle the tips with bare hands. The highest quality fine, non-serrated forceps are recommended.

Inside the box, the SilicaTips are held down by adhesion. When ready to use, pull the SilicaTip off with a pair of fine forceps. Always lift the emitter from the tip side first to avoid touching the tip to any surface.

**Pre-cut SilicaTips are ready for use out of the box and require no additional cleaving. The distal end is machine-cut and polished to ensure best possible butt-end connections are made.**

### *Mounting SilicaTips™*

SilicaTips® can be easily coupled using any variety of zero- or low-dead-volume unions available from a number of manufacturers, such as Upchurch components (MicroTight® series) which are 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.

### *Spraying*

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 SilicaTip® 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 avoided. Excessive potentials result in higher required flow rates with little gain in total ion current.

### *Flow rates*

Performance varies greatly 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 SilicaTips®. SilicaTips can generally support stable ESI over a range of flow rates. For example, a 5 µm 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:

<b>Emitter Specification (µm)</b>	<b>Flow Range (nL/min)</b>
360 OD, 75 ID, 30 tip opening	300-1000
360 OD, 75 ID, 15 tip opening	200-500
360 OD, 50 ID, 10 tip opening	50-300
360 OD, 20 ID, 10 tip opening	20-100

### *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 SilicaTip® 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 SilicaTips®.

#### **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. Best results are obtained using HPLC-grade bottled water that has been distilled in glass.

The information contained in this circular is believed reliable and accurate; however, nothing set forth herein constitutes a warranty or representation of any kind or nature. Given the variety of experimental conditions, New Objective cannot guarantee performance at a given flow rate for a given tip size. Your best guide to tip selection is empirical testing. A statement of product specifications, warranties, and safety information will be supplied upon request. CAUTION: Particular end-user applications for these products may be restricted by existing patents. Complying with any such patent is the sole responsibility of the user. SilicaTip is a trademark of New Objective, Inc. New Objective reserves the right to change product specifications without notice.  
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