Unique®

Optimization of the Unique® High-Flow Electrospray Source Parameters

Introduction

Electrospray Ionization (ESI) is a soft and efficient ionization technique that takes ions directly from the liquid into the gas phase. ESI is applicable to a large class of analytes: ionic or polar, and low or high molecular weight compounds. Only neutrals are not amenable to analysis. In ESI-MS, a dilute solution of analyte is pumped through a capillary tube, to which a high voltage (2 to 5 kV) of the corresponding polarity is applied. Ions in solution migrate from the interior to the surface of the liquid under influence of the high field strength near the tip of the needle. When the electrostatic forces exceed the inward forces from the surface tension a cone-jet is created. The highly charged surface of the liquid jet immediately disrupts into droplets that accelerate toward the sample cone.

When liquid flow is too high for nebulizing by internal electrostatic forces only (>10 µL/min), assistance is required to improve the process. The Unique® high-flow source consists of pneumatically assisted ESI in which nebulizing gas runs concurrent with sample flow and can handle liquid flow rates up to 1 mL/min. Pneumatic nebulization by pressurized gas (at 20 to 50 psi) helps create droplets, but often when a sample is introduced at high flow (>20 to 50 µL/min), droplets are still not small enough to produce ions before reaching the sample cone. The Unique® high-flow source also consists of a heated gas stream (desolvation gas) that helps reduce droplet size further by accelerating solvent evaporation from the droplets. Typical desolvation gas temperature ranges from 200 to 400°C and desolvation gas flow rates are 2 to 7 L/min. In order to protect the interface from contamination and improve sensitivity, the sample spray is directed toward the sample cone at an angle and only ions (not large droplets) are extracted from the stream by the electrical potential drop between the electrospray needle and sample cone.

The purpose of this performance note is to determine the optimal conditions for the Unique® High-Flow Nebulizer (HFN) Source. Optimization of source parameters will focus on factors affecting nebulization, desolvation, and ion formation for maximum performance.

Standards

Reserpine was obtained from Sigma-Aldrich. A 4.0 ng/µL reserpine test solution was prepared in a 50/50/1 solution of acetonitrile/water/acetic acid.

Instrumentation

Unique® LC-TOFMS with High-Flow Nebulizer Source
Harvard Syringe pump

MS Conditions

Source Voltage:	(+) 3.0 kV
Nozzle Voltage:	150 V
Interface Temperature:	100°C
Data Acquisition Rate:	3 spectra/second

Results

A surface plot was prepared for measuring signal intensity during infusions of reserpine at multiple flow rates ranging from 10 µL/min to 1,000 µL/min while varying desolvation and nebulizing conditions (Figure 1). Maximum signal intensity for each infusion rate is achieved at different source conditions.

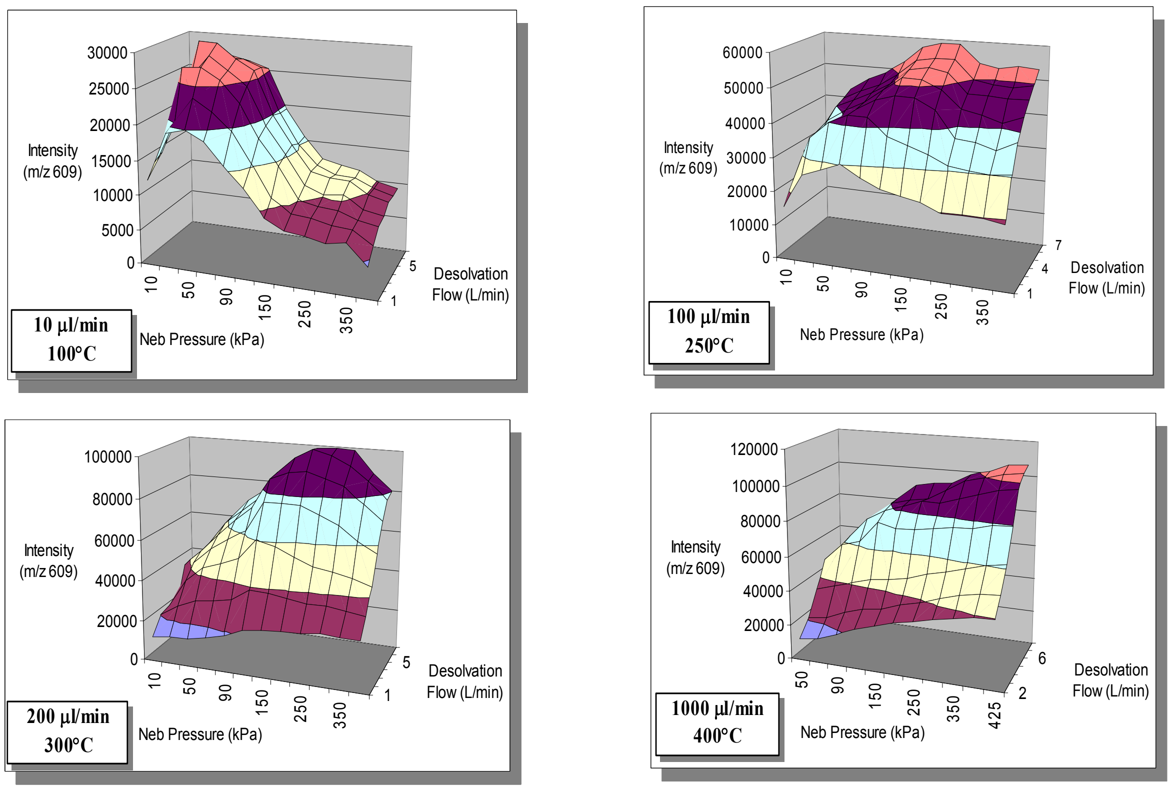

Figure 1. *Nebulization—Desolvation Surface Plots for reserpine using the HFN*

Electrospray Source

Based on the maximum signal intensity, optimal desolvation—nebulization parameters are shown below (Table 1). Increasing the infusion flow rate requires increased desolvation temperature as well as increased nebulization and desolvation gas.

Table 1. ESI Operational Parameters

Infusion Flow Rate (µL/min)	Desolvation Temp. (ºC)	Nebulization Gas (kPa)	Desolvation Gas (L/min)
10	100	25	5.0
100	200	150	7.0
200	300	200	7.0
500	400	425	7.5
1000	400	425	7.5

Conclusion

Optimal source conditions for the Unique® HFN source were determined for infusion flow rates between 10 and 1000 µL/min. These parameters provide a guide to help simplify individual method optimization and help reduce development time. It is important to remember that higher nebulization pressures and desolvation flows are required at higher infusion rates.

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***LECO Corporation* • 3000 Lakeview Ave. • St. Joseph, MI 49085-2396**
Phone: 800-292-6141 • Fax: 269-982-8977 • info@leco.com • www.leco.com
ISO-9001:2000 • No. FM 24045