Application Note

Instrument: Pegasus BT® Systems

StayClean® Ion Source Stability

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Key Words: Pegasus BT, Pegasus BT 4D, Source Stability, Reproducibility, High Matrix

Introduction

Fouled ion sources and related issues are often time consuming to diagnosis and resolve. A definitive diagnosis can only be achieved after other preventative maintenance steps (inlet maintenance, column trimming, etc.) have been tried and tested. Once the source has been pinpointed as the issue, users must remove, disassemble, clean, reassemble, reinstall and test the source before proceeding. Even routine, scheduled preventative cleaning leads to system downtime and opens the door to potential mistakes in source reassembly. In less controlled environments the incremental contamination of an ion source may not be noticed until long past the point of compromised results or impacting the stability of the overall system. Worse still, tuning routines will typically compensate for the loss in signal by increasing various voltages, not the least of which is at the detector. This decreases the detector's operational life, requiring additional maintenance time to resolve. For these reasons, LECO has specifically designed the Pegasus BT systems with an ion source that virtually eliminates the need for removal and cleaning. The purpose of this note is to provide significant evidence that the LECO StayClean ion source actually stays clean.

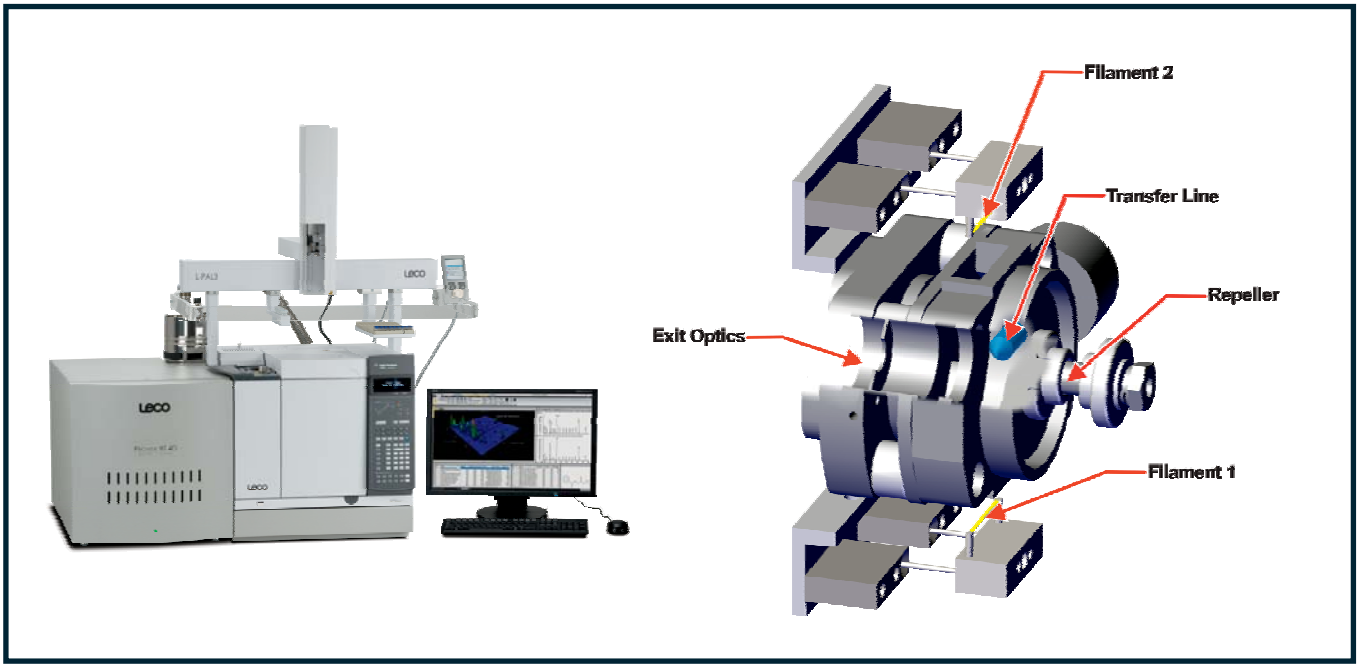

Figure 1. The Pegasus BT 4D and a simplified drawing of the StayClean ion source. The source filaments can be quickly changed without source disassembly, while the overall source design ensures maximum system stability and minimizes maintenance downtime.

As seen in Figure 1, the gridless, open-style EI source design differs significantly from traditional, closed-box style sources. With the Pegasus BT's innovative design the metal source body components are relatively distant from the ionization region. Additionally, this source format facilitates removal of neutrals and other contaminants into the vacuum and out of the flight path. These improvements dramatically reduce source fouling while generating EI spectra with high fidelity to commercially available GC-MS libraries. The only maintenance the source will require is periodic filament replacement, which has also been simplified as part of the system's design. Minimization of system downtime was among the key requirements of the Pegasus BT system design.

Experimental

A QuEChERS spinach extract spiked with various pesticides at 50 ppb was used as the test matrix. In each test round 100 spiked test matrix injections were made using parameters listed in Table 1. At the end of each round, the inlet liner and gold seal were replaced and 0.25 m was trimmed from the guard column. Following inlet maintenance, the system auto-tune was run and another 100 spiked test matrix injections followed. This sequence was repeated for a total of 400 matrix injections.

Table 1. GC-TOFMS Conditions

Mass Spectrometer	**LECO Pegasus BT**
Ion Source Temperature	250 °C
Mass Range	45-570 m/z
Acquisition Rate	8 spectra/s
Transfer Line	320 °C
Gas Chromatograph	**Agilent 7890**
Injection	1 µL Splitless, Inlet Temperature: 250 °C
Carrier Gas	He @ 1.4 mL/min, Corrected Constant Flow
Guard Column	2m, 0.25 mm i.d.(Phenomenex, Torrance, CA, USA)
Column	Rxi-5ms, 30 m x 0.25 mm i.d. x 0.25 µm film (Restek, Bellefonte, PA, USA)

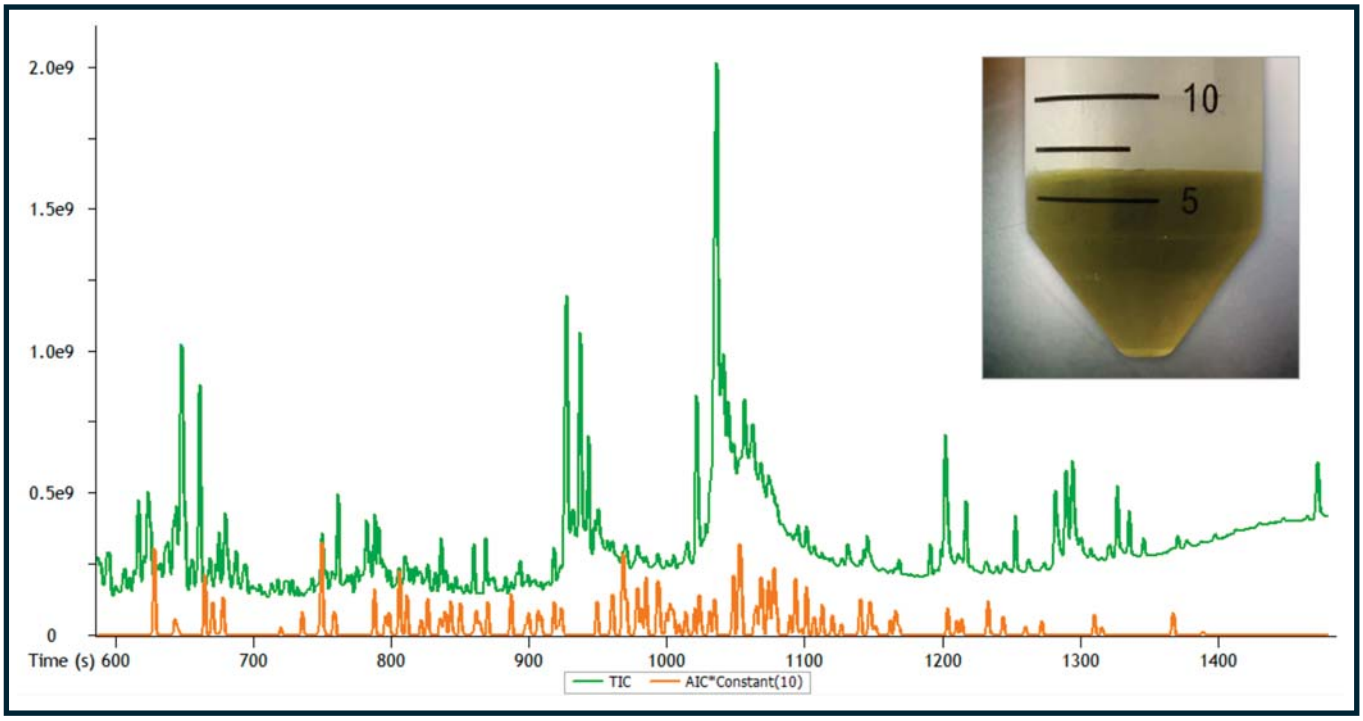

Figure 2. Total Ion Chromatogram (TIC) of the spiked spinach extract and Analytical Ion Chromatogram (AIC). The AIC is filtered to display pesticide signals ONLY, and is multiplied by 10 for visualization purposes. The insert is an image of the QuEChERS spinach extract.

Results and Discussion

A typical injection is shown in Figure 2 where significant matrix is evident; the analytes of interest (pesticides) are at least a factor of ten less intense than the background. The examples in Figure 3 show a consistent response pattern for the spiked pesticides in each of the sampling rounds. The signal degrades over the course of the 100 replicate injections, but returns to the initial response after inlet maintenance and column clipping, without cleaning the ion source. This suggests the ion source is not experiencing any signs of contamination even with a very dirty spinach matrix extract. All data were processed with the same method settings and no changes were made to the software-determined peak integrations.

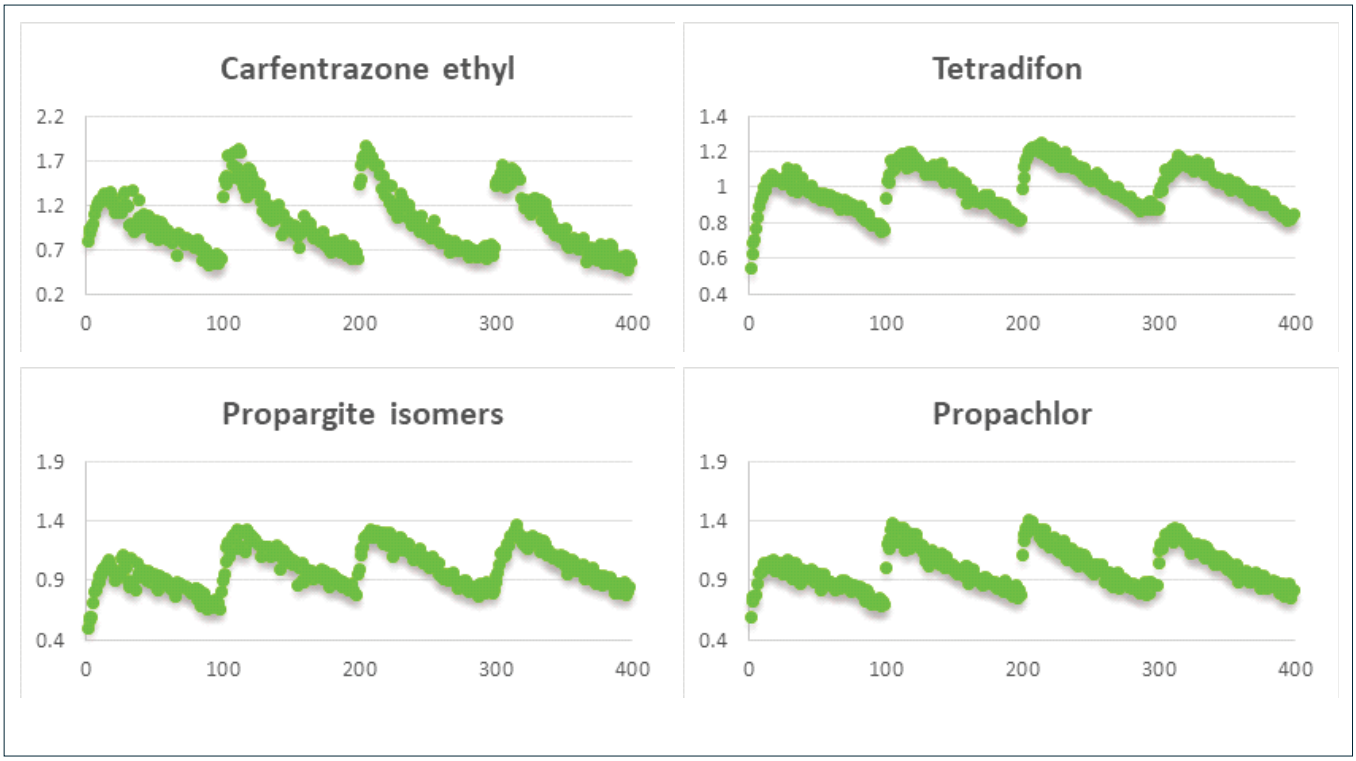

Figure 3. Response trends of four different pesticides showing signal recovery after only inlet maintenance and guard column trimming.

To verify that system tuning had not compensated for a fouling source, the tuning logs were reviewed. After the first round of injections the detector increased 12V (~0.6% of the overall voltage) and remained static for all subsequent tunings. Other values set during the automatic tuning were similarly consistent over the test period. Despite the heavy sample matrix load, the ion source remained unaffected.

Conclusion

Fouled ion sources lead to problems with test accuracy and system stability. Resolving these issues takes time and resources away from your testing schedule. Fortunately, LECO's StayClean source resists fouling even after hundreds of injections of matrix heavy extracts. Combined with the system's excellent sensitivity, integrated data collection, and analysis software, the Pegasus BT systems provide a superior testing platform for your most challenging samples.

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