Is a Never-Clean Ion Source Possible? Is it Possible to Prove It?

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INTRODUCTION

Fouled ion sources and related issues are often time-consuming to diagnose 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 to the next important sample. 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 that of the detector. This process decreases the detector's operational life, requiring additional maintenance time to resolve. For these reasons, LECO has specifically designed the Pegasus® BT with an ion source that virtually eliminates the need for removal and cleaning. The purpose of this poster is to provide significant evidence that the LECO StayClean® ion source actually stays clean.

METHOD

Two different methods of testing for fouling of the ion source were performed.

In the first test, QuEChERS spinach extract was spiked with various pesticides at 50 ppb and was used as the test matrix. In each test round, 100 spiked matrix injections were made via splitless injection. After each set of 100 injections, the inlet liner and gold seal were replaced and 0.25 m was trimmed from the guard column. Following the inlet and pre-column 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. Peak areas were integrated for the most abundant ion in the MS spectrum for each pesticide. GC and MS conditions are presented in Table 1.

In the second experiment, raw sludge samples were Soxhlet extracted for 4 hours, the neat extract was diluted 100 fold, and then injected into the GC-MS system through an uncoated capillary column. After each set of several hundred sludge extract injections, an analytical column was installed, and OFN was injected to monitor sensitivity.

Table 1. GC-TOFMS Instrument Conditions

MS	LECO Pegasus® BT
Ion Source Temp	250 °C
Mass range	45-570 m/z
Acquisition Rate	8 spectra/sec
Transfer Line Temp	320 °C
GC	**Agilent 7890**
Injection	1µL splitless @ 250 °C
Guard Column	2 m x 0.25 mm i.d. (Phenomenex)
Analytical Column	Rxi-5ms, 30 m x 0.25 mm i.d. x 0.25 µm (Restek)
Carrier Gas	He @ 1.40 mL/min, constant flow
Oven Program	75 °C (1.75 min), ramp 10.5 °C/min to 310 °C, hold 6 min

Pegasus BT / *Pegasus* BT 4D
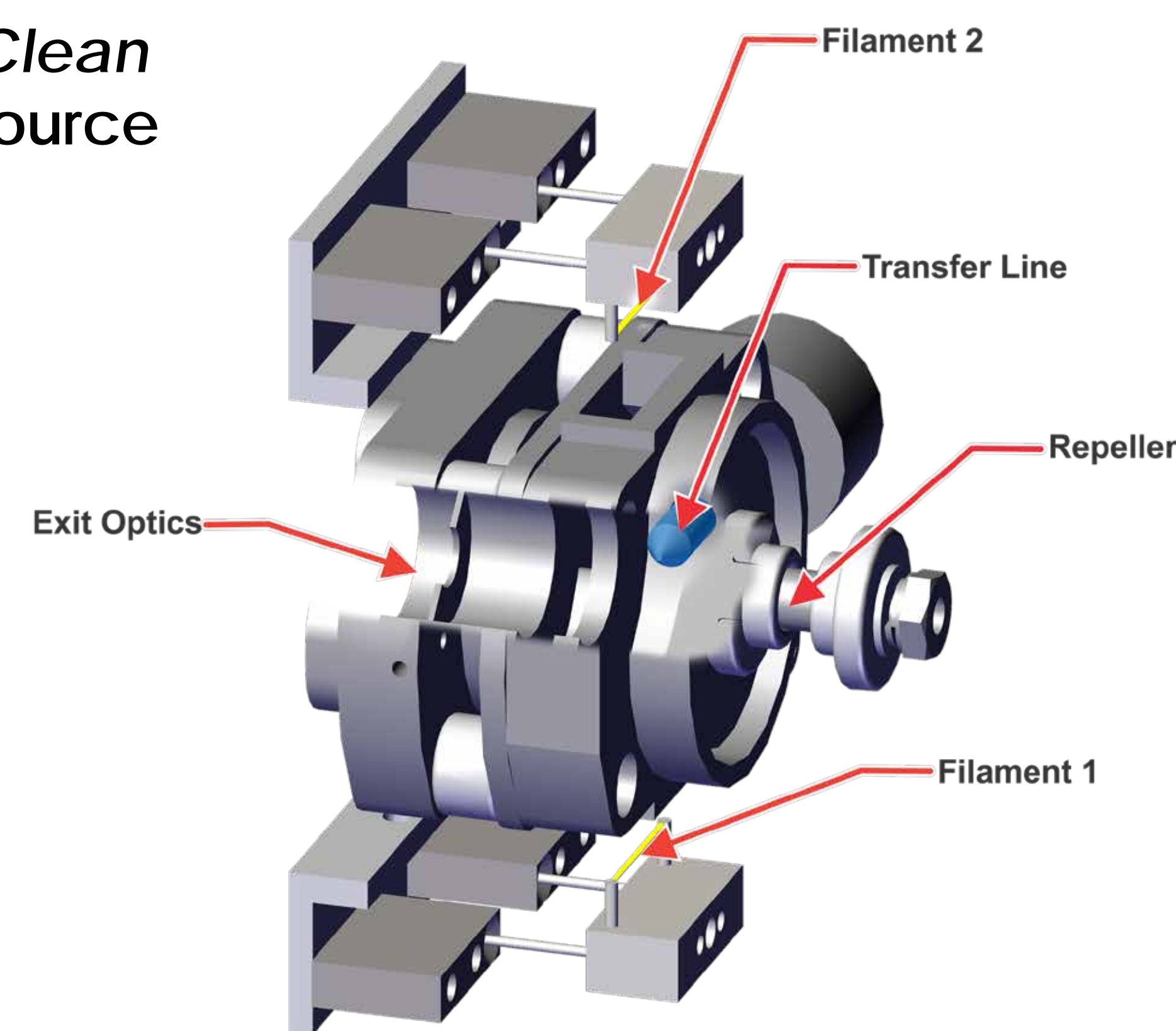
StayClean Ion Source

Figure 1. Modified CAD drawing of the StayClean Ion Source available on the Pegasus BT and Pegasus BT 4D systems.
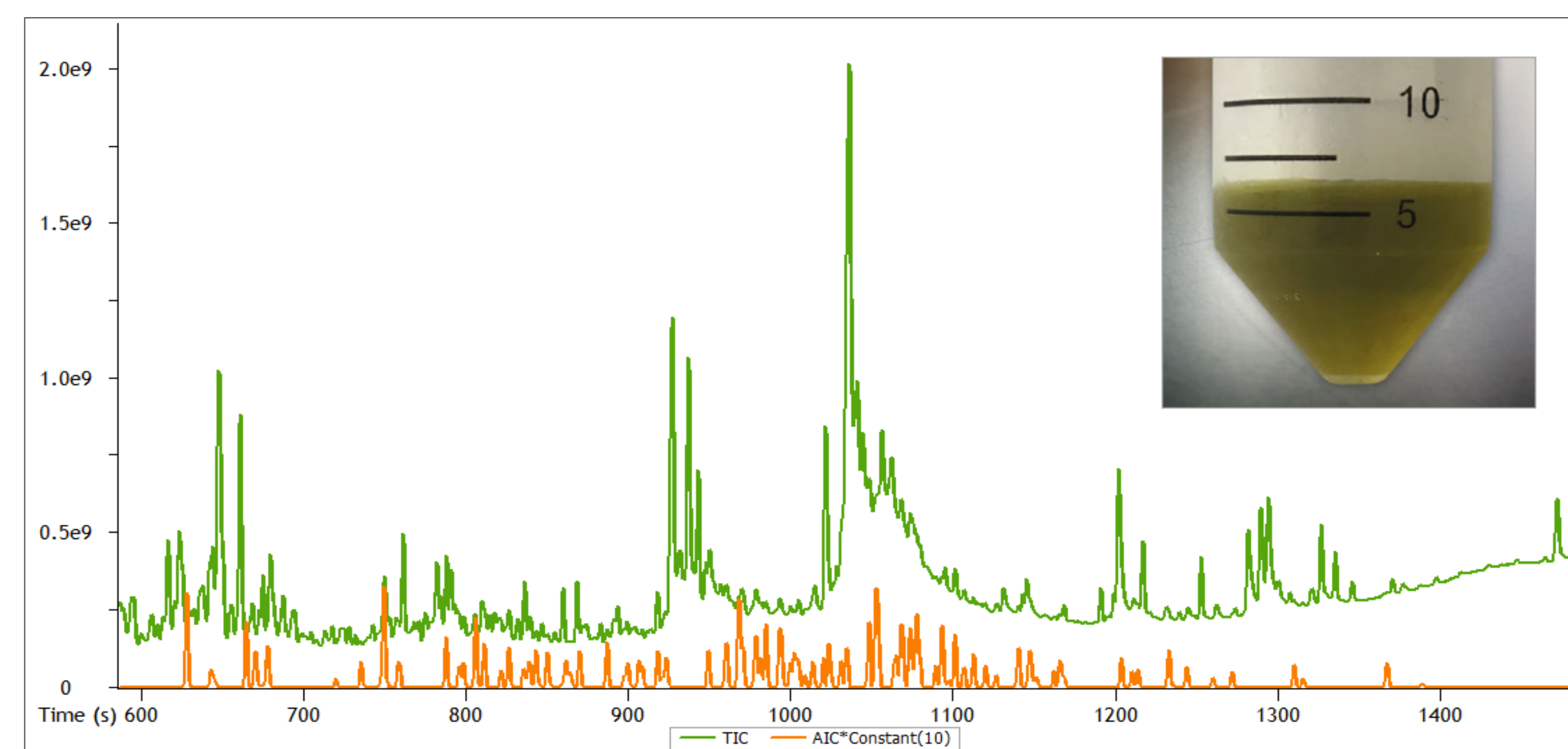

Figure 2. Overlay of the total ion chromatogram (TIC) and analytical ion chromatogram (AIC, all spiked pesticides total) for a QuEChERS spinach extract, inset in the upper right corner; Note AIC magnified x10.
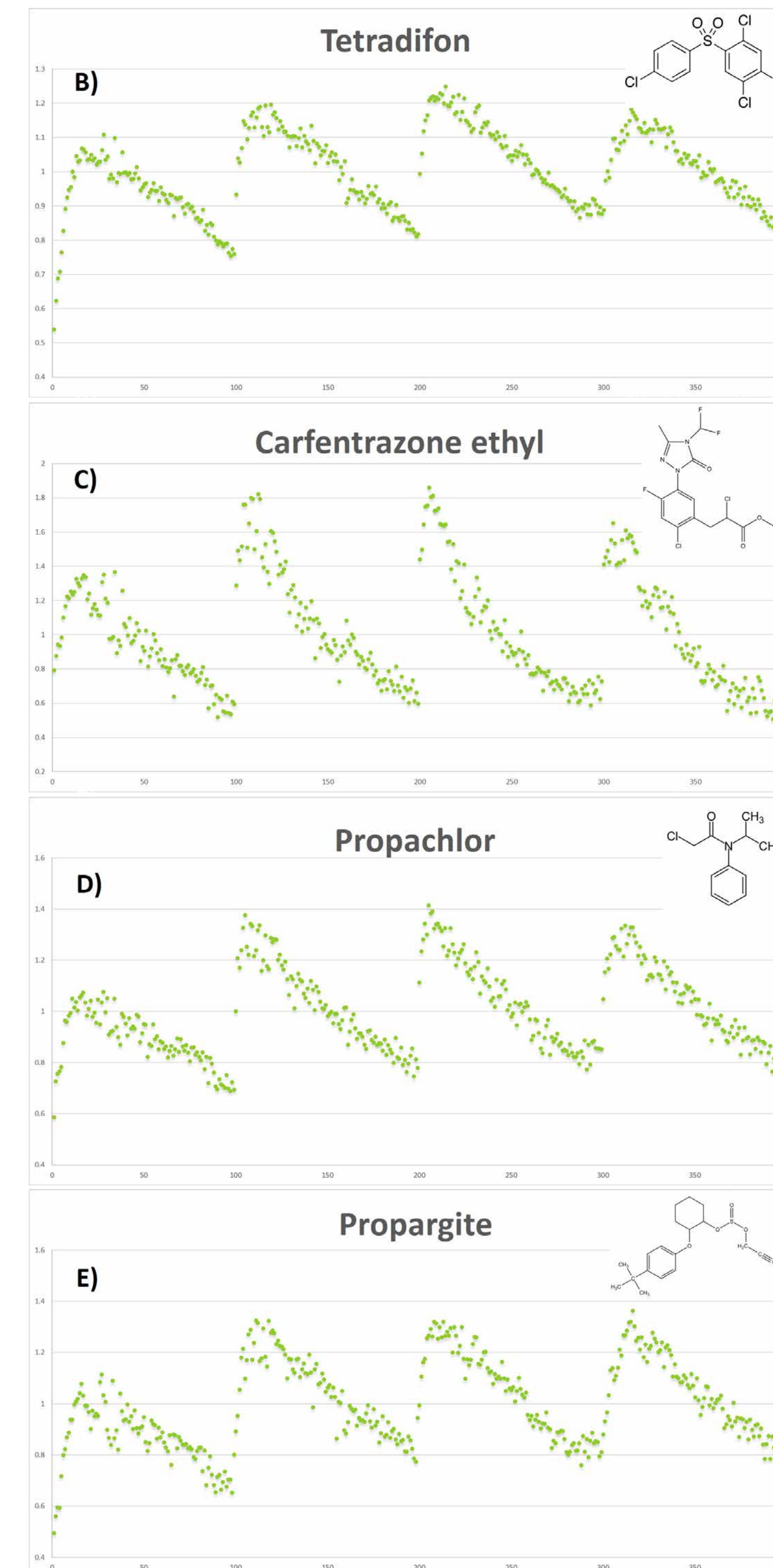

Figure 3 B to E. Peak area versus injection number for several pesticides in QuEChERs Spinach extracts.

RESULTS

A typical injection is shown in Figure 2, where significant matrix is evident—the analytes of interest (pesticides, orange) are at least a factor of ten less intense than the background. This matrix level indicates and confirms that there is significant probability for each injection to foul the source. This source of contamination is also evident by the decrease in response of each injection before conducting inlet and GC maintenance. The examples in Figure 3B) through Figure 3E) 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, all without cleaning the ion source. This suggests the ion source is not experiencing any signs of contamination even with injections of 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.

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.

As shown in the bar graphs, in Figure 4, no performance degradation was observed even after 2000 sludge extract injections.
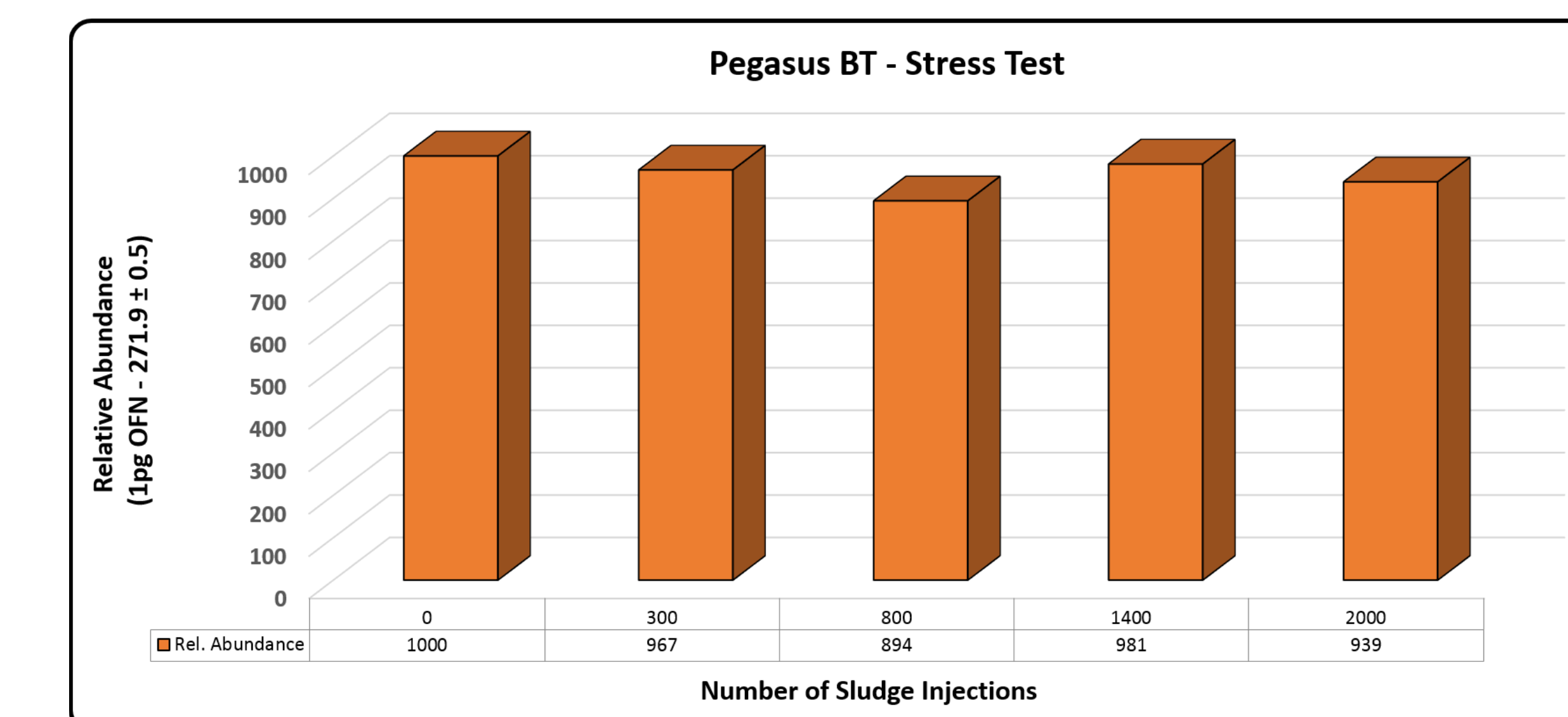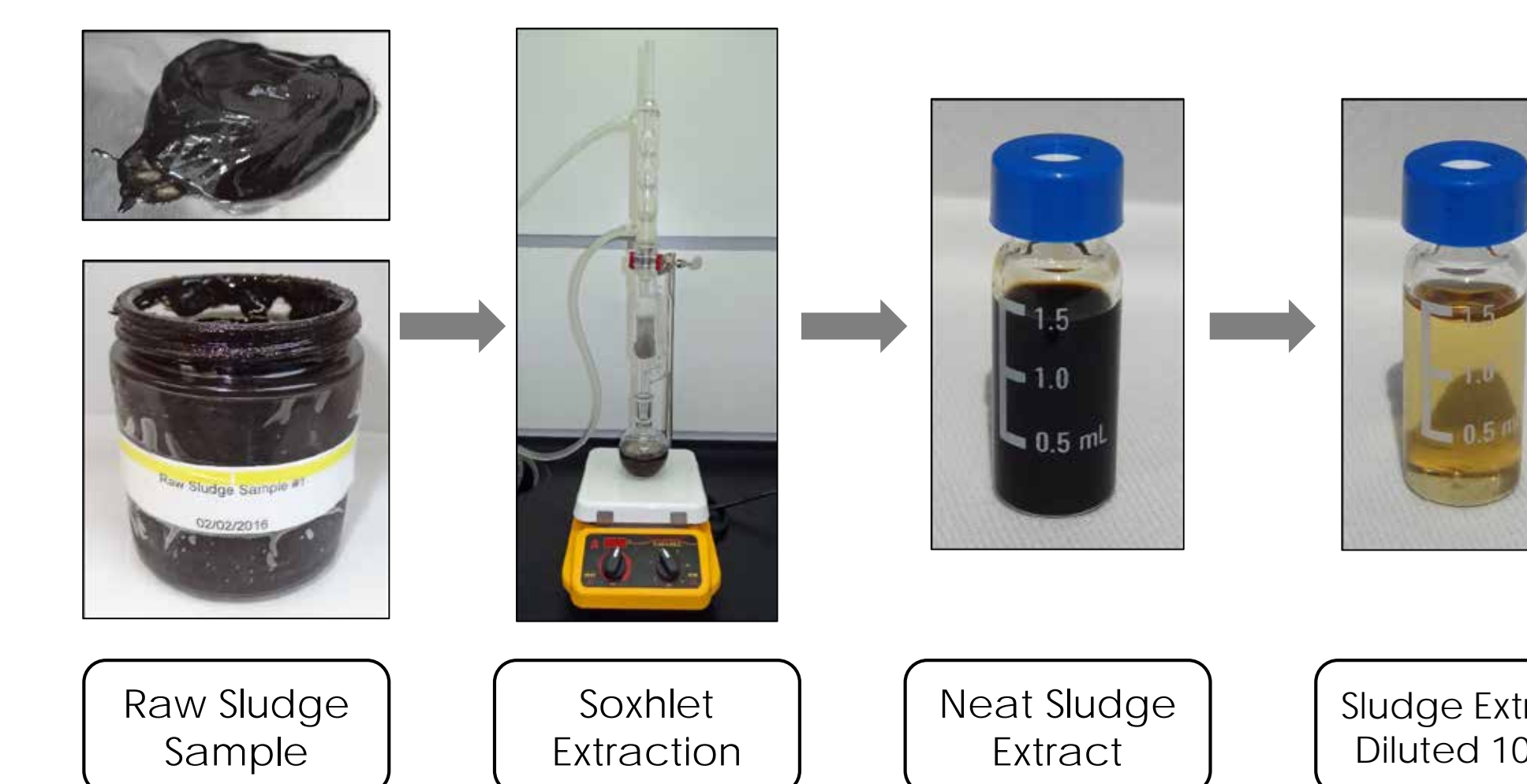

Raw Sludge Sample	Soxhlet Extraction	Neat Sludge Extract	Sludge Extract Diluted 100:1

Figure 4. Relative abundance of several 100 injections of diluted sludge extract into the StayClean ion source.

CONCLUSIONS

In this work we have shown that the open-style EI source labeled as StayClean is not fouled by dirty matrix injections. This eliminates the need for special setups for cleaning (including the use of Hydrogen gas), clean source exchanges, or standard downtime that is typical for cleaning a "closed box" style source.

ACKNOWLEDGEMENTS