The Triple-Axis Detector: Attributes and Operating Advice

Technical Overview

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The new Triple-Axis detector (TAD) uses a unique geometry and employs an improved electron multiplier design that will provide the user with several practical advantages:

- At a given operating gain, the new detector provides higher signal intensity by collecting more ions emerging from the quadrupole.
- Although signal is enhanced, neutral noise is substantially reduced through the off-axis design.
- Detector lifetime is also increased under proper operation.

This technical overview provides operating advice and describes other attributes that may be observed for this advanced detector.

Attributes

The user will find signal is greatly enhanced and neutral noise reduced with the TAD. The increased signal provides many benefits in analysis, such as enhanced detection limits, increases in compound relative response ratios, and calibration curves with greater slopes. In operation, because the TAD is more sensitive, the user will find tuning voltages can be lower. When autotuning on the PFTBA calibrant gas, the TAD will produce a very slight “tilt” in abundance favoring the lower masses over what was experienced in the previous detector. This “tilting” could be incorrectly interpreted as a decrease in response toward the higher masses. In fact, Gain Normalized methods will show better response for compounds with higher mass fragments, such as 502 and beyond. For example, air and water may appear higher by roughly 2-fold over what the user experienced in the other detector design, but only in absolute counts; abundances relative to *m/z* 69 will remain the same. Using the BAKE menu command will accelerate the reduction in background.

The electron multiplier (EM) lifetime is greatly improved in this new design if carefully operated. Atune and typical operating voltages will appear lower than the previous detector; operation should take this into account to avoid saturating the detector.

Operation

It is strongly recommended that the new detector be used with Gain Normalized tuning and methods. The technical note “Enhancements to Gain Normalized Instrument Tuning” [1] should be read and understood. The previous and common approach of setting the electron multiplier voltage in the MSD method parameters, as the Autotune electron multiplier voltage plus some additional voltage (for example, ATUNE+400V), **should be avoided**. The detector could potentially be damaged by excessive signal current. Any signal, in tuning or acquisition, near 8 million counts is essentially “saturated” and the multiplier voltage should be reduced. This is easily recognized as flat-topped or “clipped” peaks. While transient events near saturation are usually unavoidable in complex samples, the data should be examined and a Gain Factor should be set to prevent analytes from producing these high values for ion abundance. The situation is especially serious in selected-ion monitoring (SIM), where the detector is exposed to the ion current for a relatively long duration. To avoid saturation during SIM, the user should employ the ***EM Saver*** function located under the **Method** menu item (Figure 1). ***EM Saver*** sets a limit to the total ion current the detector can experience in the course of a SIM acquisition. Using ***EM Saver*** protects the TAD (as well as the standard detector) against high currents, which will degrade any EM, and provides the user with more stable compound responses even if high signals are unavoidable in the samples. ***EM Saver*** should be enabled in all SIM methods with the TAD; however, because ***EM Saver*** is not on by default, it must be selected by the user. The default maximum count setting is 10^8 and should be a good setpoint for the majority of SIM methods.

All electron multipliers have some degree of sensitivity to exposure to air and the TAD is no exception. If not in use and under vacuum in the analyzer, the TAD must be stored in a desiccator under dry nitrogen or argon. When the EM is replaced in the TAD, immediately install the replacement EM after the sealed bag containing the EM is opened.

An Example

In the absence of dominant chemical noise, a comparison between data acquired by the standard detector and the new TAD under Gain Normalized tuning conditions should reveal roughly a factor of two increase in overall "sensitivity." A typical

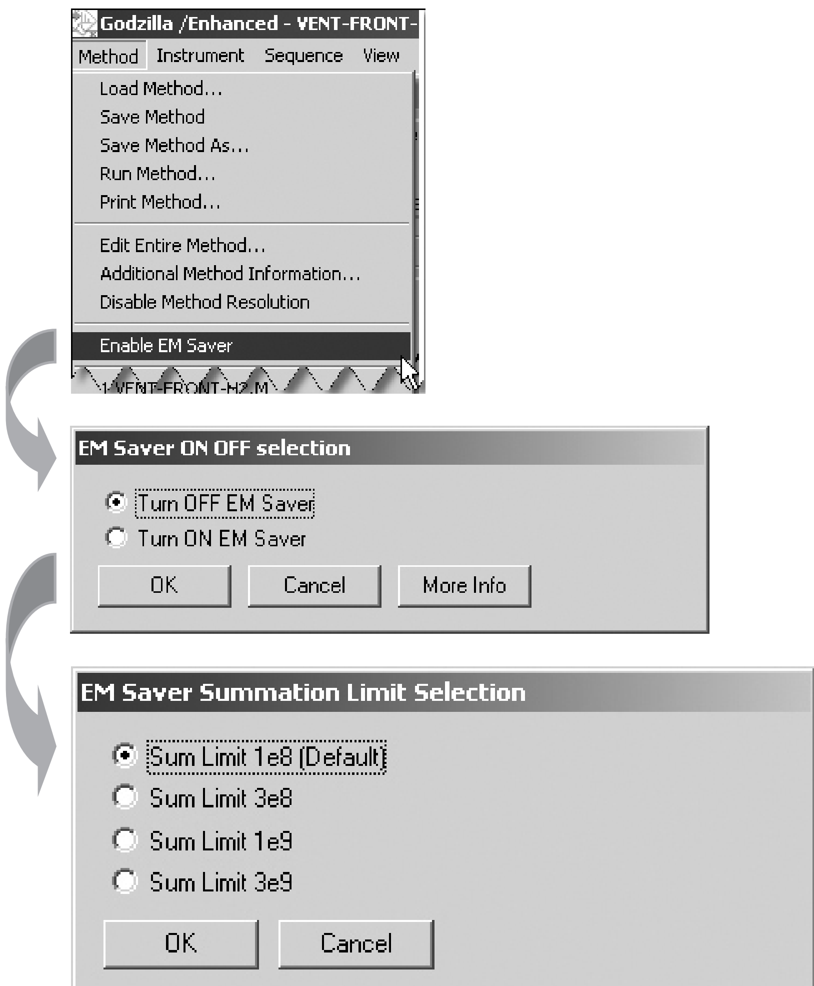

Figure 1. ***EM Saver*** **menu item. By default,** ***EM Saver*** **is off and must be turned on by the user.**

result is shown in Figure 2. The signal for this compound is enhanced but the neutral noise has been decreased. Another example in quantitation (Figure 3) shows that the slope of the calibration curve is roughly doubled as the definition of sensitivity implies. Also the instrumental detection limits (IDL) are further extended to even lower concentrations over the standard detector. The better reproducibility at lower concentrations can result in better method detection limits if the analysis is signal limited.

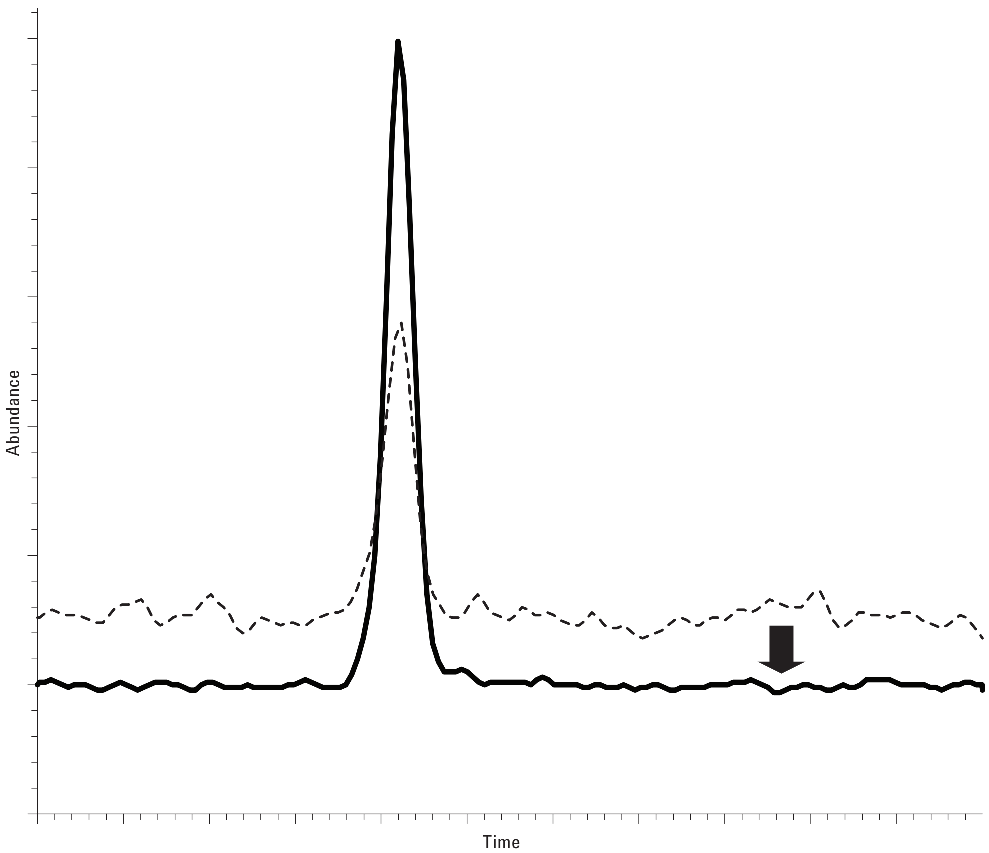

Figure 2. Acquisition of a hexachlorobiphenyl standard with the TAD (solid line) and standard detector (dashed line). Note that the increased signal and decreased neutral noise in the baseline results in a 7-fold increase in the signal-to-rms noise.

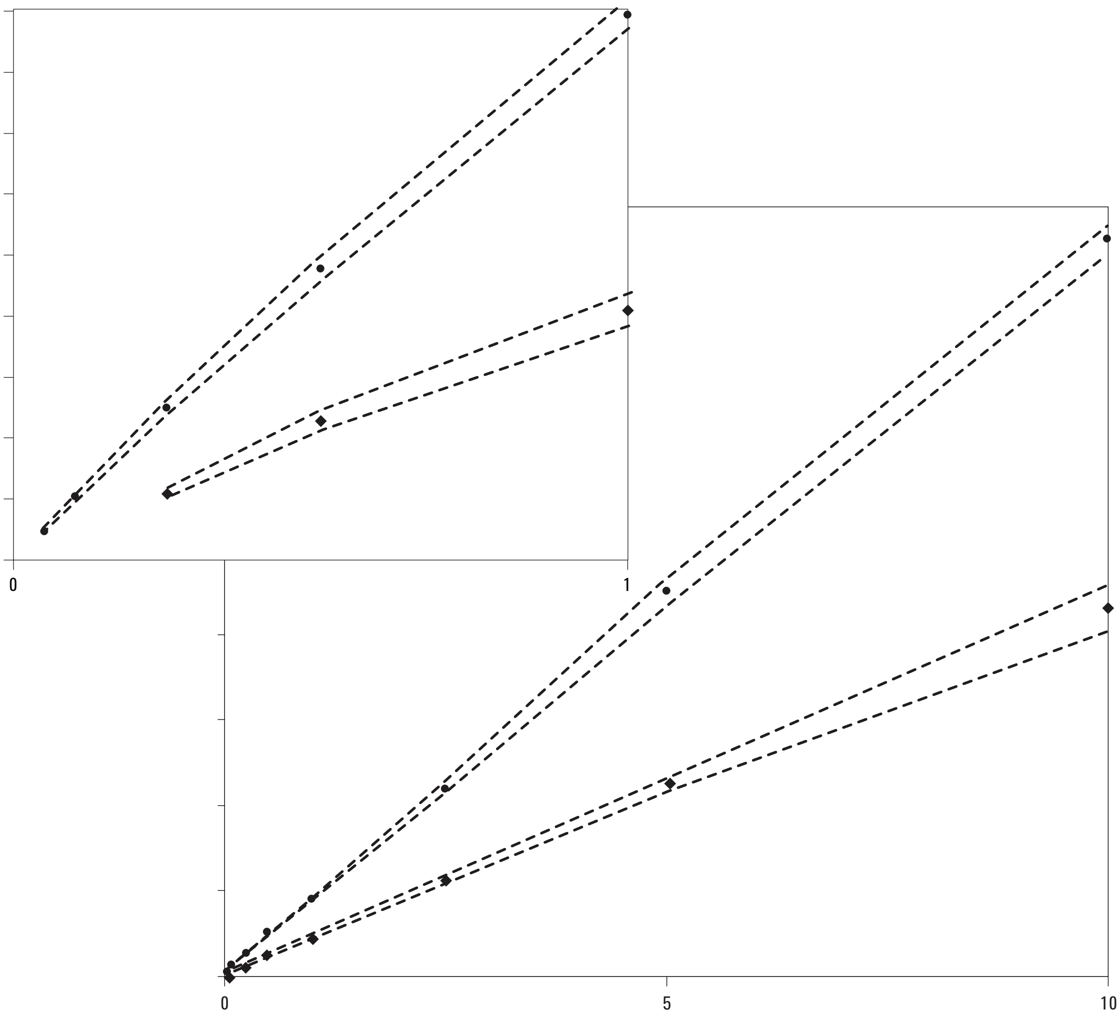

Figure 3. **Example of the enhanced detection limits and sensitivity provided by the TAD compared with the standard detector in a plot of response versus amount (pg) of a trichlorobiphenyl. The upper series of data points are for the TAD, the lower series for the standard detector. Dashed lines represent 95% confidence limits. Note the higher slope for the TAD response curve, which is by definition the sensitivity, and the high degree of reproducibility even at femtogram amounts (enlarged section).**

References

1. H. Prest, J. Foote, J. Kernan, D. Peterson "Enhancements to Gain Normalized Instrument Tuning," Agilent publication 5989-7654EN.

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