

EVALUATION OF ETP ELECTRON MULTIPLIER IN HP5973

SGE would like to thank Ms. Carol Lovett of Severn-Trent Laboratories (Austin, Texas) for providing GC-MS instrumentation, analytical materials and production of raw data, and also Mr. Larry Garretson (Owner, Scientific Instruments Sales & Service, Round Rock, TX) for evaluation of data and providing written contents of this application note.

The HP 5973 MSD has proven to be an extremely sensitive analyzer. However, due to this increase in mass spectrometer sensitivity, many users have experienced severe limitations in system linearity. In applications where high concentration samples are routinely analyzed, the original electron multiplier supplied with the MSD has been shown to have a limited range of linearity. SGE's ETP Active Film Multiplier™ was installed to evaluate the potential for extended linear range performance.

Severn-Trent Laboratories (formerly Radian International, Austin, Texas) provided their recently acquired HP 5973 MSD to be utilized in this evaluation. Prior attempts to calibrate the system using the standard K&M electron multiplier revealed a significant reduction in MSD linearity. To evaluate the ETP multiplier, EPA SW-846 Method 8270 was selected due to the well-defined calibration criteria required for acceptable method performance. Method 8270 employs the use of multiple internal standards and requires a minimum of five levels for GC-MS calibration. Due to the unpredictable nature of samples analyzed by Method 8270, the analyst would prefer their instrument calibration cover as wide an analytical range as possible. Sample concentrations, which exceed the calibration range, must be diluted and re-analyzed reducing productivity and throughput.

For purposes of comparing the original electron multiplier supplied with the HP 5973 and the ETP Active Film Multiplier, two sets of calibration data were acquired. Utilizing MS tuning criteria specified by Method 8270, a seven-point calibration was produced first on the standard K&M electron multiplier. The ETP multiplier was then installed and the instrument tuned to the same MS tuning criteria. An identical set of seven-point calibration data was generated. The calibration levels selected for this comparison were 10, 20, 40, 50, 75, 100 & 150 µg/mL (ppm). The six deuterated internal standards were held constant in each level at 40ppm.

From the multi-component Method 8270 standard, three representative compounds were selected to evaluate electron multiplier response. **Table 1** presents the three compounds selected and the internal standards employed for quantitation.



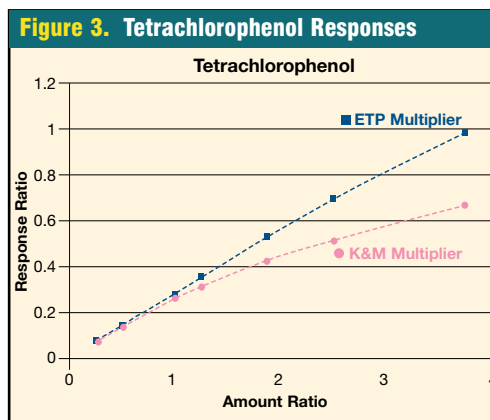
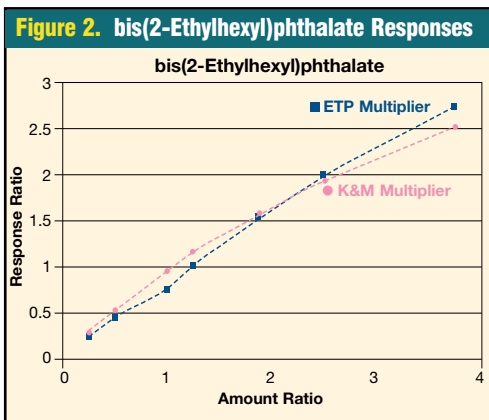
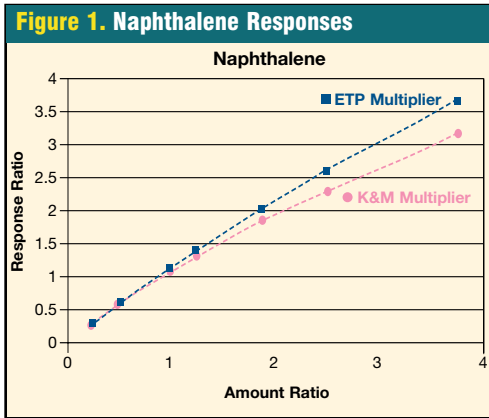
Table 1.

Method 8270 Compound	Internal Standard for Quantitation
Naphthalene	Naphthalene-d ₈
bis(2-Ethylhexyl)phthalate	Acenaphthene-d ₁₀
2,3,4,6-Tetrachlorophenol	Chrysene-d ₁₀

Method 8270 provides specific criteria for evaluating the acceptance of initial multi-point calibration performance. Typically, relative standard deviation (RSD) is calculated for each component across the concentration range of the initial calibration. RSD's greater than 10% are indicative of non-linear response for compounds in the multiple calibration range. Calibration curves can be generated for each compound illustrating at which point along the curve the compound becomes non-linear in response. In most cases, this non-linear response results from the electron multiplier exceeding its dynamic range due to excessive signal levels. This phenomenon is known as detector saturation and occurs at higher calibration levels.

Seven-point calibration curves were generated for the three representative compounds selected for electron multiplier comparison. **Figures 1 – Figure 3** provide a visual presentation on the performance of the two multipliers.

As can be seen from the calibration curves, the standard K&M multiplier exhibits non-linear response at the sixth calibration level, corresponding to 100ppm. In the case of Naphthalene and Tetrachlorophenol, detector saturation is indicated beginning at the fifth calibration point corresponding to only 75ppm. The ETP Active Film Multiplier shows linear response throughout the entire calibration range, including 150ppm. When statistical factors are applied to each of the calibration curves, improved performance with the ETP multiplier is clearly shown. **Table 2** provides the statistical representation of these three compounds when evaluated for response factor relative standard deviation and response factor correlation coefficient (r^2).



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Table 2.

Multiplier	Naphth (RSD)	Naphth (r^2)	Bis(2-EH) (RSD)	Bis(2-EH) (r^2)	TCP (RSD)	TCP (r^2)
K&M	14.4	0.998	16.7	0.976	14.8	0.976
ETP	7.9	0.996	6.6	0.994	3.1	0.998

As shown in **Table 2**, the linear response of the ETP multiplier reduced standard deviation between 190-477%. Subsequent standard analysis indicates the ETP multiplier was linear in calibration ranges up to 200ppm. This represents a greater than 250% improvement in calibration range linearity when compared to the standard K&M electron multiplier. In critical applications where a wide variety of sample matrices are encountered, the ETP Active Film Multiplier demonstrated superior linear performance even when detecting analytes at very high concentrations.