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Importance of GC Column deactivation technology for the analysis of challenging analytes

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Introduction and Methods

Gas chromatography of semivolative compounds continues to be limited by the activity of residual silanols on the surface of fused silica capillaries, which allow adsorption, resulting in peak distortion and reduced sensitivity - especially when considering acidic and basic analytes at trace levels.. Column deactivation plays a central role in eliminating these active sites and ensuring stable adhesion of the stationary phase. However, incomplete or unstable deactivation can accelerate thermal degradation of the phase, reducing long-term chromatographic performance. This study examines an advanced deactivation system designed to balance the performance of acidic, basic, and neutral compounds, improve column robustness under thermal stress, and support consolidated GC-MS/MS workflows for complex analyte lists.

Multiple GC columns manufactured with a novel deactivation, and evaluated through controlled quality-control (QC) tests and targeted stress studies. Lot-to-lot consistency was assessed using QC probe compounds sensitive to silanol activity. Columns were subjected to 15 thermal-stress cycles at 330°C with extended high-temperature holds to accelerate phase deterioration. Peak shape, symmetry, and retention drift were monitored before and after stress cycling. In addition, 63 acidic, basic, and neutral compounds were calibrated according to EPA 8270E criteria (0.5-5000 ppb) to evaluate linearity, response-factor stability, and mid-level peak symmetry. A broader mixture of >150 semivolatiles was analysed at 10 pg on-column to assess suitability for large analyte lists and simultaneous multi-class quantification.

Column Lifetime and Variability

Methods: The lot-to-lot consistency of multiple RMX-5Sil MS column were QC checked by injection of a test mixture to monitor column performance across the following areas: adsorption, activity, acid/base characteristic, separation efficiency, and film thickness. Following this an RMX-5Sil MS column was challenged with 15 thermal stress cycles wherein the column was heated to 330°C and held for four hours to expediate phase deterioration, after each hold the column was returned to 125°C. QC probes were monitored before and after each thermal stress cycle to track column performance over time.

Cleaner deactivation results in better reproducibility in manufactured columns. QC probes sensitive to interaction with surface silanols showed consistent response across multiple manufacturing lots.

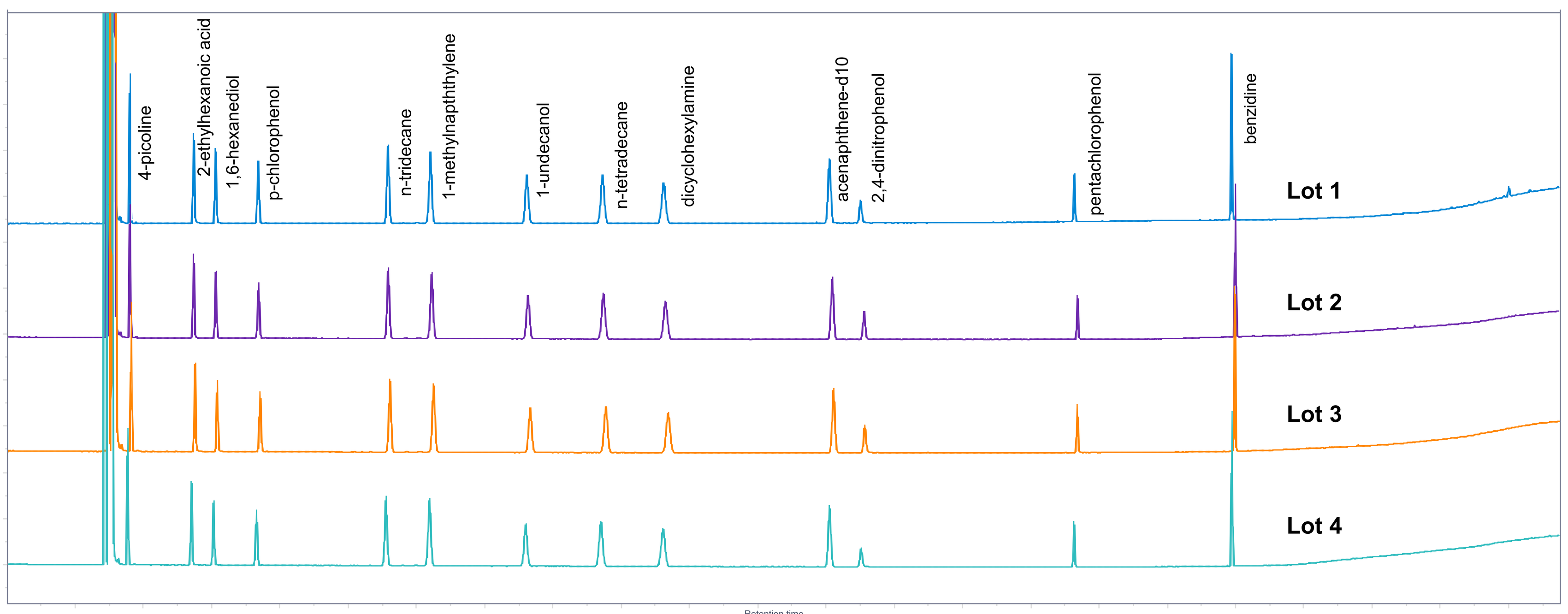


Figure 1: QC test chromatograms from four RMX-5Sil MS columns manufactured in different lots

Complete deactivation leaves fewer free-silanols to catalyse phase degradation under thermal stress conditions. Peak shapes are consistent and resilient throughout 15 thermal stress cycles.

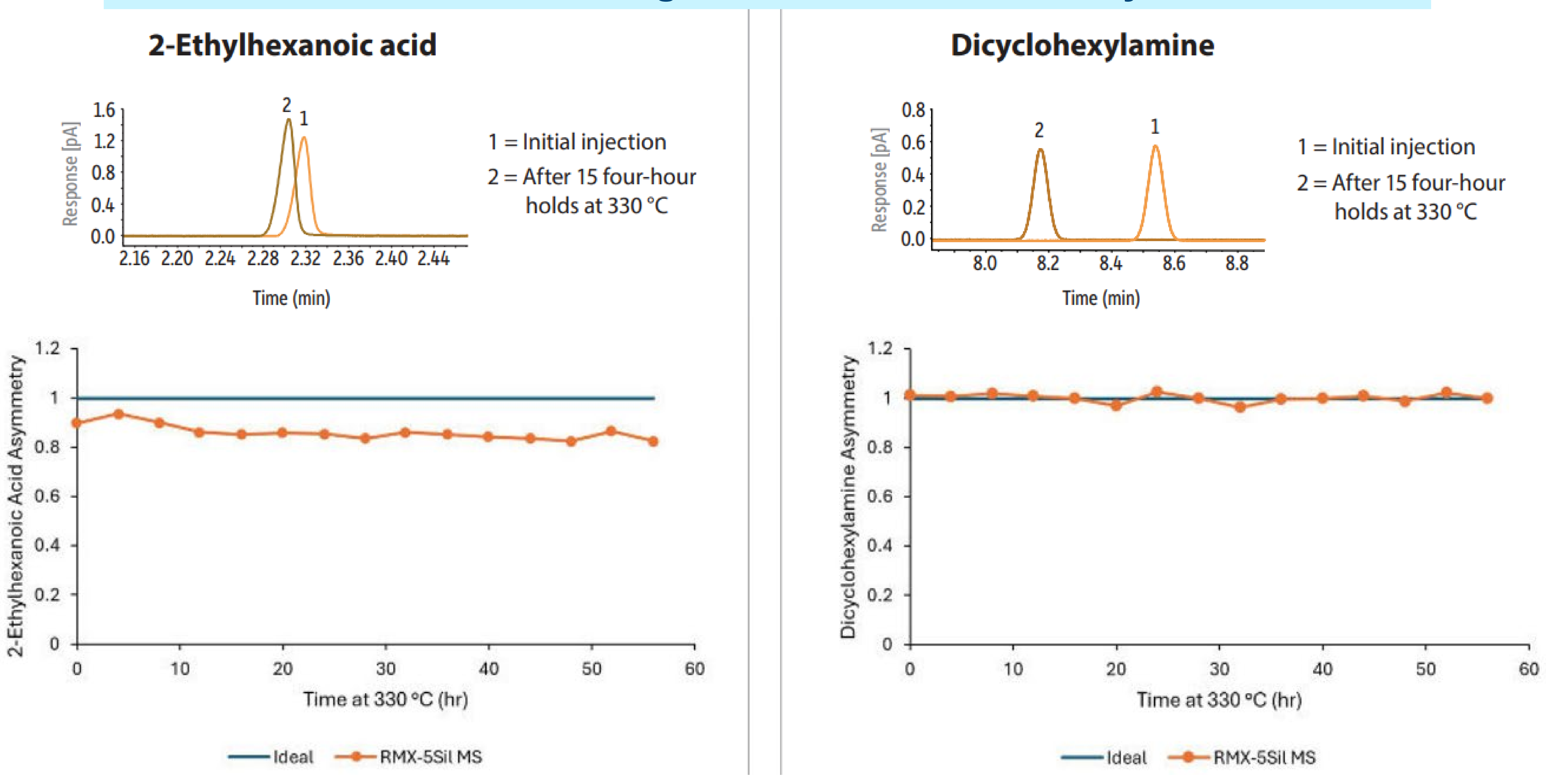


Figure 2: (Top) Peak shape of acidic (left) and basic (right) analytes before and after thermal stress cycling, with changes in peak symmetry monitored throughout stress cycling (bottom).

Low-level Calibration for Sensitive Instruments

Methods: Sixty-three challenging acidic, basic, and neutral compounds were calibrated according to EPA method 8270E by GC-MS/MS at 0.5-5000 ppb. Linear (no-weighting) calibrations were fit to each compound to compare linearity at low levels. Peak symmetry was evaluated at 50 ppb, which served as a mid-range calibration point for most compounds. Linearity and peak shape were assessed on four additional 5-type (silarylene) columns, and linearity parameters (R^2, RRF and RSD) were assessed for each column. A suite of >150 semivolatiles (with surrogates and internal standards) was assessed on the RMX-5Sil MS to demonstrate peak shape and clarity at low-pg amounts on column to demonstrate versatility of the RMX-5Sil MS surface for challenging compounds.

Deactivation technology influences peak shape of acidic and basic compounds, traditionally favouring performance of acids. Improved technology allows for balance between acidic and basic compounds.

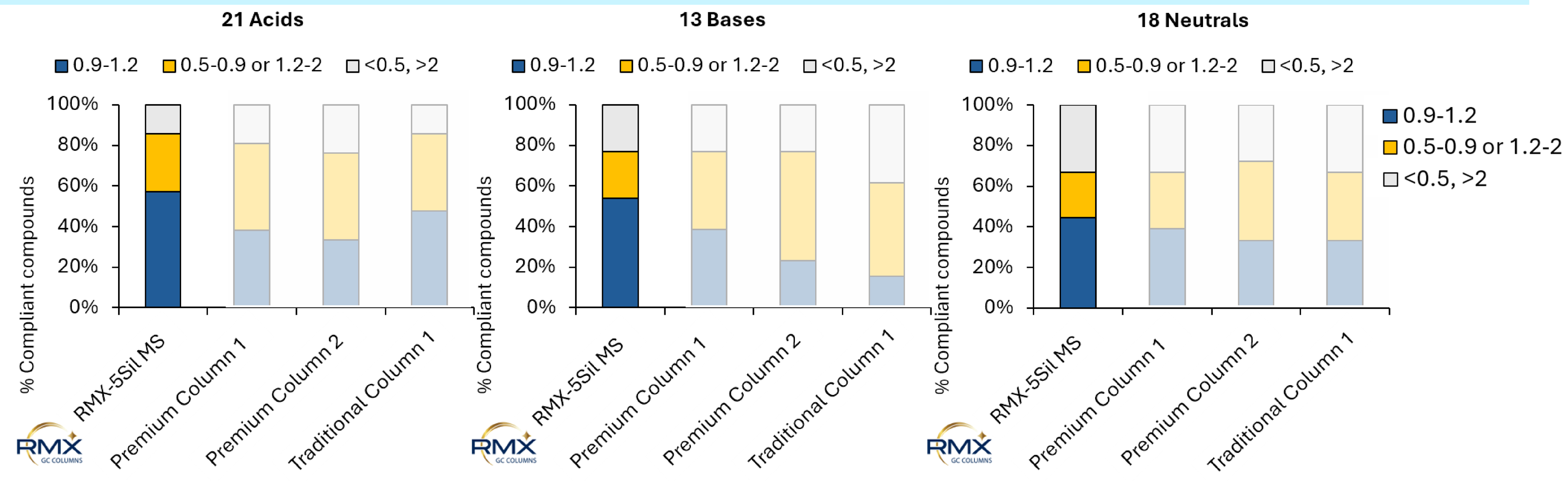


Figure 3: Proportion (%) of challenging compounds that meet peak shape quality (asymmetry) criteria at 50ppb, on various GC columns.

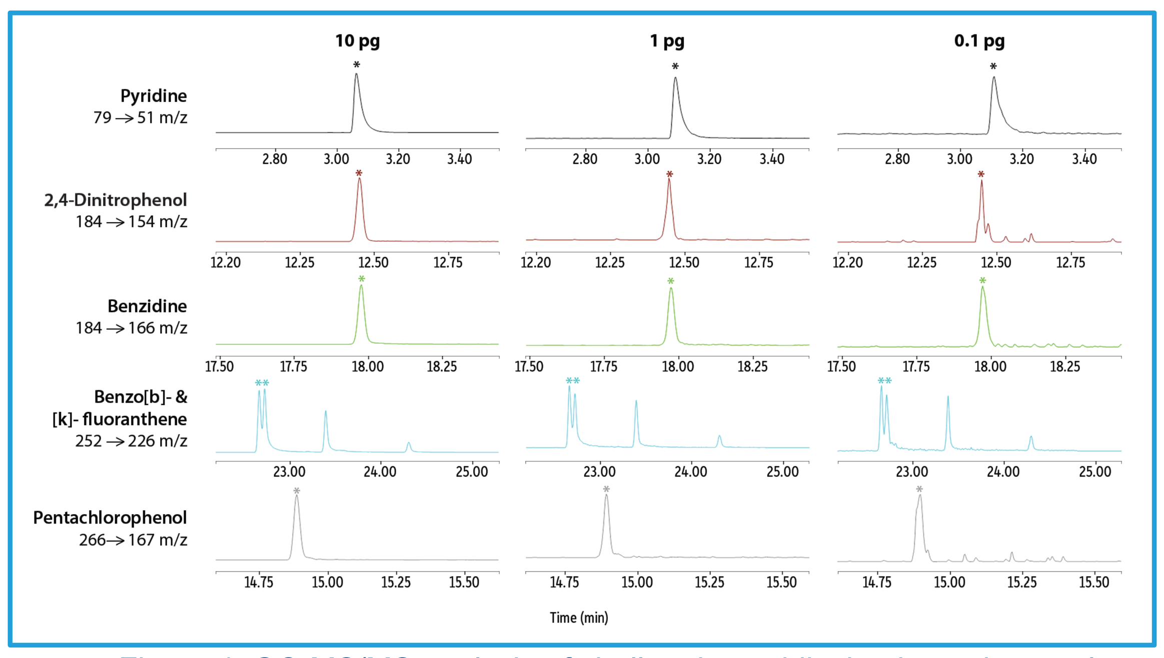


Figure 4: GC-MS/MS analysis of challenging acidic, basic, and neutral compounds associated with EPA 8270E at decreasing amounts on column.

Peak shape dictates the ability of software to recognize and integrate during instrument calibration. Improved peak shapes therefore allow for improved calibration linearity at low levels.

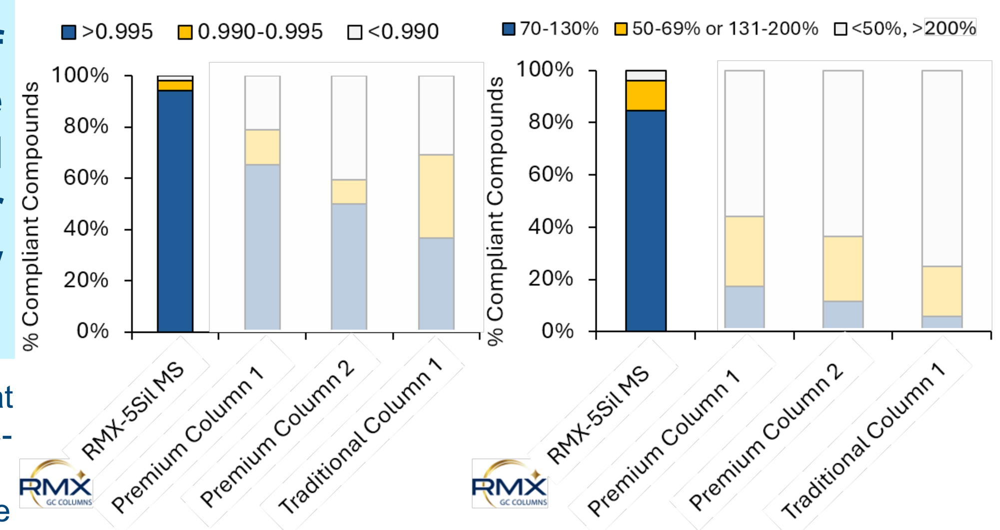


Figure 5: Proportion (%) of challenging compounds that meet EPA 8270E compliance criteria with low level GC-MS/MS calibrations, on various GC columns. LHS calibration curve linearity by R^2 value. RHS percentage recovery at lowest calibration point

Ease of integration for challenging compounds ensures successful expansion of analyte lists and consolidation of methods to improve lab efficiency. Over 150 semivolatiles are analysed simultaneously by GC-MS/MS at only 10 pg on column.

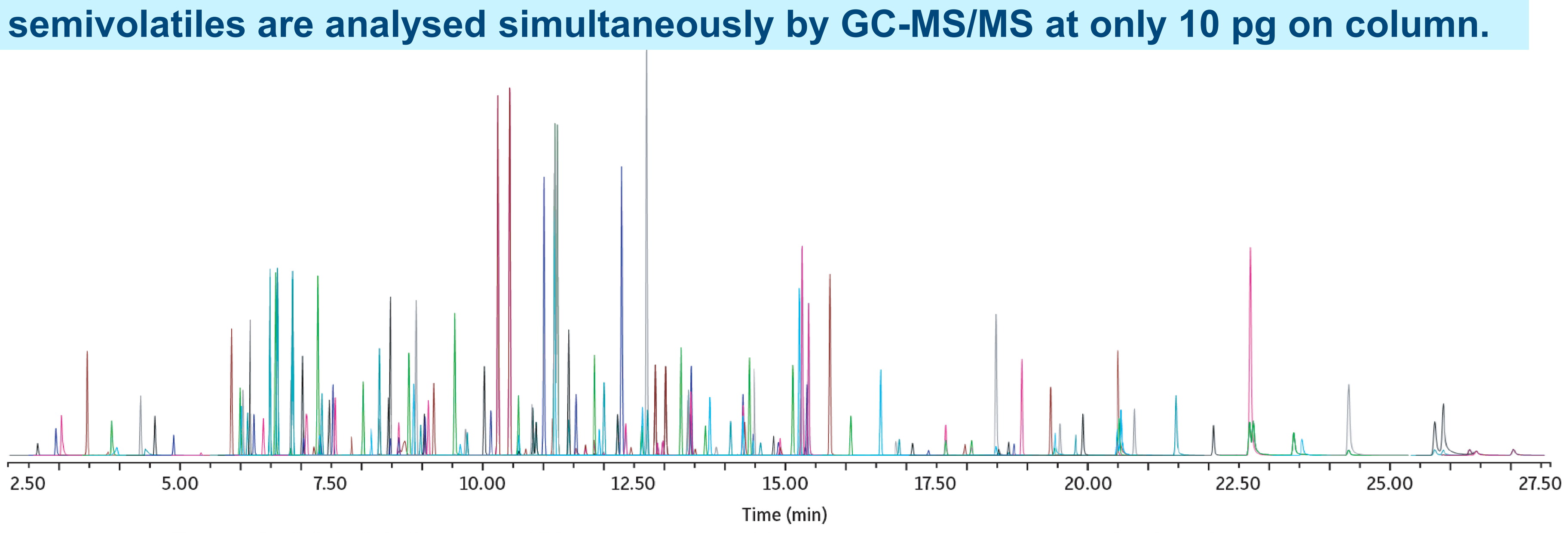
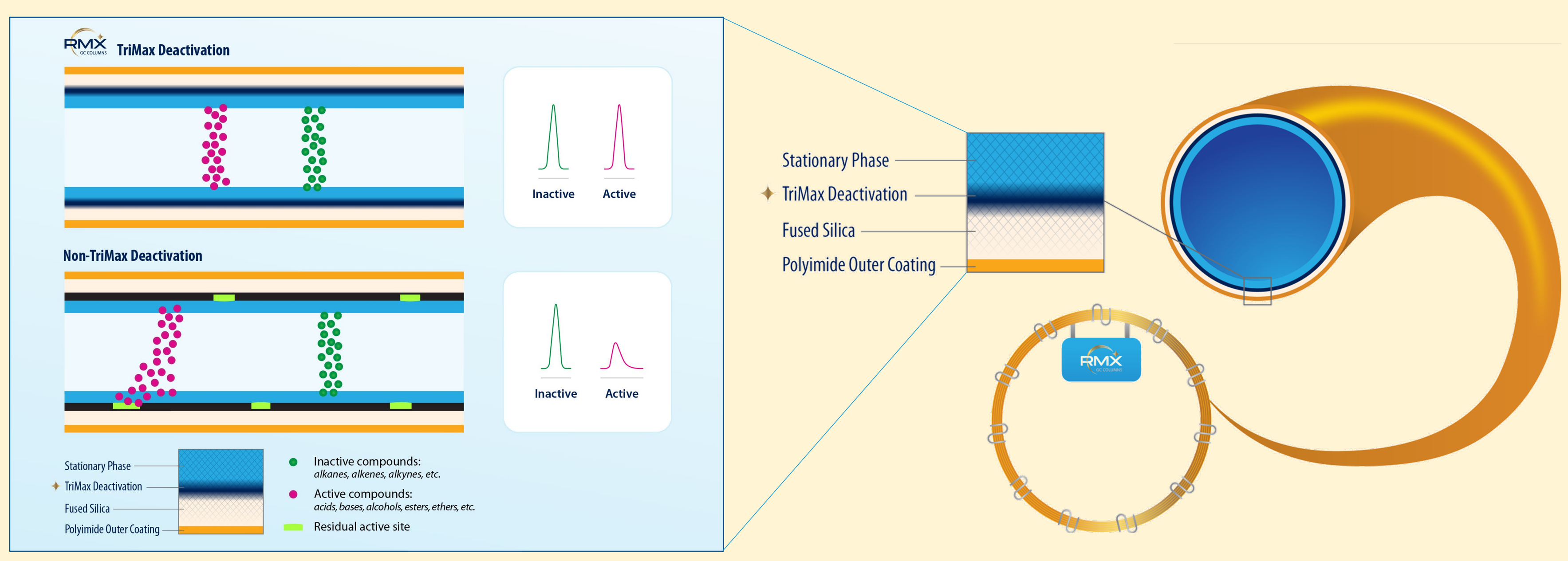


Figure 6: GC-MS/MS of over 150 semivolatiles (10 pg on column) comprised of acidic, basic, and neutral compounds with a variety of functionalities.

The role of deactivation in gas chromatography



Lower instrument detection limits

- Sharper peak shapes at low levels allow for low level accuracy and precision

Adapt to new instruments and methods

- Reduce sample size and solvent use with alternative extraction methods

Consolidate methods with confidence

- Balance performance of acidic, basic, and neutral analytes in the same run.

Consistent, extended column performance

- Resilience to harsh matrix maintains consistent performance with maintenance