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Importance of GC Column Deactivation Technology in the Analysis of Challenging Compounds

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Background

A recent **advancement** in gas chromatography (GC) **column deactivation** balances chromatographic performance of acidic, basic and neutral analytes at **trace level concentrat**ions. Deactivation is a critical step in GC column manufacturing that modifies the fused silica capillary surface activity to promote coating and adhesion. Residual surface silanols, which result from incomplete deactivation or surface contamination, adsorb acidic and basic compounds resulting in peak area loss or tailing.

Deactivation procedures vary between products and manufacturers, with individual benefits and drawbacks that become exacerbated during sensitive applications. There is increasing demand to improve identification and quantification of reactive analytes with complex or consolidated methodologies. New GC deactivation technology improves polymer adhesion and minimizes residual surface activity for a **long lasting, broadly inert surface**.

Columns built with the new GC deactivation technology were challenged with a wide variety of applications, and against traditional 5-type columns. Overall, the new deactivation presents an advancement over traditional GC columns, providing unparalleled inertness to a broader range of acids, bases, and neutrals. These improvements offer opportunity for **method consolidation and trace level analysis** that would not be successful with traditional technology.

Low-level calibration for sensitive instruments

Methods: 63 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 was assessed on **4 additional 5-type (silarylene) columns**, and linearity parameters (**R2, RRF RSD**) was 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.

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

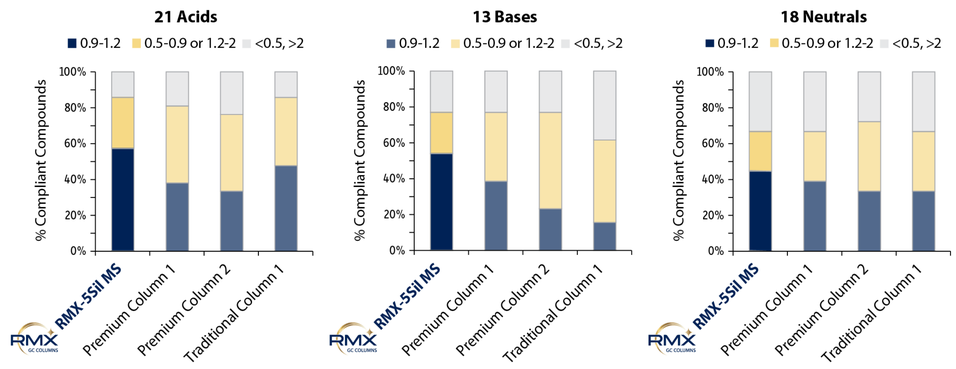


Figure 1: Proportion (%) of challenging compounds that meet peak shape quality criteria at 50ppb, on various GC columns.

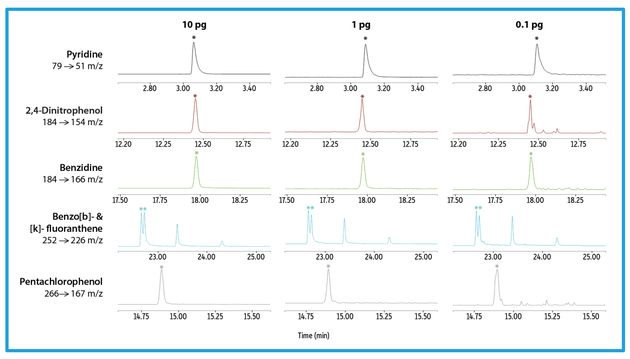


Figure 2: GC-MS/MS analysis of challenging acidic, basic, and neutral compounds associated with EPA 8270E at decreasing amounts on column.

2 **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.**

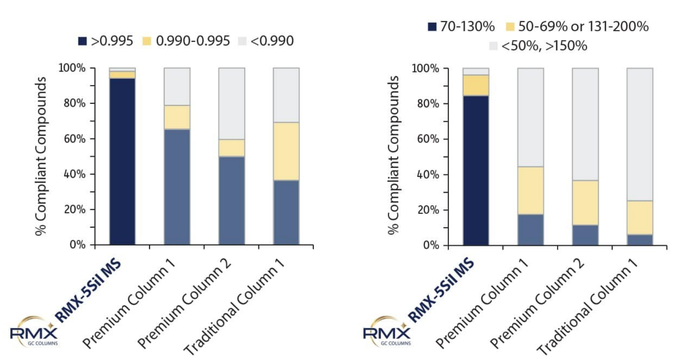


Figure 3: Proportion (%) of challenging compounds that meet EPA 8270E compliance criteria with low level GC-MS/MS calibrations, on various GC columns.

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

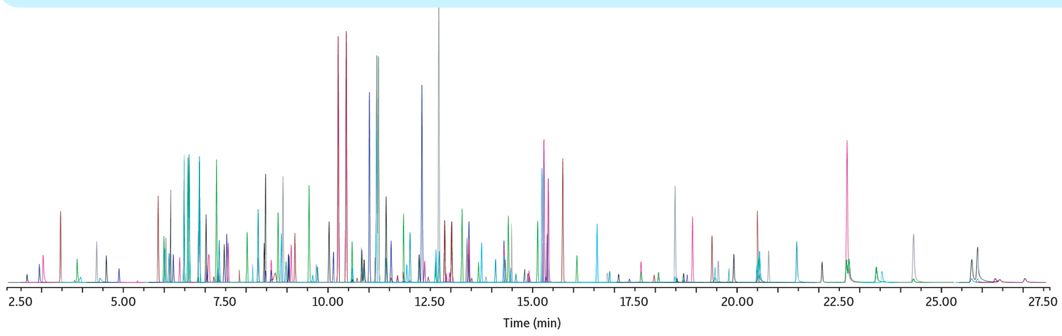
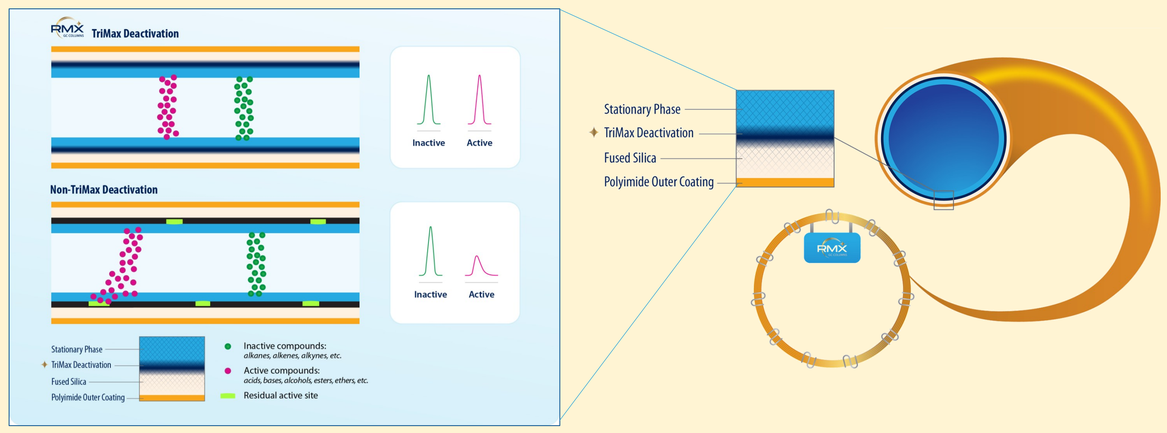


Figure 4: GC-MS/MS of over 150 semivolatiles (10 pg on column) comprised of acidic, basic, and neutral compounds with a variety of functionalities.

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

Column Lifetime and Variability

Methods: QC performance of **multiple** RMX-5Sil MS **lots** were evaluated and compared to demonstrate consistent performance throughout varying manufacturing conditions. An RMX-5Sil MS column was then challenged with **15 thermal stress cycles** wherein the column was heated to 330°C and held for four hours to expediate phase deterioration. QC probes were monitored before and after each thermal stress cycle to track column performance over time.

4 **Cleaner deactivation results in less variation during manufacturing. QC probes sensitive to surface silanols show consistent responses across multiple manufacturing batches.**

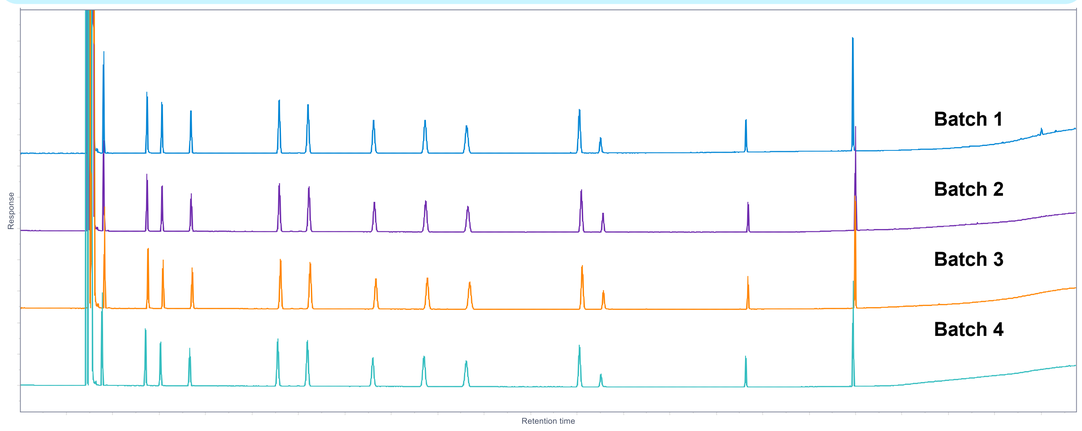


Figure 5: QC test chromatograms from four RMX-5Sil MS columns manufactured in different batches to comprise varying conditions.

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

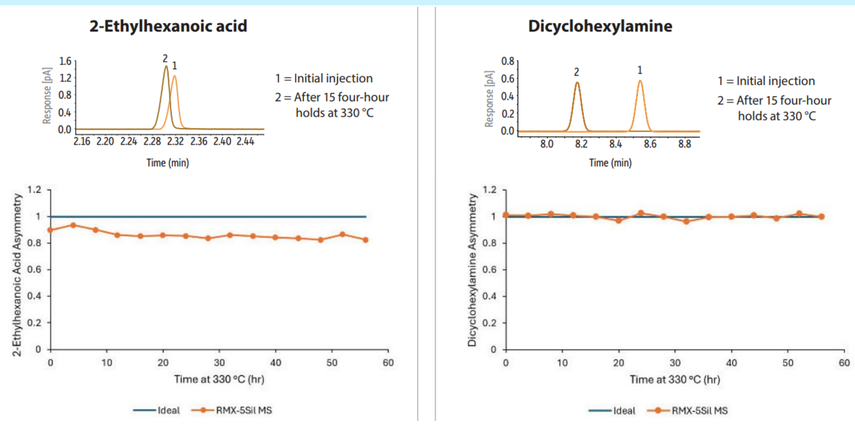


Figure 6: (Top) Peak shape of acidic (left) and basic (right) analytes before and after thermal stress cycling, with changes in peak symmetry monitored throughout stress cycling (bottom).

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