

# Achieving Lower Detection Limits for Semivolatiles

## Improve GC-MS/MS Sensitivity with Highly Inert RMX-5Sil MS Columns

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### Key Highlights

- Highly effective TriMax column deactivation produces an exceptionally inert sample flow path for acids, bases, and neutrals.
- Maximum inertness results in maximum sensitivity for a wide range of challenging semivolatiles.
- Compared to a premium competitor column, the RMX-5Sil MS column had a lower MDL for 60% and lower LLOQ for 63% of the 52 compounds tested.



### Abstract

In this study, we examined the impact of GC column deactivation on lowering detection limits for semivolatiles analysis when using GC-MS/MS. The MDLs and LLOQs generated using an RMX-5Sil MS column were found to be significantly lower for approximately two thirds of the compounds tested compared to results obtained on a premium competitor column. Better sensitivity was achieved for a wide range of compound chemistries due to the more effective deactivation used in RMX-5Sil MS columns.

### Introduction

Accurate quantification of semivolatile organic compounds at trace levels in environmental matrices is critical for assessing contamination, regulatory compliance, and risks to human and ecological health. GC-MS/MS is a cornerstone technique for lowering detection and quantitation limits for semivolatiles due to its high selectivity and sensitivity. Greater sensitivity allows labs to adopt alternative sample preparation methods that scale down extraction volumes and thereby reduce the use of chlorinated solvents. Method sensitivity is typically characterized by two key parameters: the method detection limit (MDL) and the lower limit of quantitation (LLOQ). The MDL represents the lowest concentration that can be distinguished from method blank results with 99% confidence.

In contrast, the LLOQ defines the lowest concentration at which a laboratory demonstrates that an analyte can be accurately quantified.

Lowering the MDL and LLOQ in GC-MS/MS semivolatiles methods involves optimizing multiple factors, including sample preparation, injection techniques, chromatographic conditions, and mass spectrometer parameters. In addition, the inertness of the sample flow path plays a critical role in lowering detection and quantitation limits for semivolatiles. GC column manufacturers use different deactivation processes to neutralize the column surface and prevent interactions that reduce sensitivity through unstable peak shapes, but traditional deactivations tend to work better for some compound classes than others. Restek has developed a next-generation TriMax deactivation used on all RMX columns that creates an exceptionally inert surface that is effective across a wide range of semivolatile compound chemistries.

This study compares the detection and quantitation limits that could be achieved with an RMX-5Sil MS column compared to another manufacturer's premium column by assessing MDL and LLOQ levels. Experiments were conducted using solvent-based standards instead of matrix in order to evaluate column performance specifically without the impact of sample handling and extraction.

### Related Products

- RMX-5Sil MS column 30 m, 0.25 mm ID, 0.25  $\mu$ m (cat.# 17323)
- Topaz 4 mm Precision inlet liner with wool (cat.# 23267)
- Restek electronic leak detector (cat.# 28500)
- 8270 Calibration Mix #1 (cat.# 31618)
- 8270 Calibration Mix #2 (cat.# 31619)
- 8270 Calibration Mix #5 (cat.# 31995)
- Acid Surrogate Mix (4/89 SOW) (cat.# 31025)
- Base Neutral Surrogate Mix (4/89 SOW) (cat.# 31024)
- Revised SV Internal Standard Mix (cat.# 31886)
- GC-MS Tuning Mix (cat.# 31615)

## Experimental

### Standard and Sample Preparation

Calibration standards were prepared in methylene chloride at 0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000, 2000, and 5000 ppb. For both columns, calibration curves were run on day 1 along with triplicate injections of the 0.5-100 ppb standards. The linear range of the calibration curve was determined for each semivolatile based on compound response. On days 2 and 3, fresh 0.5-100 ppb standards were prepared and injected in triplicate and used to determine the MDL and LLOQ for each semivolatile on each column.

### Instrument Conditions

Samples were run on an RMX-5Sil MS column and a competitor's premium column in a 30 m, 0.25 mm ID, 0.25  $\mu$ m format. A Thermo TRACE 1310 GC paired with a TSQ 8000 mass spectrometer was used for semivolatiles analysis under the conditions listed below.

Injection volume: 1  $\mu$ L

Liner: Topaz 4 mm Precision inlet liner with wool (cat.# 23267)

Injection port: 280  $^{\circ}$ C; 10:1 split; 1.2 mL/min

Carrier gas: helium

Oven: 40  $^{\circ}$ C (hold 1 min) to 280  $^{\circ}$ C at 12  $^{\circ}$ C/min to 310  $^{\circ}$ C at 3  $^{\circ}$ C/min

Detector: MS/MS; SRM mode; 280  $^{\circ}$ C transfer line temp; 330  $^{\circ}$ C source (see chromatogram for SRM transitions)

### Data Quality Evaluation

MDLs were calculated for each compound on each column by multiplying the standard deviation of the recalculated amount of its lowest calibration point by  $t=2.896$  ( $n=9$ ). LLOQ was determined from the same samples and defined as the lowest calibration point with a recovery between 80-120% (or closest % recovery available) for each semivolatile on each column.

## Results and Discussion

The MDL was lower on the RMX 5Sil MS column for 60% (31/52) of the compounds evaluated (Table I, Figure 1). Similarly, the LLOQ was lower on the RMX 5Sil MS column for 63% (33/52) of the target analytes (Table I, Figure 2). Individual results for each compound are presented in Table II. Lower MDL and LLOQ values mean that greater sensitivity can be achieved on the RMX-5Sil MS column, which can be attributed to the superior inertness produced by Restek's new surface deactivation technology. Notably, lower detection limits were achieved for a wide range of compound classes, including acidic, basic, and neutral semivolatiles when using the RMX-5Sil MS column.

**Table I:** Overall, highly inert RMX-5Sil MS columns produced lower LLOQs and MDLs for more compounds than a competitor's premium column, allowing lower detection limits for semivolatiles analysis.

Column	LLOQ (ppb)				RMX-5Sil Has Best Performance	MDL (ppb)			
	Average	Min	Max			Average	Min	Max	RMX-5Sil Has Best Performance
RMX-5Sil MS	14	1	100	33/52 compounds (63%)	33/52 compounds (63%)	1	0.1	14	31/52 compounds (60%)
Premium Competitor Column	19	1	100			2	0.1	51	

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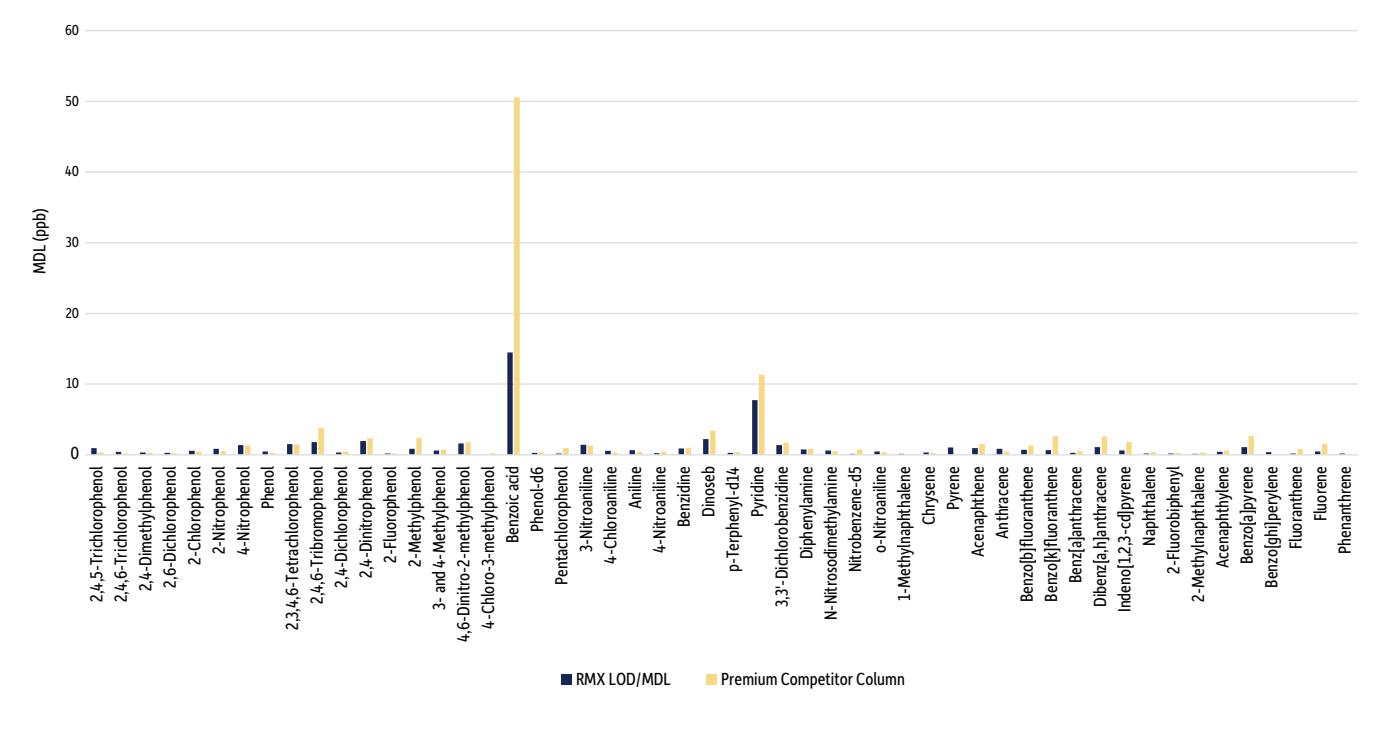


**Table II:** LOD and MDL Results for Individual Semivolatiles (Gold indicates superior RMX-5Sil MS performance.)

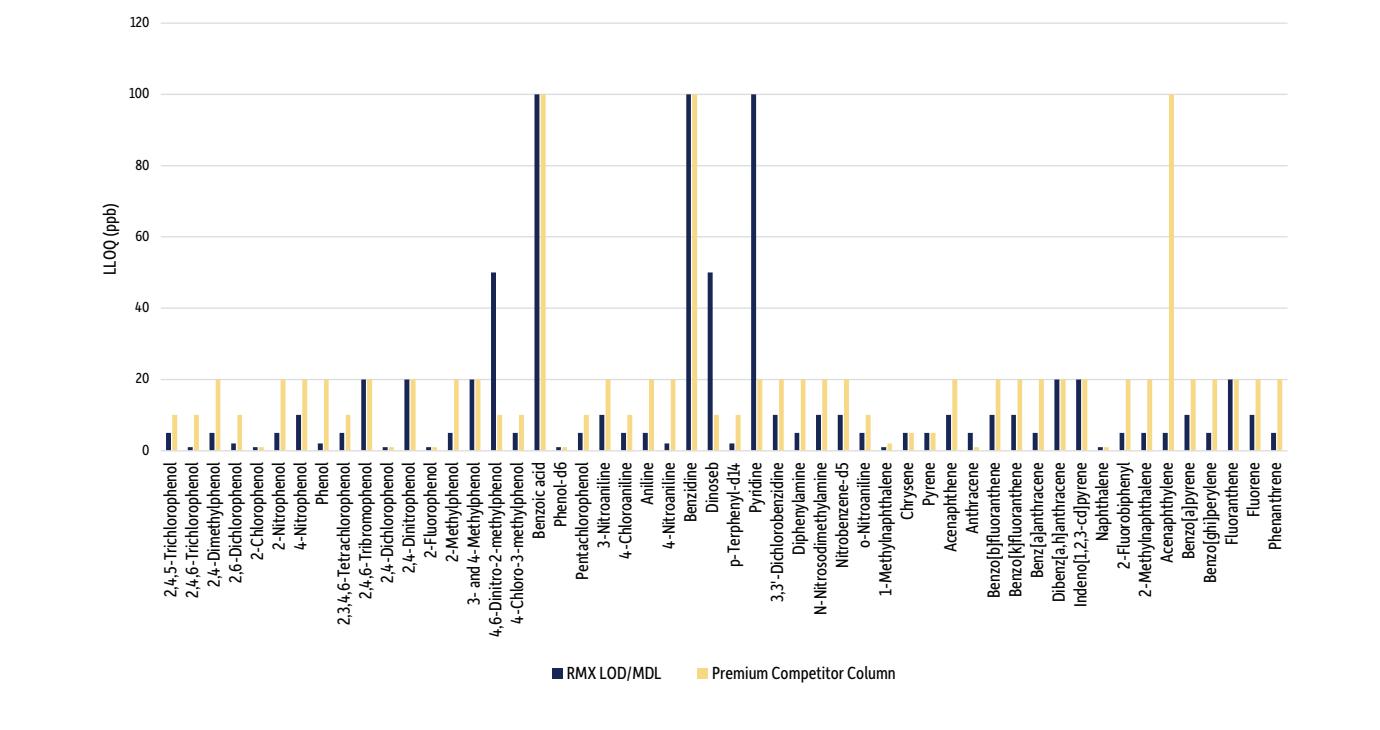
Compound	RMX-5Sil MS		Premium Competitor Column	
	LLOQ	MDL	LLOQ	MDL
Acenaphthylene	5	0.42	100	0.60
Phenol	2	0.45	20	0.27
4-Nitroaniline	2	0.22	20	0.43
2,4-Dimethylphenol	5	0.33	20	0.30
2-Nitrophenol	5	0.84	20	0.51
2-Methylphenol	5	0.84	20	2.37
Aniline	5	0.67	20	0.43
Diphenylamine	5	0.74	20	0.85
Benz[a]anthracene	5	0.29	20	0.49
2-Fluorobiphenyl	5	0.20	20	0.30
2-Methylnaphthalene	5	0.15	20	0.32
Benzo[ghi]perylene	5	0.39	20	0.08
Phenanthrene	5	0.19	20	0.15
4-Nitrophenol	10	1.38	20	1.31
3-Nitroaniline	10	1.40	20	1.33
3,3'-Dichlorobenzidine	10	1.37	20	1.76
N-Nitrosodimethylamine	10	0.63	20	0.53
Nitrobenzene-d5	10	0.13	20	0.74
Acenaphthene	10	0.93	20	1.53
Benzo[b]fluoranthene	10	0.71	20	1.31
Benzo[k]fluoranthene	10	0.67	20	2.65
Benzo[a]pyrene	10	1.08	20	2.63
Fluorene	10	0.48	20	1.54
2,4,6-Trichlorophenol	1	0.40	10	0.16
2,6-Dichlorophenol	2	0.30	10	0.21
p-Terphenyl-d14	2	0.30	10	0.36
2,4,5-Trichlorophenol	5	0.92	10	0.33

Compound	RMX-5Sil MS		Premium Competitor Column	
	LLOQ	MDL	LLOQ	MDL
2,3,4,6-Tetrachlorophenol	5	1.52	10	1.48
4-Chloro-3-methylphenol	5	0.06	10	0.21
Pentachlorophenol	5	0.18	10	0.93
4-Chloroaniline	5	0.55	10	0.32
o-Nitroaniline	5	0.49	10	0.36
1-Methylnaphthalene	1	0.13	2	0.05
2-Chlorophenol	1	0.55	1	0.47
2,4,6-Tribromophenol	20	1.79	20	3.79
2,4-Dichlorophenol	1	0.32	1	0.41
2,4-Dinitrophenol	20	1.94	20	2.34
2-Fluorophenol	1	0.20	1	0.20
3- and 4-Methylphenol	20	0.59	20	0.69
Benzoic acid	100	14.47	100	50.54
Phenol-d6	1	0.29	1	0.32
Benzidine	100	0.89	100	0.97
Chrysene	5	0.30	5	0.23
Pyrene	5	1.03	5	0.10
Dibenz[a,h]anthracene	20	1.06	20	2.58
Indeno[1,2,3-cd]pyrene	20	0.59	20	1.81
Naphthalene	1	0.18	1	0.35
Fluoranthene	20	0.16	20	0.80
Anthracene	5	0.86	1	0.45
4,6-Dinitro-2-methyl-phenol	50	1.59	10	1.80
Dinoseb	50	2.23	10	3.39
Pyridine	100	7.73	20	11.33

**Figure 1: MDL Comparison of Semivolatiles on an RMX-5Sil MS Column vs. a Premium Competitor Column (Compounds are sorted by groups of acids, bases, and neutrals.)**



**Figure 2: LLOQ Comparison of Semivolatiles on an RMX-5Sil MS Column vs. a Premium Competitor Column (Compounds are sorted by groups of acids, bases, and neutrals.)**



## Conclusion

An inert sample flow path allows for lower detection limits for semivolatiles, maximizing the high sensitivity that can be achieved with GC-MS/MS instruments. The work summarized here shows that exceptionally inert RMX-5Sil MS columns provide greater sensitivity than a competitor's premium column. Assessments of both MDL and LLOQ showed that lower limits of detection and quantification could be achieved for a wide range of compound chemistries on the RMX-5Sil MS column.

## Featured Products

### RMX-5Sil MS GC Capillary Column

Catalog No.	Product Name	Units
17323	RMX-5Sil MS GC Capillary Column, 30 m, 0.25 mm ID, 0.25 $\mu$ m	ea.



### Topaz Precision Inlet Liner

Catalog No.	Product Name	Units
23267	Topaz, Precision Inlet Liner, 4.0 mm x 6.3 x 78.5, for Thermo TRACE 1300/1310, 1600/1610 GCs w/SSL Inlets, w/Quartz Wool, Premium Deactivation	5-pk.



### Restek Electronic Leak Detector

Catalog No.	Product Name	Units
28500	Restek Electronic Leak Detector (includes carrying case; universal AC power adaptor [U.S., UK, Europe, Australia, Japan]; 6-ft USB charging cable)	ea.



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## 8270 Calibration Mix #1

2000 µg/mL, Methylene Chloride, 1 mL/ampul

Catalog No.	Contains	Units
31618	Benzoic acid (65-85-0)	2-Methylphenol (o-cresol) (95-48-7)
	4-Chloro-3-methylphenol (59-50-7)	3-Methylphenol (m-cresol) (108-39-4)
	2-Chlorophenol (95-57-8)	4-Methylphenol (p-cresol) (106-44-5)
	2,4-Dichlorophenol (120-83-2)	2-Nitrophenol (88-75-5)
	2,6-Dichlorophenol (87-65-0)	4-Nitrophenol (100-02-7)
	2,4-Dimethylphenol (105-67-9)	Pentachlorophenol (87-86-5)
	4,6-Dinitro-2-methylphenol (Dinitro-o-cresol) (534-52-1)	Phenol (108-95-2)
	2,4-Dinitrophenol (51-28-5)	2,3,4,6-Tetrachlorophenol (58-90-2)
	Dinoseb (88-85-7)	2,4,5-Trichlorophenol (95-95-4)
		2,4,6-Trichlorophenol (88-06-2)

## 8270 Calibration Mix #2

2000 µg/mL, Methylene Chloride:Methanol (85:15), 1 mL/ampul

Catalog No.	Contains	Units
31619	Aniline (62-53-3)	3-Nitroaniline (99-09-2)
	Benzidine (92-87-5)	4-Nitroaniline (100-01-6)
	4-Chloroaniline (106-47-8)	N-Nitrosodimethylamine (62-75-9)
	3,3'-Dichlorobenzidine (91-94-1)	N-Nitroso-di-n-propylamine (621-64-7)
	Diphenylamine (122-39-4)*	Pyridine (110-86-1)
	2-Nitroaniline (88-74-4)	

\*N-Nitrosodiphenylamine is a reactive species that can initiate premature decomposition of other compounds in the mix. For these reasons, diphenylamine is used in the preparation of this mixture.

## 8270 Calibration Mix #5

2000 µg/mL, Methylene Chloride, 1 mL/ampul

Catalog No.	Contains	Units
31995	Acenaphthene (83-32-9)	Dibenz[a,h]anthracene (53-70-3)
	Acenaphthylene (208-96-8)	Fluoranthene (206-44-0)
	Anthracene (120-12-7)	Fluorene (86-73-7)
	Benz[a]anthracene (56-55-3)	Indeno[1,2,3-cd]pyrene (193-39-5)
	Benzo[a]pyrene (50-32-8)	1-Methylnaphthalene (90-12-0)
	Benzo[b]fluoranthene (205-99-2)	2-Methylnaphthalene (91-57-6)
	Benzo[g,h,i]perylene (191-24-2)	Naphthalene (91-20-3)
	Benzo[k]fluoranthene (207-08-9)	Phenanthrene (85-01-8)
	Chrysene (218-01-9)	Pyrene (129-00-0)

**Acid Surrogate Mix (4/89 SOW)**

2000 µg/mL, Methanol, 1 mL/ampul

Catalog No.	Contains	Units
31025	2-Fluorophenol (367-12-4) Phenol-d6 (13127-88-3) 2,4,6-Tribromophenol (118-79-6)	ea.

**Base Neutral Surrogate Mix (4/89 SOW)**

1000 µg/mL, Methylene Chloride, 1 mL/ampul

Catalog No.	Contains	Units
31024	2-Fluorobiphenyl (321-60-8) Nitrobenzene-d5 (4165-60-0) p-Terphenyl-d14 (1718-51-0)	ea.

**Revised SV Internal Standard Mix**

4000 µg/mL, Methylene Chloride, 1 mL/ampul

Catalog No.	Contains	Units
31886	Acenaphthene-d10 (15067-26-2) Chrysene-d12 (1719-03-5) 1,4-Dichlorobenzene-d4 (3855-82-1) 1,4-Dioxane-d8 (17647-74-4) Naphthalene-d8 (1146-65-2) Perylene-d12 (1520-96-3) Phenanthrene-d10 (1517-22-2)	ea.

**GC-MS Tuning Mix**

1000 µg/mL, Methylene Chloride, 1 mL/ampul

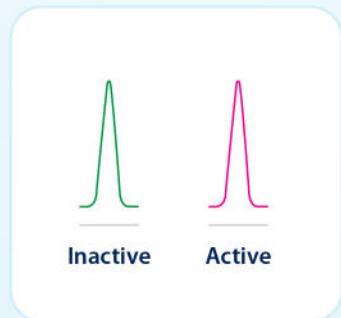
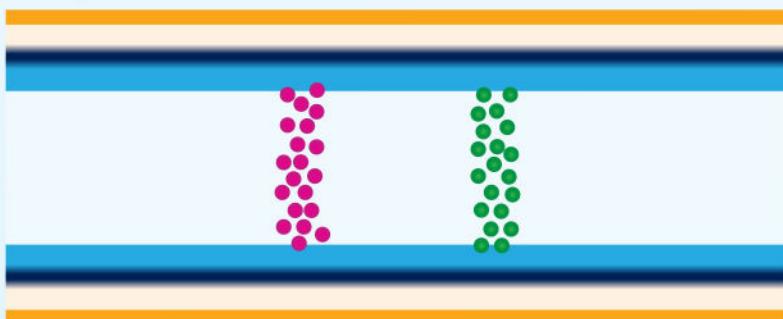
Catalog No.	Contains	Units
31615	Benzidine (92-87-5) 4,4'-DDT (50-29-3) DFTPP (decafluorotriphenylphosphine) (5074-71-5) Pentachlorophenol (87-86-5)	ea.

# What Makes RMX Columns Better?

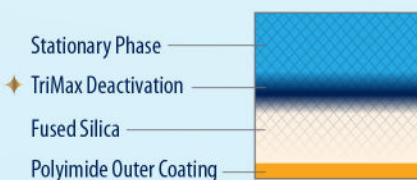
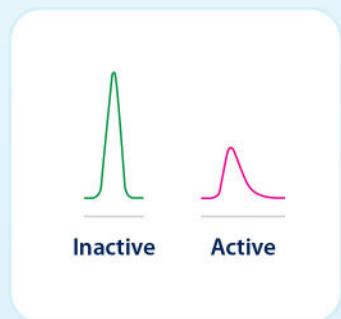
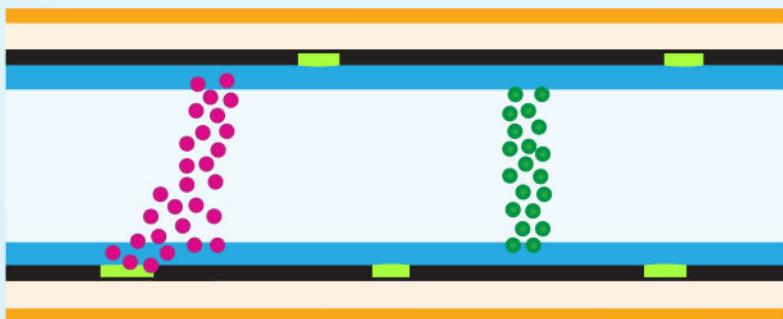
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## TriMax Deactivation



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- Active compounds: acids, bases, alcohols, esters, ethers, etc.
- Residual active site

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