

GC Inlet Liner Deactivations for Basic Drug Analysis

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- Base-deactivated inlet liners are inert to basic drugs, for greater responses.
- Inertness of Rtx®-5Amine column is enhanced for basic compounds.
- Use this liner / column combination for the lowest %RSDs for basic drugs.

Clinical and forensic toxicologists are required to detect low levels of abused drugs in body fluids and confirm their presence by GC/MS. Typical limits of detection are 1-15ng/mL, depending on the sample matrix. For basic drugs (e.g., Figure 1), selecting the proper surface treatment for the GC inlet liner is important, because this parameter can affect responses. The surface of a glass inlet liner contains active silanol groups (Si-OH) that can act as electron pair acceptors, and react with nitrogen or oxygen electron pair donors in basic drug molecules (Figure 2).¹ These reactions usually are rapid and reversible, but they are expressed chromatographically as broad, tailing peaks and/or reduced responses. To eliminate these acid-base reactions, make chromatographic peaks sharp, Gaussian, and easy to integrate, and thereby help ensure reproducible and accurate responses, the -OH groups on the glass surface must be deactivated.

Using GC/FID responses, we evaluated several alternatives for deactivating inlet liners, to determine maximum sensitivity for basic drugs. We prepared reference standards of the free base forms of alprazolam, benzphetamine, cocaine, codeine, ketamine, methadone, and phencyclidine (Figure 1) at 100, 50, 25, 10, and 5 ng/mL concentrations, then analyzed the drugs on a base-deactivated 15m, 0.25mm ID, 0.25µm Rtx®-5Amine column (5% diphenyl/95% dimethylpolysiloxane stationary phase), using a 4mm single gooseneck inlet liner that was untreated, deactivated through an intermediate polarity deactivation process (standard liner deactivation procedure), deactivated through a base deactivation process, or deactivated through the Siltek® deactivation process. We obtained three replicate analyses for each reference standard-liner treatment combination, and evaluated the response data statistically to determine which deactivation treatment maximized sensitivity and reproducibility. We used these results to generate box plots that display the range of data distribution, or variation – an indication of the reproducibility of the performance. We chose phencyclidine (PCP) and cocaine plots to represent the nitrogen-containing and nitrogen/oxygen-containing drugs, respectively (Figure 2). The line in each box indicates the mean response.

Figure 1 Nitrogen- and oxygen-containing compounds can react with silanol groups on glass surfaces, causing poor chromatography.

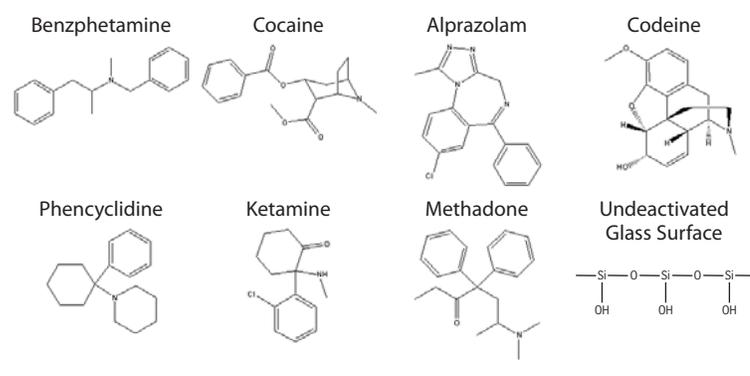


Figure 2 A base-deactivated inlet liner provides highest mean responses for PCP.

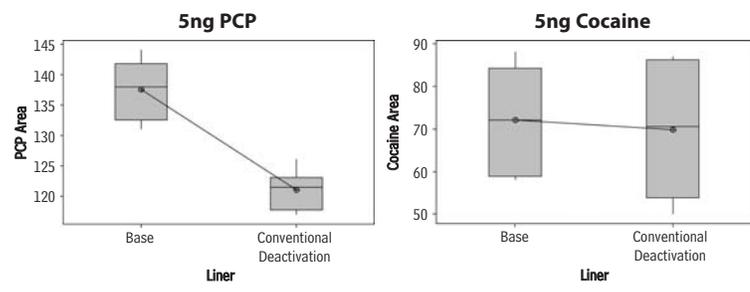
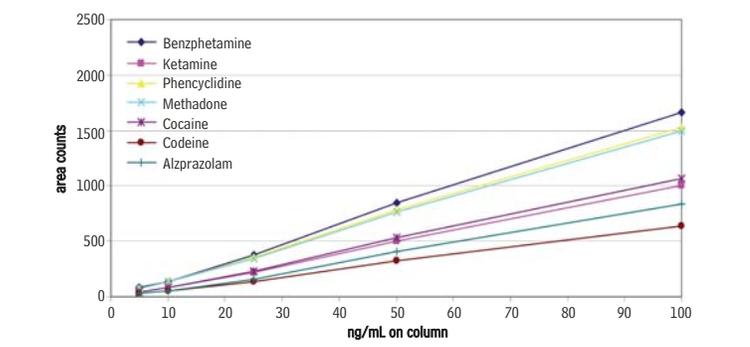


Figure 3 Linearity plots for all drugs, analyzed using a base-deactivated inlet liner and an Rtx®-5Amine column.



Base Deactivated Inlet Liners for Basic Drug Analysis

For Agilent GCs	ea.	cat.#/price	
		5-pk.	25-pk.
Gooseneck Splitless (4.0mm ID** x 6.5mm OD x 78.5mm)			
	20798-210.1	20799-210.5	20800-210.25
	\$43	\$126	\$474
Gooseneck Splitless w/ Base Deactivated Wool (4.0mm ID** x 6.5mm OD x 78.5mm)			
	20798-211.1	20799-211.5	20800-211.25
	\$46	\$141	\$509
Split Straight w/ Base Deactivated Wool (4.0mm ID** x 6.3mm OD x 78.5mm)			
	20781-211.1	20782-211.5	20783-211.25
	\$40	\$127	\$454
Cycloplitter® (4.0mm ID** x 6.3mm OD x 78.5mm)			
	20706-210.1	20707-210.5	20708-210.25
	\$57	\$214	\$742

**Nominal ID at syringe needle expulsion point.

For liners for other instruments, refer to our catalog or website.

qty.	Base-Deactivated			Base-Deactivated w/ Base-Deactivated Wool		
each	-210.1	\$14	addl. cost	-211.1	\$17	addl. cost
5-pk.	-210.5	\$45	addl. cost	-211.5	\$60	addl. cost
25-pk.	-210.25	\$145	addl. cost	-211.25	\$180	addl. cost

Description	qty.	cat.#	price
Base-Deactivated Wool	10 grams	20999	\$51



Description	qty.	cat.#	price
Mini Wool Puller/Inserter	2-pk.	20114	\$14



Description	qty.	cat.#	price
Inlet Liner Removal Tool	3-pk.	20181	\$26



ID	df (µm)	temp. limits	length	cat. #	price
0.25mm	0.25	-60 to 300/315°C	30-Meter	12323	\$480
0.25mm	0.25	-60 to 300/315°C	15-Meter	12320	\$315



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The data show that undeactivated liners and liners that received intermediate polarity treatment provided poorer responses or reproducibility, compared to base-deactivated or Siltek® treated liners, due to the acidic nature of the undeactivated glass surface or to a small but influential number of residual acidic sites remaining on the intermediate polarity deactivated surface.

Because the undeactivated liners and intermediate polarity treated liners exhibited either low mean response or high variation, we reanalyzed the data, excluding these treatments and comparing the remaining data (for base-deactivated liners and Siltek® treated liners) for responses and reproducibility. As shown by the examples in Figure 2, base-deactivated liners and Siltek® treated liners performed equally well for cocaine, but the base-deactivated liners yielded the best responses and reproducibility for PCP. Ultimately, a base-deactivated liner would give the best overall performance. Figure 3 shows the linearity plots for all analyzed drugs, obtained using a base-deactivated liner and an Rtx®-5Amine column. Low %RSD values for ketamine (3%), phencyclidine (2%), methadone (2%), cocaine (3%), codeine (5%), and alprazolam (12%) confirm the reproducibility of data obtained from this combination.

Because nitrogen- and oxygen-containing drugs react with silanol groups on glass surfaces, it is important to use properly deactivated glass inlet liners when analyzing these compounds by GC. This work demonstrates that a base-deactivated inlet liner, used in combination with a base-deactivated column, produces high and reproducible responses for basic drugs.

Reference

1. Seyhan N. and D.C. Ege, *Organic Chemistry Health and Company*, 1984, pp.124-136.

recommended reading

Forensic Applications of Mass Spectrometry

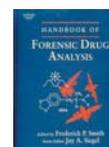
Applies current developments in mass spectrometry to forensic analyses. Techniques discussed include capillary GC/MS, thermospray LC/MS, tandem mass spectrometry, (MS/MS), pyrolysis GC/MS and isotope ratio mass spectrometry.



J. Yinon, CRC Press LLC, 1994, 320pp., ISBN 0-8493-8252-1
cat.# 23056 (ea.) \$169.95

Handbook of Forensic Drug Analysis

Provides in-depth, up-to-date methods and results. Chapters by leading researchers discuss the various forms of drugs, as well as the origin and nature of samples.



F. Smith and J. Siegel, Elsevier Academic Press, 2004, 584pp., ISBN 0-12-650641-8 cat.# 23055 (ea.) \$140