Rtx-Wax: A Rugged and Reliable Choice for the Analysis of Glycols in Water

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- Rugged enough to withstand repeated aqueous injections.
- Maintains symmetrical peaks even after 600 water injections.
- Ultra-low bleed allows for accurate detection down to 0.5 ng of glycols.

Introduction

From airplane and runway deicing to hydraulic fracturing operations, glycols are common chemicals used in industry, and as a result, chemical and environmental laboratories often test samples for their presence. However, the most frequently used sample introduction technique—direct aqueous injection—puts a significant demand on the analytical column and injection port liner. The columns best suited for water injections employ polyethylene glycol (PEG) stationary phases. PEG phases offer a unique selectivity, and their polar nature is compatible with an aqueous injection. An evaluation of ethylene glycol (EG) and propylene glycol (PG) response on PEG columns from a variety of vendors was conducted under optimized conditions and is presented here.

Benchmarking

Three different columns were evaluated in this study for linearity, lifetime, and bleed using split injection under the conditions shown in the chromatograms. The optimized split injection method employed here is further detailed in application note EVAN2873, which is available at www.restek.com.

- **Restek Columns**
  - Rtx-Wax (cat.# 12455)
- **Non-Restek Columns**
  - Column A
  - Column B

All columns used in this study for the analysis of glycols in water were new and of a 30 m, 0.53 mm, 1.0 µm format. They were all conditioned for an hour at their labeled maximum temperature.

Results

The results of the benchmarking study for the analysis of glycols in water are summarized in Table I and discussed in detail below.

Table I: Column Benchmarking Results

Columns	LIFETIME			LINEARITY		BLEED
	# of Passing Injections*	Peak Symmetry (Final Injection)		Final r^2		FID Response (pA) at 240 °C
		EG	PG	EG	PG	
	600	0.99	0.92	0.9999	0.9999	29
Column A	*Lower calibration limits not met, so lifetime studies not performed*					53
Column B	*Lower calibration limits not met, so lifetime studies not performed*					60

*The experiment was designed to determine the number of passing injections up to 600 injections, at which point the experiment was terminated. Passing injections were defined as injections with ChemStation symmetry values >0.5. All injections were 1 ng on-column.

Linearity

For each column, an initial calibration curve from 0.5 ng to 100 ng on-column was collected to establish suitable linearity for the analysis of glycols in water and to evaluate peak shape at different concentrations. The two lowest levels of the calibration curve, 0.5 and 1.0 ng on-column, were not detected on competitor columns A and B because the column bleed was too high to observe EG and PG at those levels. These results disqualified columns A and B from the subsequent lifetime study.

In contrast, excellent linearity was obtained on the Rtx-Wax column for both EG and PG (Figures 1 and 2). Even after the punishing conditions of 600 splitless aqueous injections, which were used to exacerbate the potentially column-damaging effects of water, the Rtx-Wax column would easily pass a calibration check. The high response for EG and PG at low levels, which is a function of column's low bleed characteristic, is one factor that contributed to obtaining good linearity results across the entire calibration range.

Figure 1: Linear responses were obtained for propylene glycol (0.5–100 ng on-column) analyzed on an Rtx-Wax column even after 600 water injections.

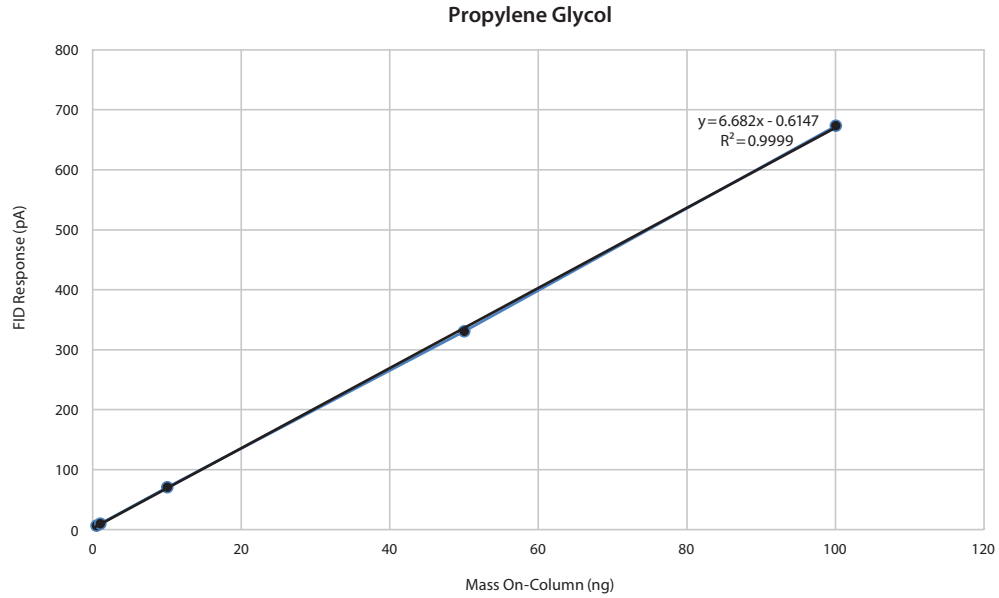
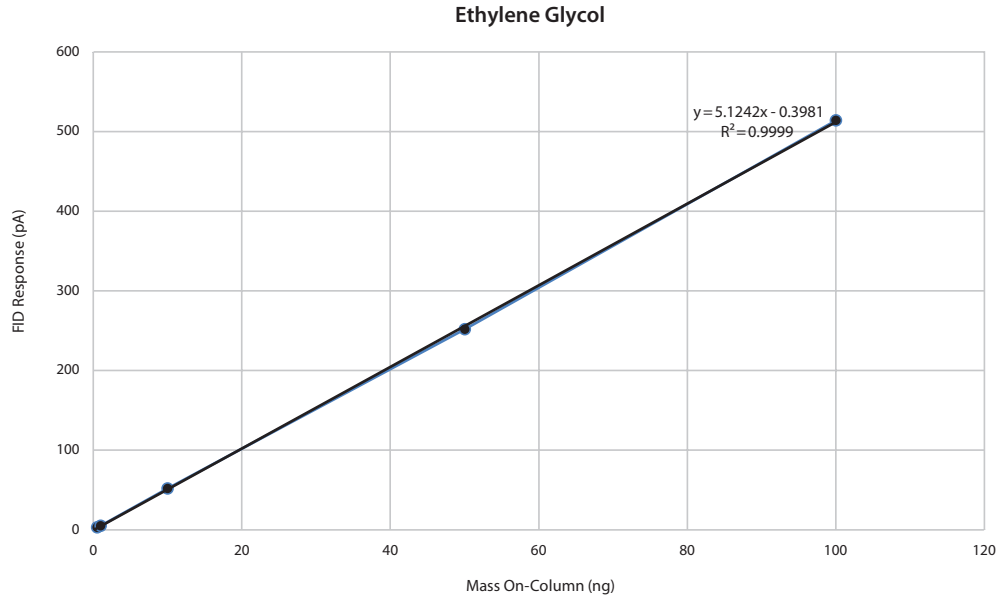


Figure 2: Linear responses were obtained for ethylene glycol (0.5–100 ng on-column) analyzed on an Rtx-Wax column even after 600 water injections.

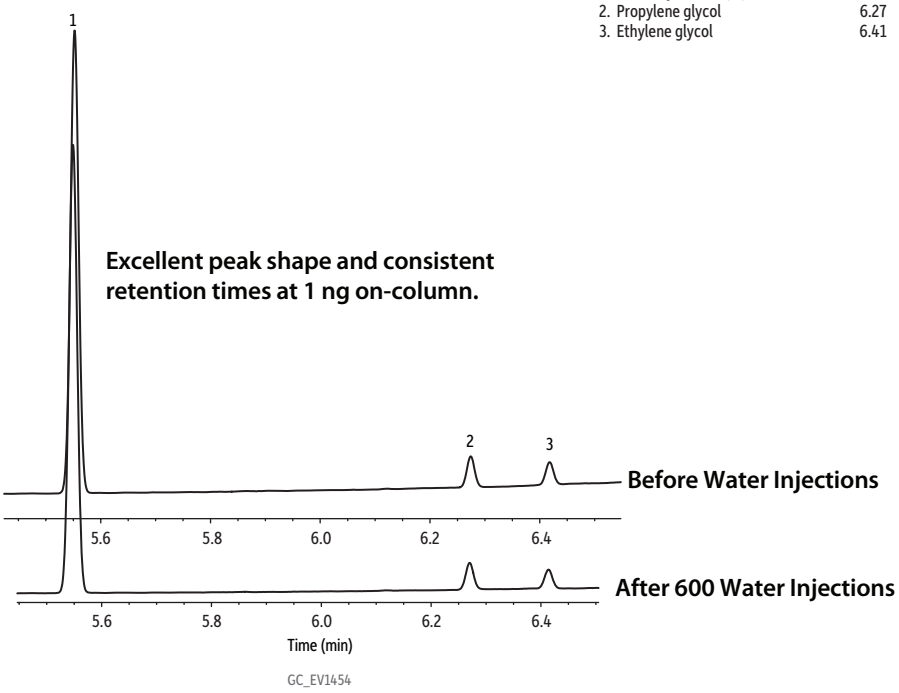


Lifetime

For the Rtx-Wax column, the lifetime study consisted of ten 1 µL splitless injections of water followed by a 50:1 split injection of a glycols standard, delivering 1 ng on-column. The experiment was designed to repeat until the peak symmetry (values determined using ChemStation software) dropped below 0.5 or the column reached 600 water injections, whichever came first. As shown in Figure 3, the Rtx-Wax column maintained excellent peak shape, even after 600 injections. In fact, one Rtx-Wax column was subjected to more than 1600 injections in an extended lifetime experiment, and it still exhibited symmetry values greater than 0.9 at the end of the experiment.

Figure 3: Propylene glycol and ethylene glycol peak shapes and retention times on an Rtx-Wax column are virtually identical, even after the column was exposed to 600 water injections.

Peaks	t_R (min)
1. 2-Butoxyethanol (IS)	5.55
2. Propylene glycol	6.27
3. Ethylene glycol	6.41

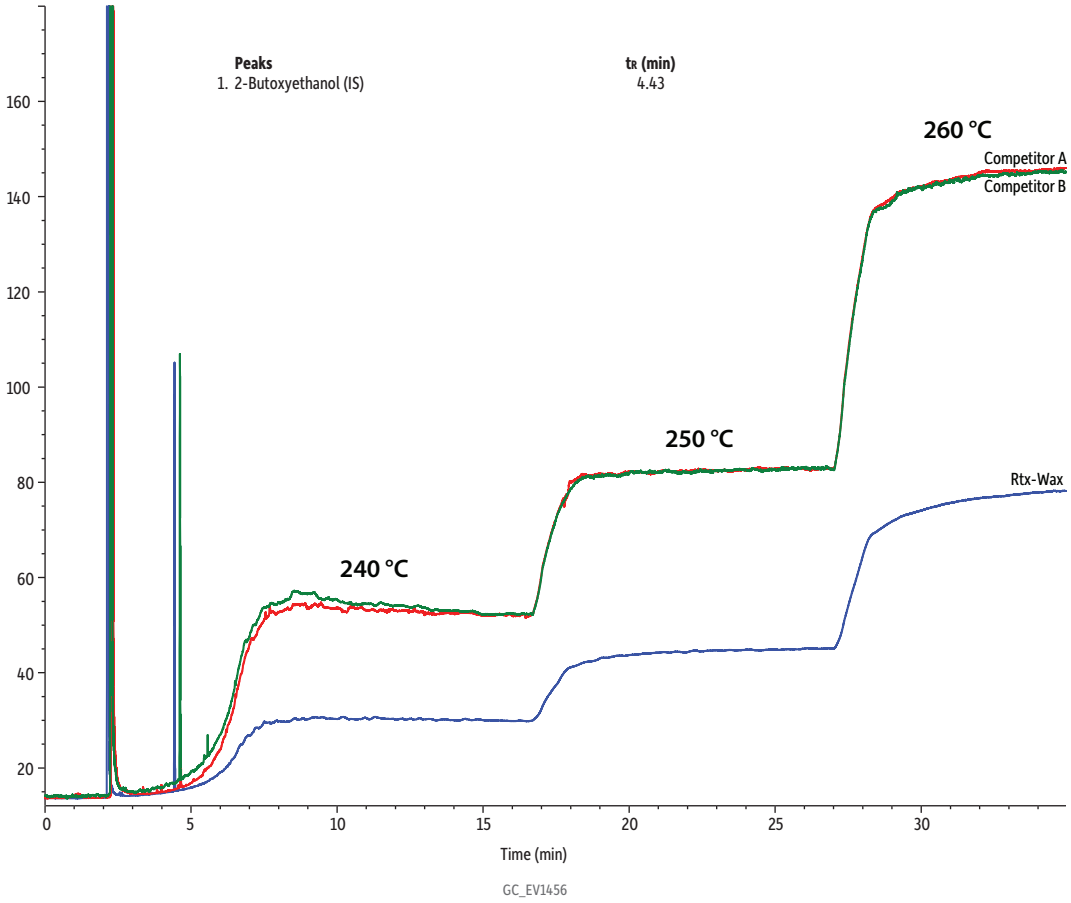


Column	Rtx-Wax, 30 m, 0.53 mm ID, 1.00 µm (cat.# 12455)
Sample	Glycols standard (cat.# 30471) 2-Butoxyethanol
Diluent:	Water:methanol (90:10)
Conc.:	50 µg/mL (1 ng on-column)
Injection	
Inj. Vol.:	1 µL split (split ratio 50:1)
Liner:	Premium 4 mm Precision inlet liner w/wool (cat.# 23305.1)
Inj. Temp.:	250 °C
Oven	
Oven Temp.:	40 °C (hold 1 min) to 250 °C at 30 °C/min
Carrier Gas	He, constant flow
Flow Rate:	5.7 mL/min
Linear Velocity:	40 cm/sec
Detector	FID @ 250 °C
Make-up Gas Flow Rate:	45 mL/min
Make-up Gas Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC

Bleed

The final test in our evaluation of PEG columns for the analysis of glycols in water was a bleed experiment. All three columns were ramped to a final temperature of 240 °C, which was the commonly achievable maximum temperature among the columns tested. The final bleed measurements showed the Rtx-Wax exhibited the lowest bleed (Table I, Figure 4). However, the maximum operating temperature for the Rtx-Wax is higher (250 °C), so it can be used with confidence when higher temperatures are required. Low column bleed allows for better sensitivity at low analyte concentrations.

Figure 4: The Rtx-Wax column exhibits the lowest bleed of the columns tested, even at temperatures that exceeded the recommended maximums (240 °C for competitor columns, 250 °C for Rtx-Wax column).



Column	Rtx-Wax, 30 m, 0.53 mm ID, 1.00 µm (cat.# 12455)
Sample	2-Butoxyethanol
Diluent:	Water:methanol (90:10)
Conc.:	200 µg/mL (10 ng on-column)
Injection	
Inj. Vol.:	1 µL split (split ratio 20:1)
Liner:	Premium 4.0 mm ID Precision inlet liner w/wool (cat.# 23305.1)
Inj. Temp.:	240 °C
Oven	
Oven Temp.:	40 °C to 240 °C at 30 °C/min (hold 10 min) to 250 °C at 30 °C/min (hold 10 min) to 260 °C at 30 °C/min (hold 10 min)
Carrier Gas	He, constant flow
Linear Velocity:	40 cm/sec
Detector	FID @ 240 °C
Make-up Gas Flow Rate:	45 mL/min
Make-up Gas Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC
Notes	All column dimensions: 30 m, 0.53 mm ID, 1.00 µm

Conclusion

Of the polyethylene glycol-based columns evaluated in this study, the Rtx-Wax performed best in terms of linearity, lifetime, and bleed, demonstrating that even after exposure to the aggressive conditions of 600 aqueous injections it performed as well as the first injection. The Rtx-Wax is a rugged and reliable solution for the analysis of glycols in water.

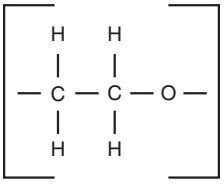
Rtx-Wax Columns (fused silica)
polar phase; Crossbond polyethylene glycol

- Best polyethylene glycol (PEG) phase for alkenols, glycols, and aldehydes.
- Temperature range: 20 °C to 250 °C.
- Equivalent to USP G14, G15, G16, G20, G39 phases.

Description	temp. limits	qty.	cat.#
Rtx-Wax 20 m, 0.10 mm ID, 0.10 µm	20 to 250 °C	ea.	41602
Rtx-Wax 10 m, 0.10 mm ID, 0.20 µm	20 to 250 °C	ea.	41603
Rtx-Wax 20 m, 0.10 mm ID, 0.20 µm	20 to 250 °C	ea.	41604
Rtx-Wax 15 m, 0.25 mm ID, 0.25 µm	20 to 250 °C	ea.	12420
Rtx-Wax 30 m, 0.25 mm ID, 0.25 µm	20 to 250 °C	ea.	12423
Rtx-Wax 60 m, 0.25 mm ID, 0.25 µm	20 to 250 °C	ea.	12426
Rtx-Wax 15 m, 0.25 mm ID, 0.50 µm	20 to 250 °C	ea.	12435
Rtx-Wax 30 m, 0.25 mm ID, 0.50 µm	20 to 250 °C	ea.	12438
Rtx-Wax 60 m, 0.25 mm ID, 0.50 µm	20 to 250 °C	ea.	12441
Rtx-Wax 30 m, 0.32 mm ID, 0.25 µm	20 to 250 °C	ea.	12424
Rtx-Wax 60 m, 0.32 mm ID, 0.25 µm	20 to 250 °C	ea.	12427
Rtx-Wax 15 m, 0.32 mm ID, 0.50 µm	20 to 250 °C	ea.	12436
Rtx-Wax 30 m, 0.32 mm ID, 0.50 µm	20 to 250 °C	ea.	12439
Rtx-Wax 60 m, 0.32 mm ID, 0.50 µm	20 to 250 °C	ea.	12442
Rtx-Wax 15 m, 0.32 mm ID, 1.00 µm	20 to 240/250 °C	ea.	12451
Rtx-Wax 30 m, 0.32 mm ID, 1.00 µm	20 to 240/250 °C	ea.	12454
Rtx-Wax 60 m, 0.32 mm ID, 1.00 µm	20 to 240/250 °C	ea.	12457
Rtx-Wax 30 m, 0.53 mm ID, 0.25 µm	20 to 250 °C	ea.	12425
Rtx-Wax 30 m, 0.53 mm ID, 0.50 µm	20 to 250 °C	ea.	12440
Rtx-Wax 15 m, 0.53 mm ID, 1.00 µm	20 to 240/250 °C	ea.	12452
Rtx-Wax 30 m, 0.53 mm ID, 1.00 µm	20 to 240/250 °C	ea.	12455
Rtx-Wax 60 m, 0.53 mm ID, 1.00 µm	20 to 240/250 °C	ea.	12458

similar **phases**

DB-Wax, CP-Wax 52 CB, ZB-Wax



Glycols Standard (2 components)

Certified reference materials (CRMs) manufactured and QC-tested in ISO-accredited labs satisfy your ISO requirements.

Ethylene glycol (107-21-1)
Propylene glycol (57-55-6)

50,000 µg/mL each in DI water, 1 mL/ampul — cat.# 30471 (ea.)

Topaz 4.0 mm ID Precision Inlet Liner w/ Wool
for Agilent GCs equipped with split/splitless inlets

ID x OD x Length	qty.	cat.#
Precision, Premium Deactivation, Borosilicate Glass with Quartz Wool Similar to: Agilent 210-4004-5		
4.0 mm x 6.3 mm x 78.5 mm	5-pk.	23305

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