

THE RESTEK ADVANTAGE

Turning Visions into Reality™

2005 vol. 3

Versatile GC Columns for Forensics

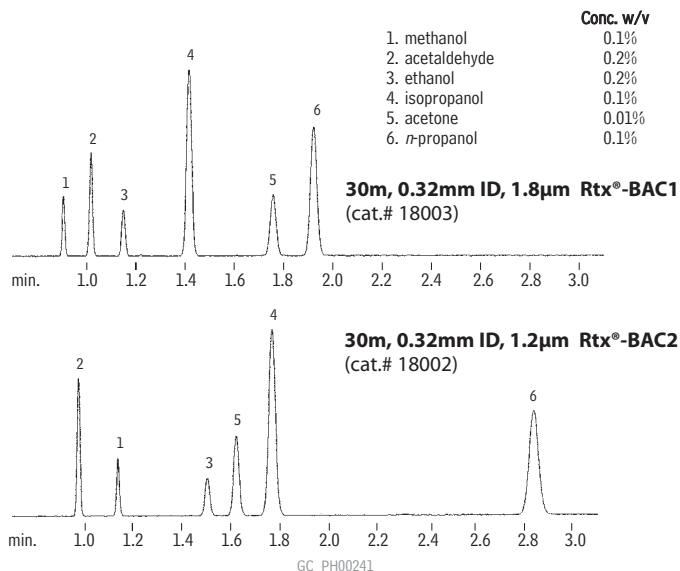
Use an Rtx®-BAC1 / Rtx®-BAC2 Column Set To Evaluate Blood Alcohol, Abused Substances, and Other Materials

By Kristi Sellers, Clinical/Forensic Innovations Chemist

- Unique column set for resolving/ confirming abused substances.
- 3-minute analysis for blood alcohols.
- Reliable data for ethylene glycol or GHB.

Analytical toxicology laboratories assist in criminal investigations by performing analyses for abused substances or poisons. Additional responsibilities include testing for accelerants or explosive materials in investigations of fires or explosions and assessing occupational/environmental exposure. Volatile and non-volatile compounds commonly tested for include blood alcohols, alkyl nitrites, anesthetics, inhalants, glycols, gamma-hydroxybutyrate (GHB), industrial solvents, petroleum hydrocarbons, and nitrogen-containing explosives.

Figure 1 Baseline resolution of all blood alcohol components in less than 3 minutes, using Rtx®-BAC1 and Rtx®-BAC2 columns.



Dual-column analysis using a two-hole ferrule.
1.0mL headspace sample of a blood alcohol mix on a PerkinElmer HS 40 headspace autosampler

Oven temp.:	40°C	Vial sampling time:	0.01 min.
Inj. temp.:	200°C	Transfer line:	0.32mm ID Hydroguard™ fused silica tubing
Carrier gas:	helium	Transfer line temp.:	200°C
Sample equilibration:	70°C, 15 min.	Injection port sleeve:	2mm ID
Vial pressure:	30psi	Split flow:	20mL/min.
Vial pressurization time:	0.15 min.		

in this issue

Versatile GC Columns for Forensics	1
Optimized, 17-Minute GC Analysis of Semivolatiles	5
High-Speed Detailed Hydrocarbon Analysis	8
Rapidly Determine Benzene and Toluene in Gasolines	10
Rapid, Dual Column Analysis for Organochlorine Pesticides	11
New Analytical Reference Materials for Fuels, Accelerants	12
Superior Moisture Dry-Down and Corrosion Resistance	14
Simple HPLC Analysis for Sudan Dyes	16
Enhanced Electronic Leak Detector	17
Genuine Restek HPLC Parts and Accessories	18
Genuine Restek Supplies & Accessories for ASE® Systems	20
Syringe Filters	21
Bulk Adsorbents	21
Instrument Innovations	22
Cool Tools	23
www.restek.com/expert	24

Where We'll Be in October

October 17-21, 2005

Society of Forensic Toxicologists (SOFT), Renaissance Nashville Hotel, Nashville, TN, booth #19

October 18-20, 2005

Gulf Coast Conference, Moody Gardens Convention Center, Galveston Island, TX

Help Us Celebrate Our 20th Birthday! Visit us at Booth 707, and receive a FREE Restek 20th Anniversary Travel Mug and Tote Bag!

October 25-27, 2005

ISA Expo 2005, McCormick Place Lakeside Center, Chicago, IL, booth #1322

Correction

In *Advantage 2005v2*, page 11: Analysis of Nitrofurans in Honey. Mobile phase components A and B contain acetic acid, not formic acid. Honey samples containing nitrofurans metabolites were dissolved in 125mM HCl, derivatized with 2-nitrobenzaldehyde, extracted, evaporated, and reconstituted with mobile phase.

Table I Retention Times for Abused Substances

Compound	Rtx®-BAC1		Rtx®-BAC2	
	Elution Order	Ret. Time (min.)	Elution Order	Ret. Time (min.)
methanol	1	1.017	5	1.237
acetaldehyde	2	1.146	1	1.063
ethyl chloride	3	1.275	2	1.071
ethanol	4	1.299	8	1.648
diethyl ether	5	1.574	4	1.167
isopropanol	6	1.607	15	1.945
isoflurane	7	1.661	13	1.922
methylene chloride	8	1.805	11	1.849
Freon® 113	9	1.864	3	1.145
enflurane	10	1.891	16	2.081
<i>tert</i> -butyl alcohol	11	1.926	17	2.154
acetone	12	1.992	10	1.787
acetonitrile	13	1.997	20	2.553
<i>n</i> -propanol	14	2.191	25	3.130
halothane	15	2.267	18	2.383
methyl <i>tert</i> -butyl ether	16	2.366	7	1.554
hexane	17	2.495	6	1.386
<i>tert</i> -butyl nitrite	18	2.736	9	1.750
chloroform	19	2.870	27	3.290
<i>sec</i> -butyl alcohol	20	2.962	30	3.793
isobutyl nitrite	21	2.973	12	1.853
<i>sec</i> -butyl nitrite	22	3.059	14	1.939
isobutyl alcohol	23	3.460	32	5.100
tetrahydrofuran	24	3.736	24	2.845
methyl ethyl ketone	25	3.768	26	3.271
ethyl acetate	26	3.800	23	2.785
carbon tetrachloride	27	3.842	21	2.565
1,1,1-trichloroethane	28	3.869	22	2.729
<i>n</i> -butyl nitrite	29	3.879	19	2.469
benzene	30	4.186	28	3.392
<i>n</i> -butyl alcohol	31	4.565	33	6.747
trichloroethylene	32	5.205	31	4.084
isoamyl nitrite	33	6.377	29	3.728
methoxyflurane	34	7.279	36	7.219
isoamyl alcohol	35	7.428	38	9.447
toluene	36	8.358	34	6.944
1,1,2-trichloroethane	37	8.498	39	10.138
methyl isobutyl ketone	38	9.510	37	7.964
tetrachloroethylene	39	9.681	35	7.081
chlorobenzene	40	11.810	41	11.012
ethylbenzene	41	12.279	40	10.704
<i>p</i> -xylene	42	12.726	42	11.038
<i>m</i> -xylene	43	12.727	43	11.046
<i>o</i> -xylene	44	13.733	44	12.280
tetrachloroethane	45	14.106	50	16.968
isopropylbenzene	46	14.845	46	12.962
<i>n</i> -propylbenzene	47	15.966	47	14.124
1,3,5-trimethylbenzene	48	16.565	48	14.711
decane	49	17.166	45	12.369
1,2,4-trimethylbenzene	50	17.586	49	15.904
butylbenzene	51	19.739	51	17.732
tetradecane	52	29.806	52	24.950

30m, 0.53mm ID, 3.0µm Rtx®-BAC1 (cat.# 18001) and
 30m, 0.53mm ID, 2.0µm Rtx®-BAC2 (cat.# 18000).
 1.0mL headspace sample
 Oven temp.: 40°C (hold 5 min.) to 240°C @ 5°C/min.
 Inj. & det. temp.: 240°C
 Carrier gas: He
 Linear velocity: 65cm/sec.

Figure 2 Rapid analysis for volatile anesthetics using Rtx®-BAC1 and Rtx®-BAC2 columns.

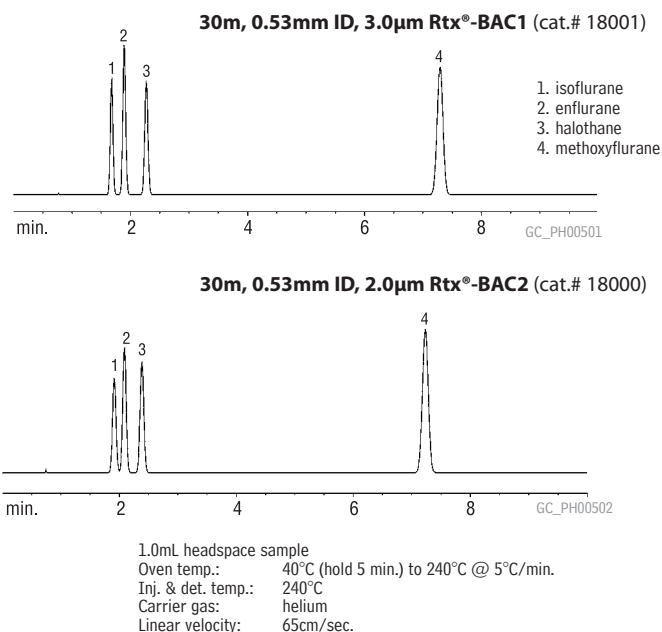


Figure 3 Screen for and confirm alkyl nitrites and metabolites on Rtx®-BAC1 and Rtx®-BAC2 columns.

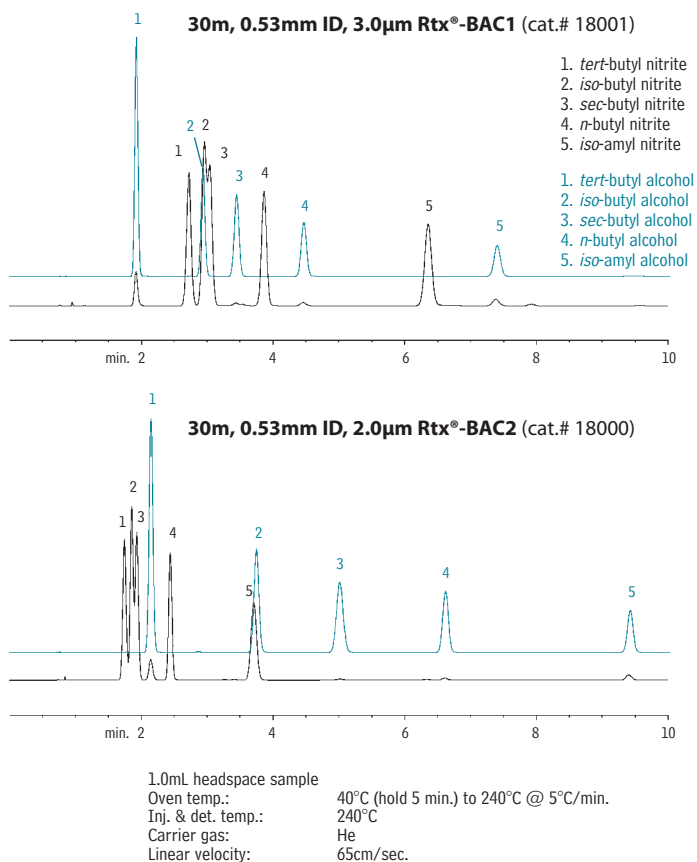
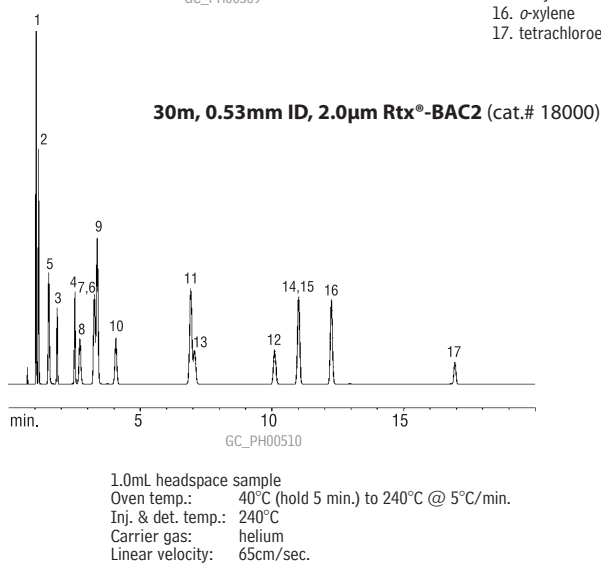
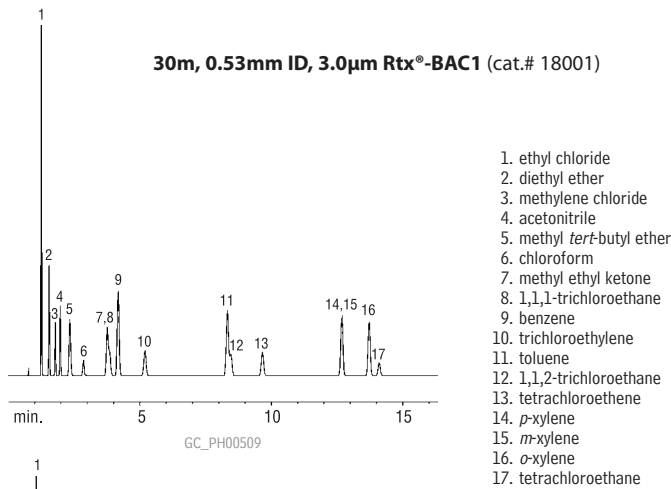


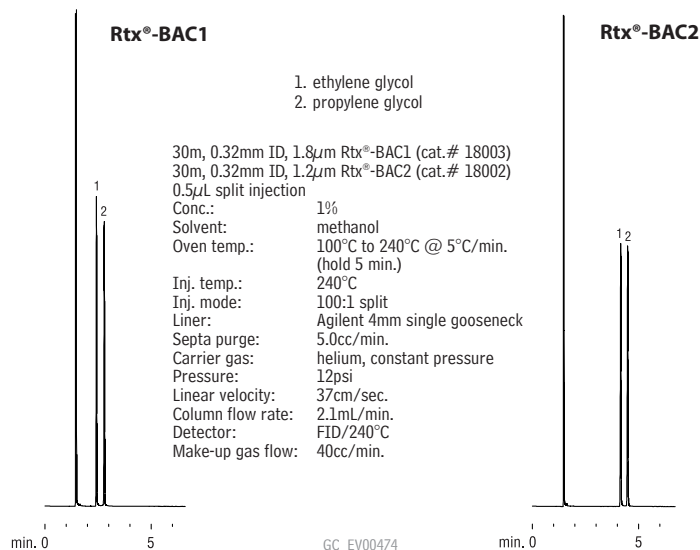
Figure 4 Elution order changes for common industrial solvents on Rtx®-BAC1 and Rtx®-BAC2 columns.



Blood Alcohol Standards

Compound	qty.	cat.#
0.015g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36232
1mL/ampul	10-pk.	36332
5mL/ampul	ea.	36240
20mL/ampul	ea.	36248
0.02g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36233
1mL/ampul	10-pk.	36333
5mL/ampul	ea.	36241
20mL/ampul	ea.	36249
0.025g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36234
1mL/ampul	10-pk.	36334
5mL/ampul	ea.	36242
20mL/ampul	ea.	36250
0.04g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36235
1mL/ampul	10-pk.	36335
5mL/ampul	ea.	36243
20mL/ampul	ea.	36251
0.05g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36257
1mL/ampul	10-pk.	36259
5mL/ampul	ea.	36258
20mL/ampul	ea.	36260
0.08g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36262
1mL/ampul	10-pk.	36264
5mL/ampul	ea.	36263
20mL/ampul	ea.	36265
0.1g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36236
1mL/ampul	10-pk.	36336
5mL/ampul	ea.	36244
20mL/ampul	ea.	36252
0.15g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36237
1mL/ampul	10-pk.	36337
5mL/ampul	ea.	36245
20mL/ampul	ea.	36253
0.2g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36238
1mL/ampul	10-pk.	36338
5mL/ampul	ea.	36246
20mL/ampul	ea.	36254
0.3g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36239
1mL/ampul	10-pk.	36339
5mL/ampul	ea.	36247
20mL/ampul	ea.	36255
0.4g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36266
1mL/ampul	10-pk.	36268
5mL/ampul	ea.	36267
20mL/ampul	ea.	36269

Figure 5 Glycols of forensic interest on Rtx®-BAC1 and Rtx®-BAC2 Columns.



Blood Alcohol Mix Resolution

Control Standard (8 components)

acetaldehyde	ethyl acetate
acetone	isopropanol
acetonitrile	methanol
ethanol (NIST certified value)	methyl ethyl ketone

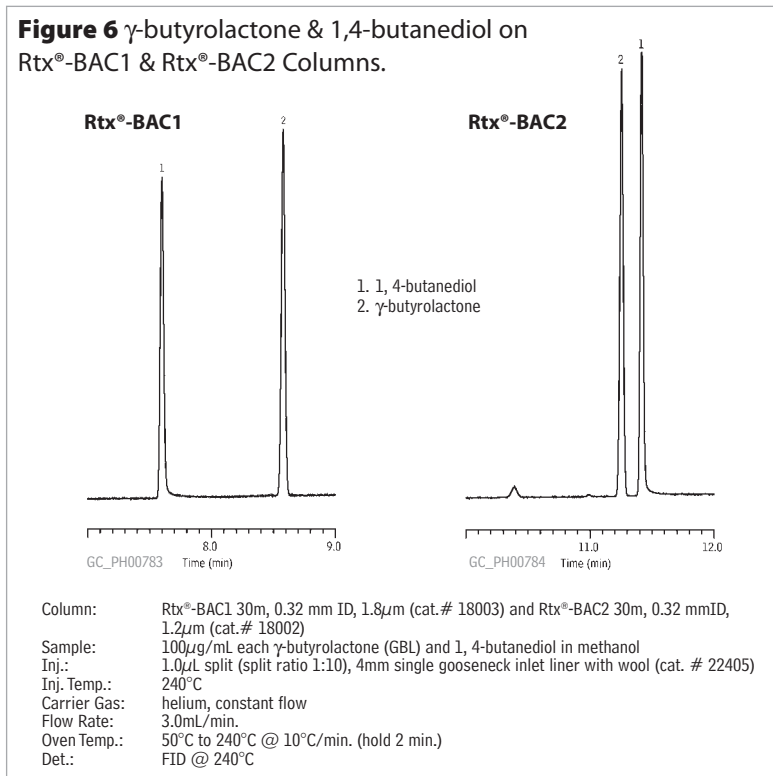
0.100g/dL each in water, 1mL/ampul
 cat. # 36256 (ea.)

We designed Rtx[®]-BAC1 and Rtx[®]-BAC2 columns for blood alcohol analysis by headspace GC/FID (Figure 1), but many other materials of forensic interest also can be analyzed and confirmed using this column pair in a headspace GC/FID system, including inhalant anesthetics, alkyl nitrites, glycols, industrial solvents, and petroleum hydrocarbons. The substances in these target groups are resolved to baseline on one column or the other. Inhalants (Figure 2) or alkyl nitrites and their alcohol metabolites (Figure 3), for example, show excellent resolution and responses, and symmetrical peak shapes, in short analysis times. Similarly, performance is excellent for common industrial solvents (Figure 4). Retention times for many compounds of interest are presented in Table 1.1

For the analysis and confirmation of blood alcohols or other materials on Rtx[®]-BAC1 and Rtx[®]-BAC2 columns, we use a GC/FID equipped with a headspace autosampler that simultaneously introduces sample onto the two analytical columns. This dual column technique increases throughput by providing screening and confirmation data from a single injection. By using 0.32mm ID columns and a high carrier gas flow rate, we achieve baseline resolution of blood alcohol compounds in less than 3 minutes (Figure 1).

Other abused substances of interest, such as gamma-hydroxybutyrate (GHB, the “date rape drug”), and poisons, such as ethylene glycol, typically are analyzed from liquid injections. The Rtx[®]-BAC1 / Rtx[®]-BAC2 dual column system coupled with FID assures excellent responses and peak shapes for ethylene glycol and propylene glycol (Figure 5) or for GHB (Figure 6), which usually is converted to gamma-butyrolactone (GBL) for the analysis.

By analyzing these abused substances and poisons simultaneously on Rtx[®]-BAC1 and Rtx[®]-BAC2 columns, compounds coeluting on one stationary phase are resolved on the complementary stationary phase, and analytical and confirmation data are obtained in half the time required with sequential injections. Analytes characteristically are eluted with excellent responses and peak shapes. These example applications establish dual column analysis and confirmation on Rtx[®]-BAC1 and Rtx[®]-BAC2 columns as a very useful and highly adaptable forensics technique.



Rtx[®]-BAC1 Columns (fused silica)

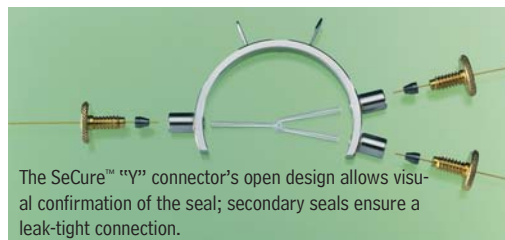
ID	df (μ m)	temp. limits	length	cat. #
0.32mm	1.80	-20 to 240/260°C	30-Meter	18003
0.53mm	3.00	-20 to 240/260°C	30-Meter	18001

Rtx[®]-BAC2 Columns (fused silica)

ID	df (μ m)	temp. limits	length	cat. #
0.32mm	1.20	-20 to 240/260°C	30-Meter	18002
0.53mm	2.00	-20 to 240/260°C	30-Meter	18000

SeCure™ “Y” Connector Kit

SeCure™ “Y” connector body, 3 knurled nuts, “Y” Universal Press-Tight® union, 3 ferrules.



Description	Ferrules Fit Column ID	qty.	cat.#
Connector Kit	0.28/0.32mm	kit	20277

Intermediate-Polarity Deactivated Guard Columns/Transfer Lines

Nominal ID	Nominal OD	5-Meter
0.32mm	0.45 ± 0.04mm	10044
0.53mm	0.69 ± 0.05mm	10045

¹For more information about analyses of anesthetics, or for analyses of petroleum hydrocarbons, please request Application Note 59548 or 59574, respectively.

additional reading

Clarke’s Analysis of Drugs and Poisons, Third Edition, A.C. Moffat, M.D. Osselton and B. Widdop (editors), Pharmaceutical Press, 2004.

Drug-Facilitated Sexual Assault: A Forensic Handbook, Marc A. LeBeau and Ashraf Mozayani, Academic Press, 2001

Handbook of Forensic Drug Analysis, Frederick P. Smith and Jay A. Siegel (editors), Academic Press, 2004