

THE RESTEK

ADVANTAGE

Industrial Solvent Analysis Simplified

Retention index table helps analysts choose the most appropriate capillary column for a given solvent matrix

Manufacturers generate residual solvents during the manufacturing process of their product. Today it is beneficial to accurately and quickly monitor these components. Capillary GC has become more popular for solvent analysis than traditional packed column GC since it provides better resolution, faster analysis times, and greater inertness. With the growing number of solvents in common use, it has become increasingly difficult to select the most appropriate capillary column polarity. By having a table of retention indices, column selection can be made with the confidence that all components will be separated on the stationary phase chosen. Columns of different polarity can be used for confirmational analysis to improve identification and quantitation.

We analyzed a wide range of solvents and determined their elution orders and retention indices on three different polarity capillary columns. The stationary phases evaluated were a non-polar, 100% dimethyl polysiloxane (Rt_x-1), an intermediate polarity, methyl trifluoropropyl polysiloxane (Rt_x-200), and a polar, polyethylene glycol (Stabilwax). The solvents analyzed also spanned a wide range of polarity. Various alcohols, ketones, aromatic hydrocarbons, and halogenated compounds were included to cover a range of typical industrial solvents. Seven saturated hydrocarbons ranging from pentane to

Figure 1 - Equation for calculating retention indices under temperature programmed conditions

$$I_a = 100N + 100n(t_{Ra} - t_{RN}) / (t_{R(N+n)} - t_{RN})$$

where: N = the carbon number of the lower hydrocarbon

n = the difference in carbon numbers that bracket the component of interest

t_{Ra} = the retention times of the component of interest

t_{RN} = the retention time of the saturated hydrocarbon eluting before the component of interest

t_{R(N+n)} = the retention time of the saturated hydrocarbons eluting after the component of interest

As an example, N-methyl-2-pyrrolidone has a retention index of 1004 on an Rt_x-1 column, meaning that it elutes approximately halfway between nonane (nC9) and undecane (nC11).

nonadecane were added as retention index reference standards.

Pages 2 and 3 list the absolute retention times and retention indices of 120 common industrial solvents. The absolute retention times show the elution order of the solvents under the run conditions used in this study. Also included are retention indices for each solvent, on all three columns, in the event other run conditions are chosen.

The retention index system, as defined by Kováts (1), is a measure of relative retention times referenced to a series of saturated hydrocarbons. Normally, retention indices are determined under isothermal conditions. However, since the vast majority of analyses are performed under temperature programmed conditions, a modified retention index equation as defined by Van den Dool and Kratz (2) was chosen for this study.

Figure 1 shows this equation and an example of how the equation was applied to this study.

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Solvent	BP	MW	Rt _x -1		Rt _x -200		Stabilwax	
			Retention Index	Retention Time	Retention Index	Retention Time	Retention Index	Retention Time
1,1,2-trichloroethane	115	132	747	15.331	867	14.632	1287	20.908
1,2,3,4-tetrahydronaphthalene	207	132	1168	32.49	1274	28.803	1576	29.571
1,2,4-trimethylbenzene	168	120	993	26.033	1083	22.723	1312	21.745
1,2-dichlorobenzene	175	146	1031	27.499	1174	25.667	1482	26.934
1,2-dichloropropane	96	112	675	12.164	802	12.053	1059	13.031
1,2-xylene	145	106	886	21.728	991	19.296	1212	18.335
1,3-xylene	139	106	863	20.685	954	17.943	1166	16.72
1,4-dichlorobutane	163	126	881	21.52	1074	22.389	1366	23.404
1,4-xylene	137	106	865	20.775	952	17.86	1160	16.501
1-heptanol	176	116	954	24.481	1078	22.509	1472	26.642
1-nonanol	211.5	144	1156	32.096	1288	29.254	1677	32.314
2,2,2-trifluoroethanol	74	100	----	4.028	534	4.302	1199	17.862
2,2-dimethyl-1-pentanol	121	116	869	20.971	986	19.136	1341	22.633
2,4-dimethyl-2-pentanol	133.1	116	780	16.871	895	15.74	1146	16.03
2,4-dimethyl-3-pentanone	124.5	114	782	16.928	969	18.505	1005	11.18
2,6-dimethyl-4-heptanone	169	142	961	24.759	1163	25.317	1194	17.686
2-bromopentane	117	150	551	7.142	879	15.105	980	10.32
2-butanol	98	74	571	7.942	691	7.81	1028	11.976
2-buten-1-ol	121.2	72	641	10.8	765	10.573	1235	19.107
2-chloropropane	36	79	----	4.925	557	4.818	695	3.589
2-chloro-2-methyl-butane	84	106	649	11.091	754	10.138	794	5.472
2-decanol	211	158	1189	33.2	1318	30.094	1631	31.069
2-decanone	210	156	1177	32.783	1429	33.132	1518	27.966
2-dodecanone	246.5	184	1382	39.339	1642	38.557	1730	33.686
2-ethyl-1-butanol	146	102	827	19.02	941	17.465	1323	22.068
2-ethyl-1-hexanol	184.5	130	1016	26.926	1136	24.467	1502	27.531
2-heptanol	161	116	884	21.643	1008	19.923	1329	22.269
2-heptanone	150	114	870	21.005	1101	23.358	1205	18.066
2-methoxyethanol (methyl Cellosolve™)	124	76	600	9.133	768	10.687	1197	17.788
2-methyl-1-pentanol	148	102	820	18.679	934	17.206	1316	21.861
2-methyl-3-buten-2-ol	97.5	86	582	8.407	704	8.177	1046	12.568
2-methyl-3-pentanol	127	102	759	15.885	864	14.505	1171	16.889
2-nonanol	193.5	144	1086	29.643	1214	26.911	1529	28.278
2-nonanone	195.3	142	1073	29.162	1319	30.122	1415	24.901
2-octanol	177.5	130	988	25.812	1108	23.602	1432	25.4
2-octanone	173	128	974	25.278	1213	26.899	1308	21.626
2-phenoxyethanol	245	138	1199	33.53	1447	33.606	----	44.134
2-propen-1-ol/allyl alcohol	97	58	521	5.941	608	5.955	1126	15.347
2-(2-butoxyethoxy)ethanol	231	162	1167	32.438	1373	31.596	1821	35.907
3-buten-1-ol	113	72	606	9.36	735	9.385	1186	17.411
3-heptanone	150	114	867	20.869	1077	22.481	1175	17.023
3-hexanol	135	102	783	16.976	891	15.571	1212	18.33
3-methyl-1-butanol/isoamyl alcohol	130	88	719	14.026	833	13.274	1227	18.826
3-methyl-2-buten-1-ol	140	86	758	15.86	889	15.514	1337	22.492
3-methyl-3-pentanol	122.4	102	740	15.006	852	14.04	1125	15.305
3-pentanone	102	86	670	11.975	875	14.963	984	10.455
adiponitrile	295	108	1099	30.16	1837	42.933	----	47.357
allyl acetate	103.5	100	676	12.205	846	13.814	1041	12.418
α-methyl styrene	169	118	977	25.382	1075	22.419	1362	23.271
α-methylbenzyl alcohol	204	122	1043	27.964	1219	27.061	1848	36.552
α-pinene	155.5	136	944	24.093	965	18.364	1026	11.911
benzene	80	78	642	10.843	733	9.308	949	9.246
benzonitrile	188	103	958	24.654	1274	28.8	1655	31.716
benzyl acetate	206	150	1141	31.573	1388	32.024	1769	34.624
benzyl alcohol	205	108	1013	26.811	1135	24.452	----	38.202
butyl ether	142.5	130	877	21.311	912	16.378	966	9.825
chloroacetonitrile	126.5	75	622	10.011	865	14.545	1435	25.489
chlorobenzene	132	112	838	19.524	947	17.665	1242	19.372
chloroform	61.5	118	586	8.567	650	6.89	1032	12.091
cumene/isopropylbenzene	154	120	920	23.155	1004	19.787	1202	17.973

Solvent	BP	MW	Rt _x -1		Rt _x -200		Stabilwax	
			Retention Index	Retention Time	Retention Index	Retention Time	Retention Index	Retention Time
cyclohexane	81	84	654	11.32	661	7.143	716	3.922
cyclopentane	50	70	550	7.093	547	4.592	602	3.223
cyclopentanol	139.5	86	767	16.264	885	15.355	1323	22.077
cyclopentanone	131	84	763	16.07	1030	20.759	1214	18.405
decahydronaphthalene	191	138	1118	30.81	1097	23.242	1180	17.219
dibromomethane	98	174	674	12.115	756	10.244	1192	17.624
dicyclohexylamine	256	181	1440	41.026	1510	35.307	1691	32.702
diethyl phthalate	298	222	1570	44.651	----	46.167	----	48.912
diethylbenzene	182	134	1048	28.16	1123	24.049	1333	22.389
dimethyl phthalate	283.7	194	1428	40.685	1853	43.258	----	47.498
dimethyl sulfoxide	189	78	785	17.088	1248	27.973	1606	30.379
dimethylacetamide	164	87	833	19.318	1223	27.19	1438	25.595
di-iso-propylbenzene	203	162	1152	31.944	1217	27.019	1385	23.967
ethyl acetate	77	88	583	8.46	757	10.267	893	7.418
ethyl decanoate/ethyl caprate	245	200	1378	39.209	1555	36.416	1454	26.084
ethyl propionate	99	102	692	12.867	845	13.773	963	9.746
ethyl trichloroacetate	168	190	970	25.124	1141	24.626	1384	23.946
ethylbenzene	136	106	856	20.348	939	17.396	1150	16.162
furfuryl alcohol/2-furanmethanol	170	98	831	19.186	1001	19.668	1685	32.54
heptadecane	302	240	1700	48.082	1700	39.989	1700	32.956
heptane	98	100	700	13.172	700	8.008	700	3.61
hexanol	156	102	852	20.184	974	18.673	1367	23.419
hexyl acetate	169	144	996	26.138	1175	25.705	1290	21.03
hexylene glycol/2-methyl-2,4-pentanediol	197	118	888	21.83	1136	24.459	1643	31.403
isopropyl acetate	85	102	638	10.67	806	12.2	907	7.804
iso-amyl alcohol	146	130	719	14.026	1038	21.052	1227	18.826
methoxyethoxyethanol	194	120	906	22.626	1128	24.23	1613	30.576
methyl hexanoate	151	130	905	22.584	1069	22.205	1209	18.21
methyl isoamyl ketone/5-methyl-2-hexanone	144	114	840	19.604	1077	22.472	1160	16.525
methylcyclohexane	101	98	725	14.329	735	9.416	746	4.524
methyldecanoate	224	186	1306	37.08	1482	34.582	1617	30.682
m-chlorotoluene	162	126	949	24.302	1071	22.279	1348	22.85
m-cresol	203	108	1048	28.169	1209	26.766	----	42.798
nitrobenzene	210.5	123	1067	28.935	1400	32.329	1799	35.357
nonadecane	330	268	1900	52.766	1900	44.272	1900	37.816
nonanal	198	142	1087	29.69	1310	29.884	1422	25.108
nonane	151	128	900	22.388	900	15.945	900	7.566
N-methyl-2-pyrrolidone	202	99	1004	26.473	1455	33.828	1720	33.43
octane	127	114	803	17.93	799	11.935	764	4.88
o-chlorotoluene	159	126	948	24.272	1057	21.758	1339	22.554
o-cresol	191.5	108	1030	27.49	1180	25.844	----	40.877
pentadecane	270	212	1500	42.798	1500	35.059	1500	27.484
pentane	36	72	500	5.083	500	3.55	500	2.823
propyl acetate	102	102	696	12.991	864	14.503	979	10.288
pyridine	115.5	79	721	14.143	897	15.814	1205	18.081
p-cresol	202	108	1048	28.173	1205	26.637	----	42.592
p-cymene	178	134	1024	27.251	1092	23.034	1296	21.242
p-methoxyphenol	243	124	1184	33.023	1413	32.699	----	49.278
sec-butanol	98	74	571	7.942	691	7.81	1028	11.976
s-tetrachloroethane	147	166	885	21.704	1012	20.078	1535	28.441
tetrachloroethylene	121	164	808	18.146	854	14.104	1032	12.117
toluene	111	92	760	15.917	845	13.775	1056	12.92
trans-1,2-dichloroethylene	48	96	540	6.711	583	5.406	849	6.56
tributyl phosphate	305	266	1617	45.895	----	47.04	----	43.282
trichloroethylene	87	130	685	12.575	747	9.89	1003	11.093
trichlorotrifluoroethane/freon113	47.6	186	519	5.859	550	4.668	585	3.158
tridecane	234	184	1300	36.912	1300	29.617	1300	21.375
triethylbenzene	215	162	1219	34.194	1276	28.868	1488	27.103
undecanal	235	170	1292	36.657	1529	35.782	1636	31.215
undecane	196	156	1100	30.208	1100	23.34	1100	14.436

Solvent Analysis (cont. from page 1)

Figure 2 shows chromatograms from each of the three columns evaluated in this study. The solvents are numbered in their elution order on the R_{t_x} -1 column. By comparing the numbering sequence of the peaks on the R_{t_x} -200 and Stabilwax columns, the effects of stationary phase polarity on the elution order of these solvents is very evident. Chromatogram A shows that allyl alcohol, peak 2, elutes very quickly on a non-polar R_{t_x} -1 column and is not completely separated from the tail of the methylene chloride peak. Chromatogram B shows that on a more polar R_{t_x} -200 column, the allyl alcohol is

completely separated from the methylene chloride. Chromatogram C shows that the allyl alcohol elutes more than 10 minutes later and is easily separated from the methylene chloride peak.

With the growing number of industrial solvents in common use, a reliable analytical approach for choosing the correct polarity capillary column is essential. The data presented is the first part of an extensive study to determine the retention indices of a wide range of solvents on a broad range of capillary column polarities. The retention indices for over 150 additional solvents will be determined on these same three polarity

columns and retention indices for all 270 solvents are being determined on several other polarity columns. Our ultimate goal is to provide the most comprehensive database available on solvent retention indices.

References

(1) Kováts, E., Giddings, J.C. and Keller, R.A., *Advances in Chromatography*, Volume 1, Chapter 7. New York: Marcel Dekker (1965).

(2) Van Den Dool, H. and Kratz, P.D. *Journal of Chromatography*, Volume 11, pp. 463-471, (1963).

Figure 2 - By changing polarity, difficult solvents can be resolved.

Peak Identification

- | | |
|----------------------------------|----------------------------|
| 1. Pentane | 11. 1,2,4-trimethylbenzene |
| 2. 2-propen-1-ol (allyl alcohol) | 12. 2-ethyl-1-hexanol |
| 3. Isopropyl acetate | 13. Undecane |
| 4. Benzene | 14. Decahydronaphthalene |
| 5. Heptane | 15. 2-decanol |
| 6. Cyclopentanone | 16. 2-phenoxyethanol |
| 7. 2-ethyl-1-butanol | 17. Tridecane |
| 8. 1,4-dichlorobutane | 18. Pentadecane |
| 9. Nonane | 19. Heptadecane |
| 10. Methyl hexanoate | |

Run Parameters

Instrumentation: HP 5890 GC with HP 5971A Mass Selective Detector and FID with HP 7673 Autosampler
effluent splitter: FID/MSD

Columns: 60m, 0.53mm ID, 3.0 μ m R_{t_x} -1 (cat.# 10188)
60m, 0.53mm ID, 3.0 μ m R_{t_x} -200 (cat.# 15088)
60m, 0.53mm ID, 1.0 μ m Stabilwax (cat.# 10658)

Conditions: 1.0 μ l split injection
using a Restek Cyclosplitter® Sleeve (cat.# 20706)

Split ratio: 50:1

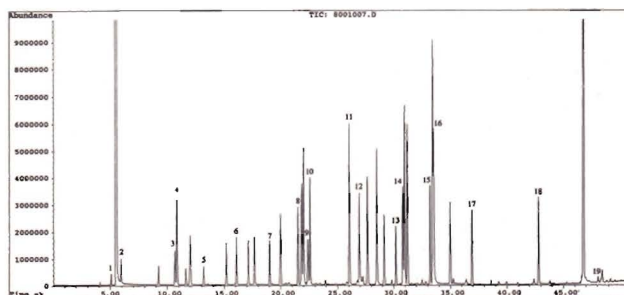
Inj. Temp.: 275°C

Det. Temp.: 285°C

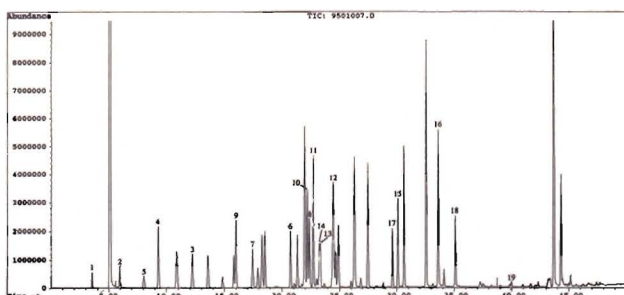
Carrier gas: Helium at approximately
40cm/sec.

Temp. program: 40°C (hold 5 min)
to 285°C (250°C for
Stabilwax) @ 5°C/min.

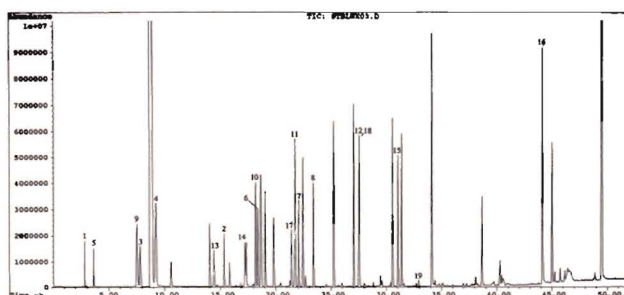
A (R_{t_x} -1)

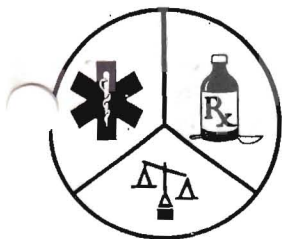


B (R_{t_x} -200)



C (Stabilwax)





Clinical Corner

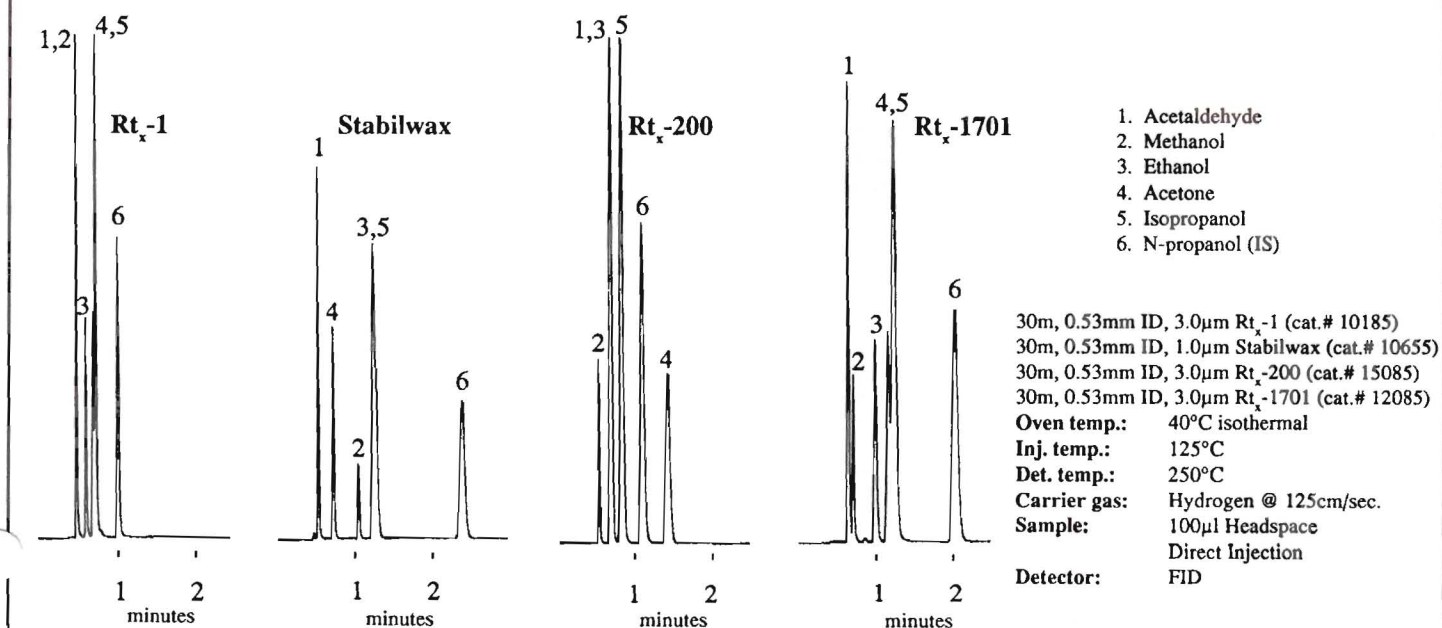
Dual Column Analysis for Blood Alcohols

Testing for the presence of ethanol in biological specimens is the highest volume test run in forensic laboratories today. Because of the potential for intoxication by ingestion of other volatile compounds, blood alcohol analysis usually includes screening and quantitation of ethanol plus methanol and isopropanol and two metabolites of these alcohols, acetone and acetaldehyde. Requirements for this analysis include positive or unique identification of all analytes with a short turnaround time. Routine testing for ethanol has been performed for more than 50 years and can be accomplished by a variety of techniques. However, each technique has its own drawbacks. Chemical tests such as oxidation by dichromate or permanganate solutions with subsequent detection by UV spectroscopy are not specific for ethanol and are rarely used any longer. Enzyme assays for ethanol can be highly

specific, have sufficient sensitivity and have short turnaround times. However, they do not yield any additional information about the presence or absence of other potential intoxicants. Today, the most widely used technique for blood alcohol analysis is gas chromatography. Gas chromatographic methods have the ability to give excellent resolution for all of the alcohols and their metabolites with short turnaround times. Mass specific detectors allows positive identification of the volatile components present in biological specimens. Unfortunately, most laboratories cannot afford to dedicate a GC/MS system to a high volume test like blood alcohol analysis and must use a non-specific detector like an FID. Confirmation of the identity of these compounds can still be achieved on an FID by using a dual column system that changes the elution order and retention time of the analytes.

The first chromatographic methods for ethanol testing were established using packed columns made with primarily two types of liquid phases, a non-polar methyl silicone phase and a polar polyethylene glycol phase. Choices for capillary columns for blood alcohol analysis have followed the same trends and focus on the same types of liquid phases. Below, we show that a combination of intermediate polarity columns can effectively be used as screening and confirmational columns for ethanol and some closely related compounds. Four different stationary phases were evaluated for use in blood alcohol testing. The most commonly used stationary phases, methyl silicone and polyethylene glycol, and two intermediate polarity stationary phases, trifluoropropylmethyl silicone and cyanopropylphenylmethyl silicone, were run under identical analytical parameters (Figure 1). Thicker film columns were

Figure 1 - The Rt_x-1701 proved to be the best screening column while the Rt_x-200 is the best confirmational column for blood alcohol analysis.



used to increase each column's resolving power and sample handling capacity. To minimize analysis time, high carrier gas flow rates using hydrogen as the carrier gas were employed. Hydrogen was chosen as the carrier gas to minimize the loss in column efficiency when run with high flow rates.* Results showed that all four columns have partial or complete co-elutions of some compounds. There are two co-elutions on the Rt_x-1 column (methyl silicone). Methanol and acetaldehyde are completely unresolved and acetone and isopropanol are only 40% resolved. The remaining compounds elute very close to one another because of the low polarity of the methyl silicone stationary phase. The Stabilwax column (polyethylene glycol) shows better selectivity than the Rt_x-1 column because of the increased stationary phase polarity, but ethanol and isopropanol co-elute with one another. The Rt_x-200 column (trifluoropropylmethyl silicone) had excellent resolution for all of the alcohols due to its selectivity for compounds with lone pair electrons. The only co-elution was with ethanol and acetaldehyde. When compared to the other phases, the Rt_x-1701 column (cyanopropylphenyl methyl silicone) had the best overall resolution. There were no complete co-elutions, with acetone and isopropanol being approximately 40% resolved.

In choosing two columns for use in a dual column system for blood alcohol analysis, overall resolution and co-elutions with ethanol are the primary considerations. Although ethanol is completely resolved on the Rt_x-1 column, it was excluded because of the two co-eluting pairs and low resolving power. The Stabilwax column was dropped from consideration because ethanol co-eluted with

isopropanol, a potential contaminant from skin preparation prior to collecting blood samples. Based upon the chromatography for the two remaining phases, the Rt_x-1701 column would be the choice for the screening column. It provides the best resolution of all compounds. The Rt_x-200 would then be the choice for the

packed column injection port and two FIDs. Installation of both columns into the injection port was achieved by using a "Y" Press-Tight® connector and a 10cm length of 0.53mm ID deactivated guard column tubing. A 1mm ID Uniliner® was used to adapt the packed column port for use with wide bore capillary columns.

"... the Rt_x-1701 column would be the choice for the screening column. ... The Rt_x-200 would then be the choice for the confirmational column."

confirmational column. Because of the unique selectivity of the trifluoropropyl phase, the elution order for acetone and acetaldehyde relative to the other alcohols is dramatically altered. This shift in retention time and elution order provides the mechanism for confirmation of identity of any volatile intoxicants present. Acetone and isopropanol, which had been unresolved on the Rt_x-1701, are now completely resolved from one another. The co-elution of ethanol and acetaldehyde on the Rt_x-200 is a minor problem since clinically significant concentrations of acetaldehyde are rarely encountered.

The Rt_x-1701 and the Rt_x-200 were then installed into an instrument containing a

Because both columns were of the same length and internal diameter, carrier gas flow and samples to be analyzed were split evenly between the two columns. The detector ends of each column were then installed in separate FIDs. All other analytical parameters were the same. The chromatography achieved with this dual column system was identical to that obtained when using a single column.

Rt_x-1701 and Rt_x-200 columns can be used in a dual column configuration that provides rapid detection and quantitation for ethanol and associated volatile compounds in biological samples.

Product Listing

Phase & Composition	length	ID (mm)	df	cat.#	price
Rt _x -1701	30m	0.53	3.0	12085	\$445
Rt _x -200	30m	0.53	3.0	15085	\$475
Guard Column	5m	0.53	---	10045	\$60
6-pack	5m	0.53	---	10045-600	\$300
"Y" Press-Tight connector	---	---	---	20405	\$55
3-pack	---	---	---	20406	\$145

* longer analysis times and some loss of resolution may occur with helium as the carrier gas

Correction



In the last issue of The Restek Advantage (November 1991, Vol. 2 No. 5), there were some peak misidentifications in Figure 5 on page 4. Corrections are shown below:

As shown in newsletter	Correction
Peak# Name	
3 Carbon tetrachloride	Tetrachloroethylene
6 n-Propyl nitrate	Bromoform
7 Methylene bromide	n-Propyl nitrate

Standards Spotlight



New Chemical Standards for EPA 505 Series Methods

- only source of complete method 505 analytes
- available with complete data pack documentation
- in stock for immediate delivery

Restek continues to expand its line of environmental chemical standards. In an effort to provide analytical laboratories with calibration standards to meet our clients requirements, we are pleased to announce the availability of this new product.

Restek is the only source of a calibration mixture for EPA Method 505 which contains every analyte specified in the protocol. We have had several rare

compounds synthesized to be able to provide this calibration standard.

As with all Restek environmental standards, a complete data pack is available to comply with EPA regulations. Restek data packs have been accepted by EPA auditors across the USA. Why take a chance? Order your "audit survival pack" when you purchase our standards.

Method 505 - Organohalide Pesticide Mix

Hexachlorocyclopentadiene	Hexachlorobenzene
Simazine	Atrazine
γ-BHC (Lindane)	Alachlor
Heptachlor	Heptachlor epoxide
Aldrin	Dieldrin
Endrin	Methoxychlor
α-chlordane	γ-chlordane
cis-Nonachlor	trans-Nonachlor
200µg/ml each in 1ml methanol	
Cat.# 32024	\$30ea.
32024-500	\$60 ea. w/data pack
32124	\$270 10pk. w/data pack

Recommended columns for this method:

30m, 0.32mm ID, 1.0µm Rt_x-1 (cat.# 10154)
 30m, 0.32mm ID, 1.0µm Rt_x-50 (cat.# 10554)

Product Modification - DMSO to be Used for USP 467 Standard

The solvent used for USP 467 Calibration Mixture (part no. 36000, 36100) advertised in *The Restek Advantage*, September 1991, has been changed from water to Dimethyl sulfoxide (DMSO). Several articles in recent issues of the *Pharmacopeial Forum* (1,2,3) have addressed the difficulty in obtaining consistent results with this method and the difficulty in preparing accurate calibration standards. Dimethyl sulfoxide was chosen as the replacement solvent for two reasons. First, all of the compounds being analyzed in USP 467 are soluble in DMSO. Second, DMSO is miscible with water and will act as a carrier for non-polar analytes that have poor solubility in water. This change should pose no difficulties to analysts using USP Method 467. This mixture can be easily diluted with water or other solvents specified in the USP monograph.

USP 467 Calibration Mix

Ethylene oxide	10mg/ml
Benzene	100
Chloroform	50
1,4-dioxane	100
Methylene chloride	100
Trichloroethylene	100
At concentrations listed in 1ml dimethyl sulfoxide.	
Packaged 1ml per ampul.	
Cat.# 36000	\$20ea.
36100	\$180 10pk.

References

- (1) Chen, T.K., Moekel, W., Surprenant, H.L., Proposed Changes to Method I for Organic Volatile Impurities <467>. *Pharmacopeial Forum* 1991; 17(1): 1475-479.
- (2) Bergren, M.S., D.W., Comments on USP General Chapter Organic Volatile Impurities <467>, and Associated Monograph Proposals. *Pharmacopeial Forum* 1991; 17(3): 1963-1968.
- (3) Krasowski, J.A., Dinh, H., O'Hanlon, T.J., Lindauer, R.F., Comments on Organic Volatile Impurities, Method I, <467>. *Pharmacopeial Forum* 1991; 17(3): 1969-1972.

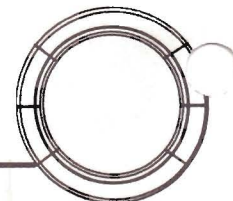
Recommended columns for this method:

Phase & Composition	length	ID (mm)	df	cat.#	price
Rtx-5	30m	0.53	5.0µm	10279	\$455
Rtx-5	30m	0.53	3.0µm	10285	\$445
Rtx-502.2	30m	0.53	3.0µm	10908	\$515
Rtx-1701	30m	0.53	3.0µm	12085	\$445

Restek is committed to providing the highest quality chemical standards!

We are constantly monitoring method modifications proposed by the governmental regulating agencies. Should additional method improvements be introduced, chemical standard mixtures will be modified to meet customer requirements.

Hints for the Capillary Chromatographer

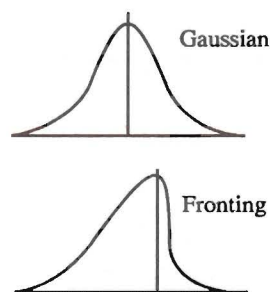


Sample Capacity and Column Overload

What is column overload?

Column overload occurs when the amount of sample injected exceeds the column's capacity for that component. Overload is normally observed as a fronting, non-gaussian peak shape (Figure 1). A column's capacity is a function of several parameters including the column's internal diameter (ID), its film thickness (df), the solubility of the compound in the column's stationary phase, and capacity factor (*k*).

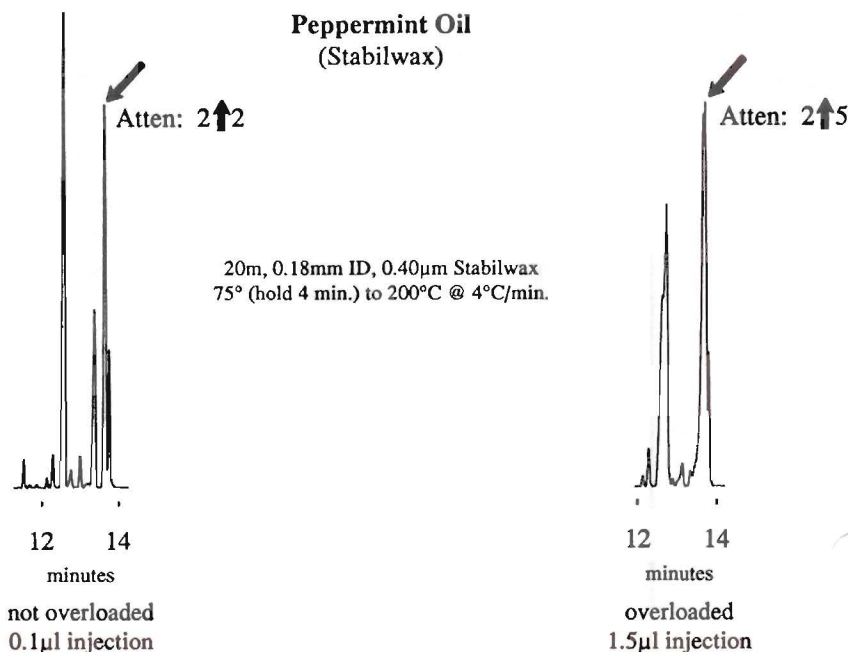
Figure 1 - Normal Gaussian vs. Overloaded Fronting Peak Shapes



Why is it important not to exceed a column's capacity?

Capillary columns have much lower sample capacities than packed columns, therefore, it is extremely important to optimize the amount of sample injected. When sample capacity is exceeded, peak symmetry is lost and resolution is affected. Because the peak shape will be much broader, resolution between two closely eluting peaks can be lost. Figure 2 shows the loss of peak symmetry and resolution in the analysis of peppermint oil on a Stabilwax column. In the first chromatogram, 0.1 µl of neat peppermint oil was injected. At these low concentrations, very good resolution between the menthyl acetate, neo-menthol,

Figure 2 - Minimize the amount of sample injected to maximize resolution.



β-caryophyllene, and terpinene-4-ol is obtained. In the second chromatogram, 1.5 µl of neat peppermint oil was injected. Because the sample concentration exceeded the column's capacity, a significant loss in resolution occurred.

How can overload be prevented?

Two choices are available to prevent overload:

- ▼ reduce the sample concentration reaching the column
- ▼ choose a column and run conditions that will allow greater sample capacity

To reduce the sample concentration reaching the column, the sample components can be diluted by increasing the split ratio, diluting with additional solvent, or by introducing a smaller amount.

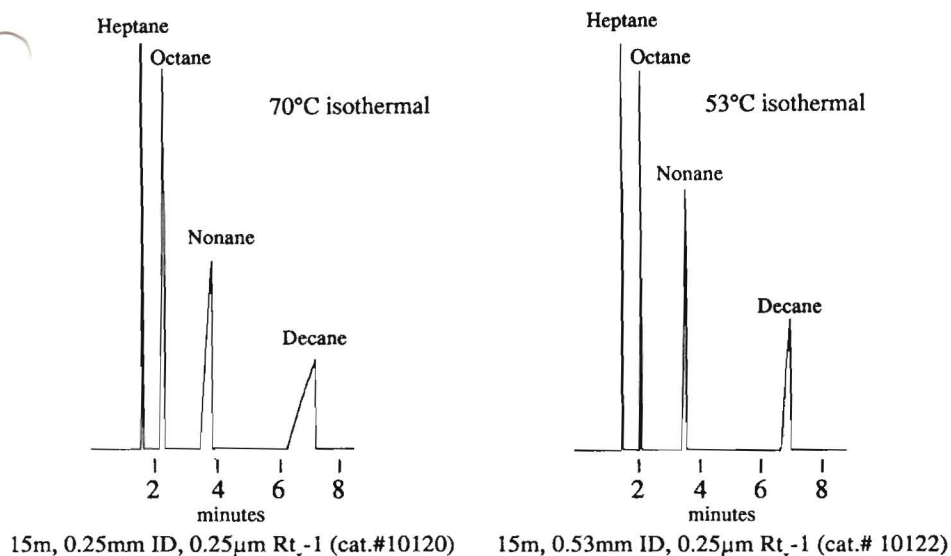
How does column ID affect sample capacity?

As the column ID increases so does sample capacity. Table 1 shows typical column capacity for several different diameter columns. Figure 3 compares sample capacity on 0.25 and 0.53mm ID columns. Four hydrocarbons (heptane, octane, nonane, and decane) were analyzed at a concentration of 1000ng on

Table 1

Column ID	0.18mm	0.25mm	0.32mm	0.53mm
Sample Capacity	<50ng	50-100ng	400-500ng	1000-2000ng

Figure 3 - Increase sample capacity by increasing column ID.



both a 0.25mm ID and 0.53mm ID column. The 0.25mm ID column exhibits overload and severe peak fronting for nonane and decane. In comparison, the 0.53mm ID column shows symmetrical peak shapes for nonane and only slight overload for decane. This illustrates the effect of increasing sample capacity by increasing column ID.

How does column film thickness affect sample capacity?

Increasing the column's stationary phase film thickness also increases sample capacity. Figure 4 shows this effect. Again, we show the same series of hydrocarbons at the 1000ng concentrations on 30 meter, 0.25mm ID, 0.25 μ m

and 1.0 μ m Rt_x-1 columns. On the 0.25 μ m column, the nonane peak shows some overload and the decane peak shows severe fronting. By increasing the film thickness to 1.0 μ m, the peak symmetry of nonane is restored and the decane peak shows only slight fronting.

How does solubility affect sample capacity?

The solubility of a sample component in the column's stationary phase also has an effect on sample capacity. The more soluble a component is in the stationary phase, the greater the column capacity for the solute. For example, a polar compound will have greater solubility in a polar stationary phase than in a non-polar

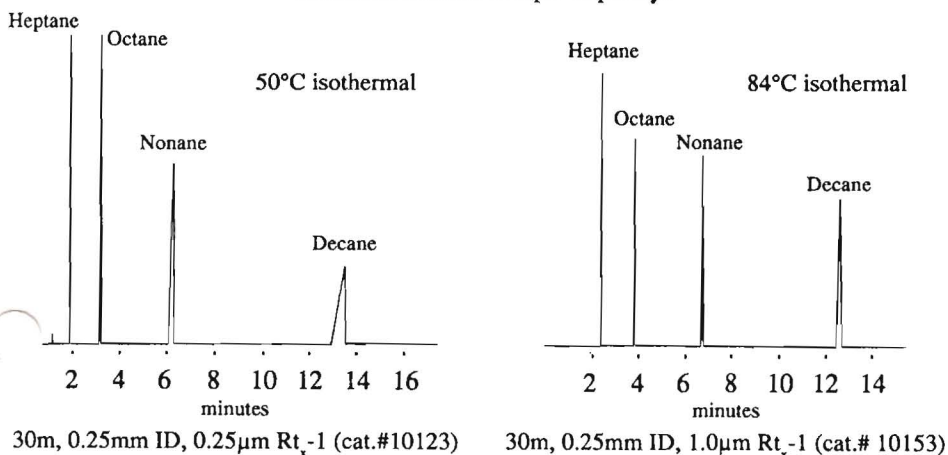
stationary phase. For environmental analysts, this phenomenon is very common when analyzing acid and base-neutral extracts on a non-polar, 5% diphenyl stationary phase. Benzoic acid, a polar compound, always exhibits very poor peak symmetry, demonstrating overload on this non-polar stationary phase. Even though it is at the same concentration, it is less soluble in this phase than the other priority pollutants and exceeds the column's capacity at a much lower concentration.

How does component retention affect sample capacity?

Sample capacity is also affected by how long the component remains in the stationary phase. The capacity factor or k gives us an indication of how long the component remains on the stationary phase. The longer a sample component remains in the stationary phase, the greater the chance for overload. The capacity for a component can be increased by selecting run conditions that will create lower k values by causing the component to elute faster from the column (faster flow rates or faster temperature programming).

When choosing a column, the analyst must keep in mind the range of component concentrations. By optimizing column ID and film thickness, and by matching the solubilities of sample components with the stationary phase, samples can be analyzed without overload. Also, by optimizing run conditions, the k value for components can be minimized resulting in better sample capacity.

Figure 4 - Increasing the stationary phase film thickness increases column sample capacity.



If there's a topic you'd like to see covered in Hints for the Capillary Chromatographer, write to:

**Hints Topics,
c/o Restek Corporation,
110 Benner Circle,
Bellefonte, PA 16823-8812.**

Peak Performers

Restek's Digital Flow Calibrator

- calibrated against NIST Standards
- large LED display for easy readout
- use with all chromatography gasses
- battery operated for portability

Restek's Digital Flow Calibrator is designed to measure and calibrate gas flows used in capillary chromatography. The flow calibrator is capable of measuring flow rates of .5-500mls/min. accurately, regardless of the gas type. It is an excellent tool for measuring the split vent flow and detector gas flows. This battery operated flow calibrator is easy to operate and is capable of displaying the split ratio.



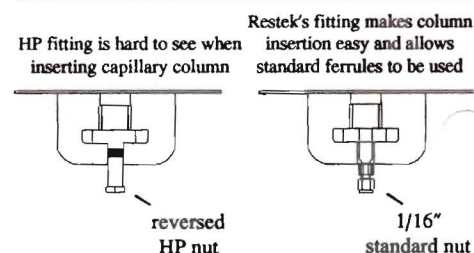
Digital Flow Calibrator:
cat.# 20123, \$495 each

HP 5890 Capillary Inlet Adaptor Fitting



The HP 5890 capillary inlet adaptor fitting has been improved to ensure a leak tight seal with the capillary column. Restek chemists have engineered

a simple HP 5890 GC capillary fitting that incorporates a standard 1/16" Swagelok® type nut and standard graphite or Vespel®/graphite ferrules.

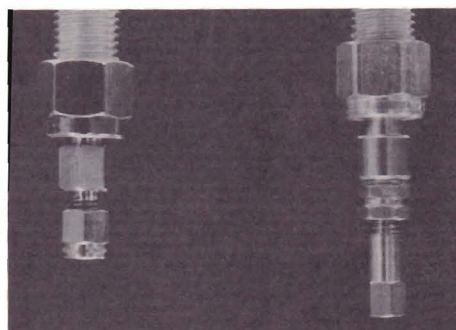


HP 5890 FID/NPD Detector Adaptor Fitting



The new HP 5890 FID/NPD Detector Adaptor fitting has been further improved to ensure a leak-tight column seal. A standard Swagelok®-type nut is incorporated

to allow use with standard graphite or Vespel®/graphite ferrules. The new adaptor replaces the awkward HP column fitting. We not only shortened the adaptor fitting to make it more compact, but we also included a wrench pad to prevent the fitting from turning when installing a column. The kit includes everything needed for installation: the adaptor fitting, 1/4" SS nut, 1/4" Vespel/graphite ferrule, 1/16" SS nut, and 0.4mm ID graphite ferrule. (Replaces HP part numbers 19244-80610 and 05921-21170.)



Restek's FID/NPD Detector Adaptor Fitting is shorter and more compact than HP's fitting, making capillary column installation easy.



We carefully machined the threads and matched the stainless steel types to eliminate seizing onto the injector body. We also paid careful attention to the fitting depth to keep the insertion distance exactly the same. The replacement inlet adaptor kit simplifies column installation due to this easy-to-use design. The kit includes everything needed for installation: the adaptor fitting, an inlet seal and washer, 1/16" SS nut, and 0.4mm ID graphite ferrule. (Replaces HP part numbers 18740-20800, 05921-21170, and 18740-20880.)



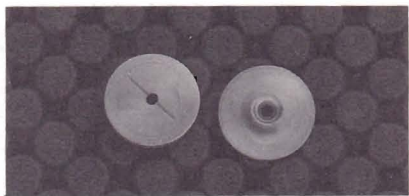
HP 5890 FID/NPD Detector Adaptor Fitting:

cat.# 20884, \$55/kit

HP 5890 Capillary Inlet Adaptor Fitting:

cat.# 20633, \$60/kit

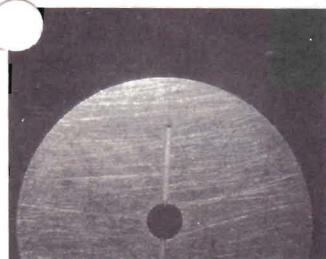
Modified Inlet Seals for HP 5890 GCs



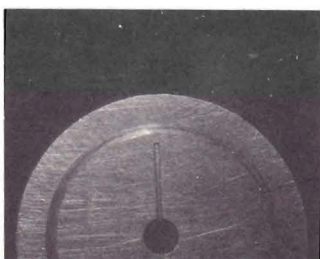
The inlet seal at the base of the HP 5890 GC injection port comes into contact with the sample and must be changed frequently to prevent adsorption of active compounds. In addition, septa fragments and sample residue accumulate on the disk surface requiring replacement.

The seal occurs by deforming the disk against the injection port base upon tightening, forming a micro ring. Originally, the disks were manufactured from 303 stainless steel which did not deform well upon tightening, resulting in a small leak. The new disk design uses 203EZ stainless steel which is softer and deforms easier, making a completely leak-tight seal.

Photomicrograph of HP inlet disks



new disk before
installation



203EZ stainless forms a deep
groove to enhance seal

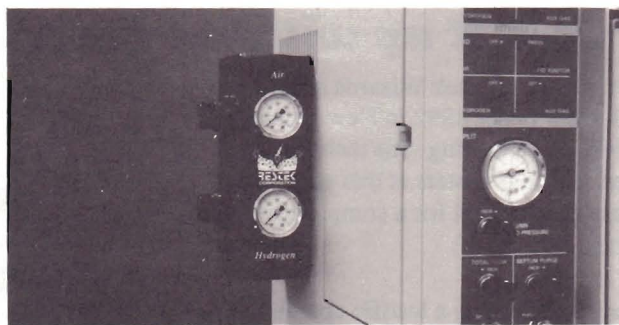
This new disk design increases column lifetime because oxygen cannot permeate into the carrier gas. Detector noise is also reduced with high sensitivity detectors such as ECDs or MSDs. (Replace HP part number 18740-20880.)

Replacement Seals for HP 5890 Split/Splitless Inlets:

cat.# 20390, \$30/2-pack

cat.# 20391, \$135/10-pack

Control HP 5890 FID Gases With a Gauge Pack



- conveniently mounts on the side of the HP 5890 GC
- standard 1/8" inlet and outlet fittings
- up to 250psi inlet pressures, 0-60psi outlet pressures
- front mounted gauges for easy visibility
- side mounted regulator knobs for easy control

HP 5890 GCs do not include the pressure regulators necessary to adjust the air and hydrogen flows to the Flame Ionization Detector (FID). Since most labs operate their gas supply lines at a higher pressure necessary for the proper operation of the FID, chromatographers must supply their own regulators to adjust flows for optimum detector performance.

Restek's new FID Gauge Pack simplifies GC installation by incorporating pressure regulators and gauges for both air and hydrogen in a single enclosure. The unit conveniently mounts on the side of the GC, and 1/8" bulkhead fittings allow easy hook-up to instrument and supply lines.

HP FID Gauge Pack:

cat.# 20129, \$175

Success Starts with Restek's 502.2 Column when Analyzing Volatile Organic Compounds!

Restek's Rt_x-502.2 capillary column separates early eluting gases without cryofocusing or subambient cooling. This column is compatible with all purge & trap and GC systems and is the recommended column in EPA Method 502.2.

Free offer:

For a limited time, receive a 502.2 Standards Kit (containing all sixty compounds and three internal standards) absolutely FREE with the purchase of a 105-meter Rt_x-502.2 column.

**105-meter Rt_x-502.2 column with FREE 502.2
Standards Kit:** cat.# 10910-250, \$1200

**Offer expires March 31, 1992. Offer not valid in conjunction with any other offer.*

News from Restek



The Wizards are Awarded a Patent

On June 9, 1992, Restek was awarded a U.S. Patent for their Cyclosplitter® sleeve

design. Issued under Patent #5,119,669, this innovative inlet sleeve provides increased vaporization surface, increased mixing, and is easy to clean. If you would like more information about Restek's deactivated inlet sleeves, please call us at 1-800-356-1688 and request a copy of our "Operating Hints for Split/Splitless Injectors".

Restek Honored

The October issue of *Inc.* Magazine contains the 1992 *Inc.* 500, which ranks this country's top 500 fastest-growing privately held companies. Restek is ranked (#246) on the eleventh annual list.

The rankings are based on the percentage increase in sales from 1987 to 1991. During this period, Restek's sales growth was 1137%.



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We Want Your Opinion

In July, Restek installed a voice mail system to help expedite phone call transfer and response. With the large volume of in-coming calls, we felt an automated system would allow us to better serve our customers.



Your opinions about our services are important to us and we would like your feedback on our automated phone system. If you have any comments or suggestions, please call Dwayne White at 1-800-356-1688, ext. 131.

Come Visit Us at EAS

November 16 through 20, the Restek wizards will be at the Eastern Analytical Symposium (EAS) in Somerset, NJ (Booth #235). In addition to our product exhibit, we will be presenting papers on "Highly Efficient and Inert Stainless Steel Capillary GC Columns: A Durable, High Temperature Alternative to Fused Silica," and "Characterization of a Novel Stationary Phase for Toxicological Analyses".

Please visit our booth and presentations. The wizards look forward to meeting you and discussing new, innovative products for chromatographic analysis.

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* U.S. Pat. No. 5,119,669.

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