

Petrochemical Applications note

cat.# 59570

Rtx[®]-1: A New Bonded Packed Column for Simulated Distillation

- Rtx[®]-1 bonded packed column requires minimal conditioning.
- Meets or exceeds all specifications of ASTM D2887 and D3710.
- Stable baseline to 350°C and repeatable RT's "right from the box".
- Deactivated Silcosteel[®] tubing and Silcoport[™] packing for high inertness.
- Column lifetime superior to existing Sim Dist columns.

Simulated Distillation (Sim Dist), according to ASTM test methods D2887 or D3710 can be performed using either packed or capillary columns. Advantages of capillary columns are the columns are preconditioned so they can be used after only minimal conditioning, and the bonded stationary phases exhibit stable baselines and retention times. There are many laboratories currently using packed columns which would like to take advantage of bonded phases but do not have GC equipment that can be easily converted for use with capillary columns.

Restek's Rtx[®]-1 Sim Dist column is the first in a new generation of bonded packed columns having superior inertness and stability compared to conventional packed columns. These improvements are obtained by preparing the columns with Silcosteel[®] tubing and bonding the Rtx[®]-1 stationary phase to a highly deactivated Silcoport[™] support. The column dimensions and packing (1/8" Silcosteel[®] with 10% Rtx[®]-1 on Silcoport[™]) are designed to exceed all requirements specified in ASTM Test Methods D2887 and D3710.

Bonded stationary phases require minimal conditioning and give stable baselines and retention times "right from the box."

Simulated distillation is a gas chromatographic procedure which differs from typical GC analysis requiring peak resolution and integration. The sample is analyzed using a linear temperature program so that the retention time of the hydrocarbons are proportional to their boiling points. The

Table 1 shows the excellent retention time repeatability obtained with the column, indicating the column is suitable for sample analysis after minimal conditioning.

Column bleed is another important consideration for selecting a Sim Dist column. The baseline must be stable and free of any artifacts during the temperature program up to 350°C. Although baseline subtraction is

Table I

Retention Time Repeatability for Calibration after only 30 minutes conditioning.

Hydrocarbon	Min Rt	Max Rt	Avg. RT	Stand. Dev.
C ₅	0.241	0.243	0.242	0.001
C ₆	0.493	0.497	0.495	0.002
C ₁₀	5.746	5.765	5.752	0.005
C ₂₀	18.482	18.491	18.486	0.004
C ₂₈	25.093	25.103	25.098	0.004
C ₄₀	32.160	32.171	32.166	0.004
C ₄₄	34.316	34.328	34.326	0.007

sample boiling range distribution is calculated by comparing the sample area and its retention time with that of an alkanes calibration standard. In order for the calibration to be valid for sample analysis, it is crucial that retention times be repeatable until the next calibration is performed. **Figure 1** is an example of the analysis of the Restek D2887 Calibration Mix (cat.# 31222) illustrating the typical pattern obtained for the alkanes under temperature programmed conditions. To demonstrate the stability of the Rtx[®]-1 column, a series of calibration standards were analyzed after only 30 minutes of conditioning at 350°C.

permitted in the method, this compensation will produce errors if the baseline is not consistent. Conventional packed columns require up to 14 hours of conditioning and frequent updating of the baseline compensation run because the stationary phase is not bonded. Rtx[®]-1 columns, however, exhibit stable

Questions?
Call Restek's technical
service staff at
800-356-1688,
ext.4

and reproducible baselines with just 30 minutes of conditioning. This results in fewer baseline blanks and less frequent calibration increasing laboratory productivity.

Rtx[®]-1 Sim Dist 2887 Packed Columns can also be used for gasoline range simulated distillation.

Simulated distillation of gasoline range hydrocarbons according to ASTM method D3710-93 can also be performed using the Rtx[®]-1 Sim Dist 2887 Packed Column. **Figure 2** shows the analysis of ASTM D3710 calibration mix with the addition of n-propane, 2-methyl propane, n-butane, n-hexadecane, and n-heptadecane. To achieve baseline separation of n-propane, 2-methyl propane, and n-butane, the GC oven was cooled to -30°C with liquid nitrogen. **Figure 3** shows the analysis of a composite gasoline sample under the same run conditions. Other volatile petroleum fractions such as kerosene and jet fuel can also be analyzed with this column.

Bonded stationary phases extend column lifetime.

The Rtx[®]-1 stationary phase is bonded to the diatomite particles resulting in an immobilized coating which is resistant to solvents and lower in bleed than conventional packing. Since the packing

is preconditioned, there is no need for extended conditioning. Extended conditioning can greatly decrease column lifetime. Since GC systems often have leaks or carrier gas that contains oxygen, it is more likely that conventional columns will be damaged during the conditioning process. **Figure 4a** shows a conventional UCW-982 column after only 170 temperature cycles, demonstrating higher bleed and more tailing than the Rtx[®]-1 Sim Dist column (**Figure 4b**). Although actual column lifetimes depend upon the system and type of samples analyzed, the bonded stationary phase should result in longer lifetime than its non-bonded equivalent.

Rtx[®]-1 Sim Dist columns have equivalent polarity to OV-101 and UCW-982.

In order for a stationary phase to be acceptable for ASTM methods, the column must not exhibit selective retention for aromatic hydrocarbons compared to aliphatic hydrocarbons. This is an important test because if the polarity of a column is different, the boiling point results will demonstrate a bias, especially for highly aromatic samples. The “polarity” of the bonded Rtx[®]-1 column was compared with OV-101 and UCW-982, two of the most common stationary phases currently

used for simulated distillation. The results of the calculated boiling points for aromatics compared to the published boiling points appear in Table II. All three silicone columns tested are essentially identical in they elute aromatics at a slightly lower temperature than the alkanes. This confirms the polarity of the Rtx[®]-1 column is equivalent, and the boiling range values obtained will agree with OV-101 and UCW-982 columns.

Rtx[®]-1 is an excellent choice for Sim Dist using packed columns.

Simulated distillation is one of the most common GC analyses performed in the petroleum laboratory. ASTM test methods D2887 and D3710 can be performed with either packed or capillary columns, but until now the benefits of bonded phases were available only to capillary users. The Rtx[®]-1 packed column uses a bonded stationary phase which is immobilized on Silcoport™-a specially deactivated support. The columns are prepared using Silcosteel® tubing for inertness unavailable with conventional metal tubing. Rtx[®]-1 bonded packed columns require minimal conditioning and give stable baselines and retention even after only 30 minutes of operation at 350°C. If your laboratory has been looking for a better Sim Dist analysis, Restek’s Rtx[®]-1 packed columns are the answer.

Figure 1

C5 to C44 calibration analysis after only 30 minutes conditioning.

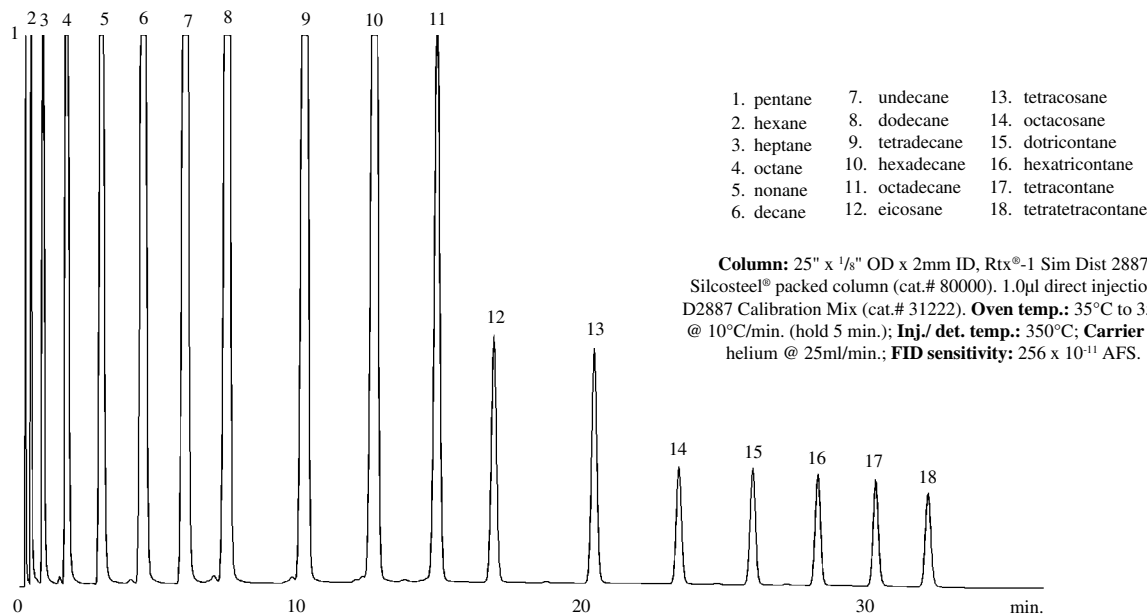
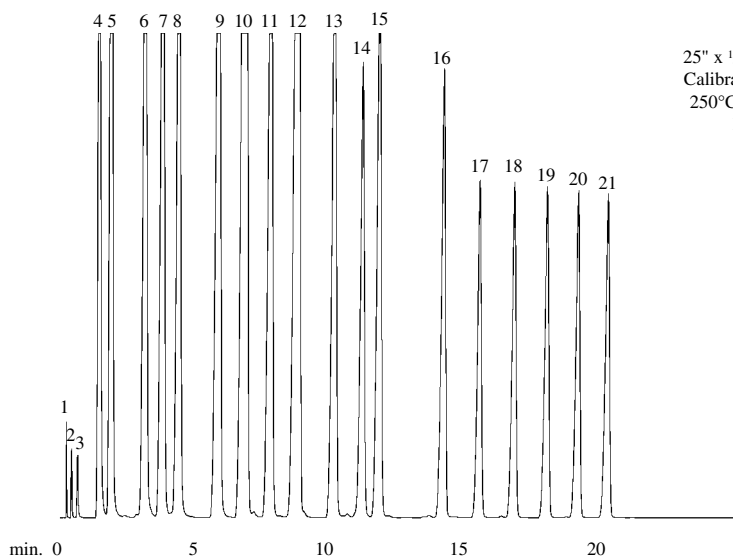


Figure 2

Rtx[®]-1 Sim Dist 2887 packed columns can also be used for ASTM D-3710 analysis..



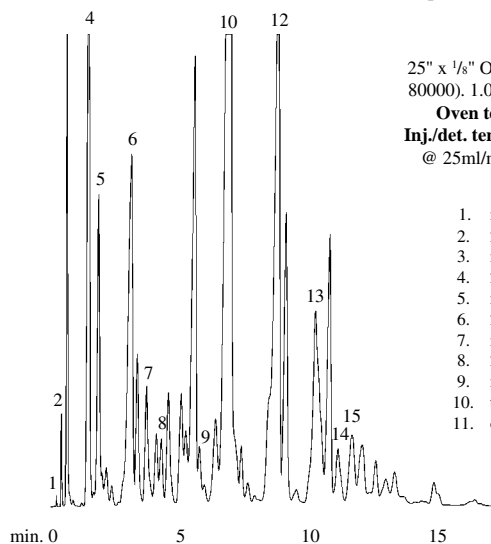
25" x 1/8" OD x 2mm ID Rtx[®]-1 SimDist 2887 (cat.# 80000). D-3710 Calibration Mix + C3, C4, C16, & C17 added. **Oven temp:** -30°C to 250°C @ 10°C/min.; **Inj./det. temp.:** 250°C / 300°C; **Carrier gas:** helium @ 25ml/min.; **FID sensitivity:** 256 x 10⁻¹¹ AFS.

- | | |
|------------------------|---------------------|
| 1. n-propane | 12. p-xylene |
| 2. 2-methyl propane | 13. n-propylbenzene |
| 3. n-butane | 14. n-decane |
| 4. 2-methylbutane | 15. n-butylbenzene |
| 5. n-pentane | 16. n-dodecane |
| 6. 2-methylpentane | 17. n-tridecane |
| 7. n-hexane | 18. n-tetradecane |
| 8. 2,4-dimethylpentane | 19. n-pentadecane |
| 9. n-heptane | 20. n-hexadecane |
| 10. toluene | 21. n-heptadecane |
| 11. octane | |

Other bonded phases are available. For more information, call Restek's technical service staff at **800-356-1688, ext.4.**

Figure 3

Perform simulated distillation of gasoline using the Rtx[®]-1 Sim Dist 2887 packed columns.



25" x 1/8" OD x 2mm ID Rtx[®]-1 SimDist 288 (cat.# 80000). 1.0µl direct injection of unleaded gasoline. **Oven temp:** -30°C to 250°C @ 10°C/min.; **Inj./det. temp.:** 250°C/300°C; **Carrier gas:** helium @ 25ml/min.; **FID sensitivity:** 256 x 10⁻¹¹ AFS.

- | | |
|------------------------|---------------------|
| 1. n-propane | 12. p-xylene |
| 2. 2-methyl propane | 13. n-propylbenzene |
| 3. n-butane | 14. n-decane |
| 4. 2-methylbutane | 15. n-butylbenzene |
| 5. n-pentane | 16. n-dodecane |
| 6. 2-methylpentane | 17. n-tridecane |
| 7. n-hexane | 18. n-tetradecane |
| 8. 2,4-dimethylpentane | 19. n-pentadecane |
| 9. n-heptane | 20. n-hexadecane |
| 10. toluene | 21. n-heptadecane |
| 11. octane | |

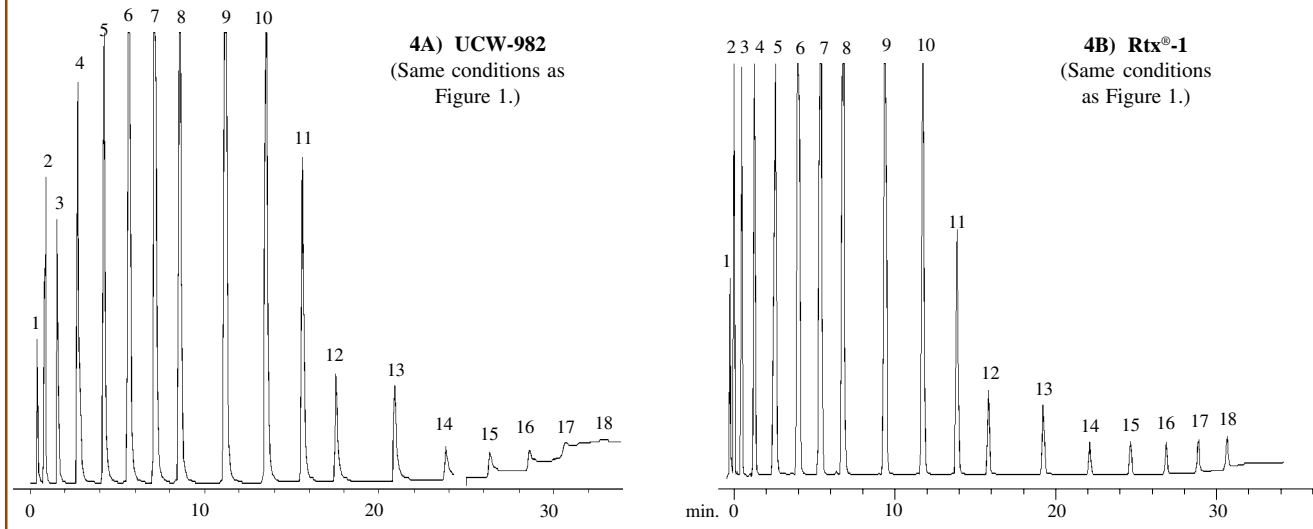
Table II

Comparison of bonded and conventional packed column indicates no polarity differences.

Aromatic Hydrocarbon	Published BP ¹ (°C)			
		Rtx [®] -1	UCW-982	OV-101
benzene	80	81.3	82	80.3
p-xylene	139	138.6	140.2	137.7
naphthalene	218	204.6	206.9	204.3
acenaphthylene	280	252.7	255.6	252.2
anthracene	342	304.1	307.2	303.4
chrysene	447	385.6	389.2	384.9
dibenzo(a,h)anthracene	524	452.3	455.7	450.4

Figure 4

Bonded packed columns exhibit lower bleed and longer lifetimes after 170 temperature cycles.



Product List

Rtx®-1 Sim Dist 2887 25" x 1/8" OD x 2mm ID Silcosteel® packed column cat.# 80000

ASTM Petrochemical Mixtures Available

Calibration and column test mixtures are available for ASTM Methods D2887 and D3710. These standards are made with the same quality and care as our environmental standards. Stock products of single ampul and cost-effective 10-packs are available for immediate shipment. Each standard is supplied with a data sheet indicating the exact concentration and a sample chromatogram.

D2887 Column Test Mix

Contain 1% (w/v) ea. of n-hexadecane and n-octadecane dissolved in n-octane

Packaged 1ml/ampul
Cat.# 31221 - single
Cat.# 31321 - 10pk.

D3710 Calibration Mix

Compound	Concentration (% w/w)
2-methylbutane	10
n-pentane	8
2-methylpentane	6
n-hexane	6
2,4-dimethylpentane	6
n-heptane	10
toluene	12
n-octane	5
p-xylene	14
n-propylbenzene	5
n-decane	4
n-butylbenzene	4
n-dodecane	4
n-tridecane	2
n-tetradecane	2
n-pentadecane	2

Packaged 1ml/ampul
Cat.# 31223 - single
Cat.# 31323 - 10pk.

D2887 Calibration Mix

Compound	Concentration (% w/w)
n-decane	12
n-dodecane	12
n-dotriacontane	1
n-eicosane	2
n-heptane	6
n-hexadecane	10
n-hexane	6
n-hexatriacontane	1
n-nonane	8
n-octacosane	1
n-octadecane	5
n-octane	8
n-tetracontane	2
n-tetratetracontane	1
n-undecane	12

Packaged 1ml/ampul
Cat.# 31222 - single
Cat.# 31322 - 10pk.

Trademarks: Rtx®, Silcosteel™ and Silcoport™ are trademarks of Restek Corporation.



© Copyright 1997, Restek Corporation

For permission to reproduce any portion of this application note, please contact Restek's publications/graphics department by phone (ext. 2128) or FAX (814) 353-9278.