RESTEK
environmental

#59190

Applications note

Separating *m*- and *p*-Xylene Isomers by US EPA Method 8260 Using an Rtx®-200 GC Column

Xylenes are aromatic hydrocarbons that naturally occur in petroleum and coal tar; they also can be commercially derived from these substrates. Three xylene isomers exist: *meta*-, *para*-, and *ortho*-xylene, usually referred to as *m*-, *p*-, and *o*-xylene, respectively. Mixed xylenes produced from petroleum contain 20% *o*- and *p*-xylene, with 44% *m*-xylene.[1] The isomers of *m*- and *p*-xylene are difficult to resolve using gas chromatography (GC) and most capillary columns. Although limited data exists suggesting toxicological differences between *m*- and *p*-xylene, there is still interest in resolving them.

The US Environmental Protection Agency (EPA) does not require separation of the xylene isomers, but rather requests their calculation as totals or sums.[2] Some states such as New York have action limits based on *m*- and *p*-xylene separately. For example the action limit in drinking water is 5µg/L for *m*-xylene and 5µg/L for *p*-xylene. A drinking water sample that has 9µg/L of total xylenes could, in fact, exceed the limits by having 9µg/L of *m*-xylene and no *p*-xylene present in the sample.[3] However, other states and agencies would consider the action limit for these two isomers as 10µg/L total.

A recent performance evaluation from New York state contained one of the isomers of xylene, which required the contracted environmental laboratories to determine actual concentrations of *m*- and *p*-xylene separately. The most common way to perform a GC separation of *m*- and *p*-xylene is by using a polyethylene glycol (PEG) stationary phase, such as the Restek Stabilwax® column. Chromatographically, baseline separation is possible; however, bleed levels are unacceptable for a mass spectrometer (MS) and sample matrices containing organic acids can contribute to bleed from the stationary phase.

The more ideal column choice for this particular separation is the Rtx®-200 column. The Crossbond® trifluoropropylmethyl polysiloxane stationary phase (**Figure 1**) features exceptionally low bleed at common volatile application working temperatures because its maximum operating temperature is 360° C.

The Rtx®-200 column provides unique separation of volatile organic compounds (VOCs) listed in US EPA Methods 524 and 8260 (**Figure 2**), making it the best column to separate xylene isomers for specific state requirements (**Figure 3**). The Rtx®-200 column's only limitation is the resolution of the gases—peaks 2, 3, 5, and 6 (**Figure 2**).

This column also is a good choice for analyzing samples having complex matrices; analyses where coelutions of several compounds can make identification of tentitively identified compounds (TICs) nearly impossible on a "624" phase. Trifluoropropyl stationary phases like that of the Rtx®-200 column have a unique selectivity because of the electrophilic nature of the fluorine-containing polymer. This creates interactions with electron-rich molecules like ketones and halogenated compounds. This unique selectivity results in different elution orders and resolves compounds that phenyl, cyano and methyl phases cannot. In this analysis, the Rtx®-200 column can be used to confirm TICs and resolve multiple coelutions.

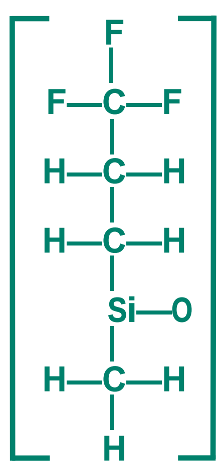
References

1. *Toxicological Profile for Total Xylenes. Prepared by Clements Associates, Inc.. under Contract No. 205-88-0608. Prepared for Agency for Toxic Substances and Disease Registry, US Public Health Services, Atlanta, GA. December 1990.*

2. *US EPA Method 8000B, Rev. 2. Determinative Chromatographic Separations. Page 7 Section 3.3.3. Washington, DC. December 1996.*

3. *Consumer Confidence Report. New York Water Service Corporation. 60 Brooklyn Avenue Merrick, New York, NY. September 1999.*

Figure 1—Phase Structure
Rtx®/MXT®-200 trifluoropropylmethyl polysiloxane



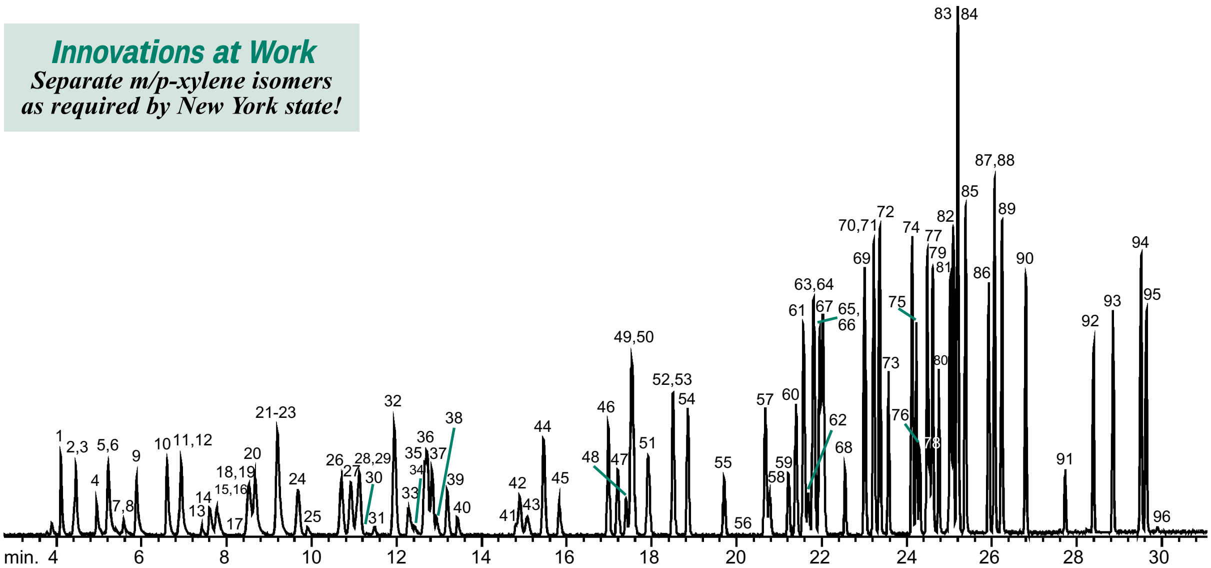
Polarity: selective for lone pair electrons.

Uses: environmental samples, solvents, Freon® samples, drugs, ketones and alcohols.

Figure 2

The Rtx®-200 column provides unique separation of the VOCs listed in US EPA Method 8260.

Innovations at Work
Separate m/p-xylene isomers as required by New York state!



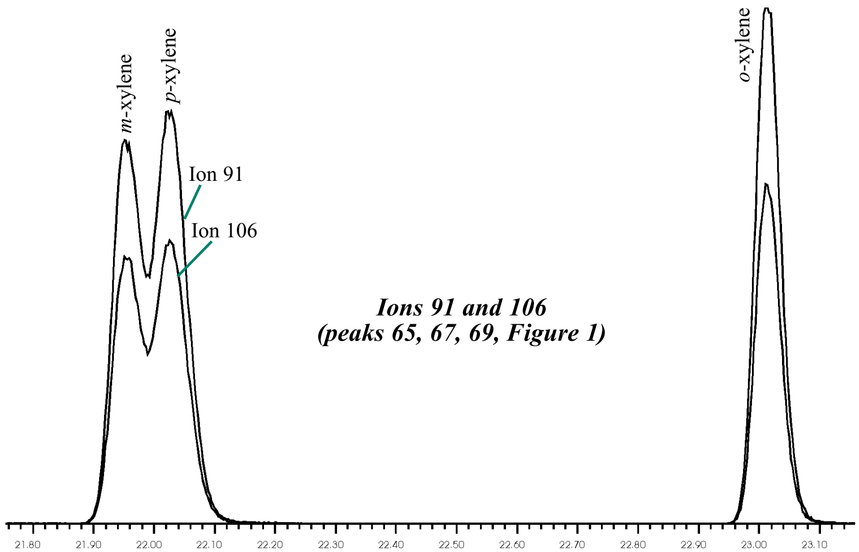
60m, 0.25 mm ID, 1.0µm Rtx-200 (cat.# 15056)
Compounds in at 10 ppb in 5mL of RO water.
Ketones, alcohols in at 2x (unless otherwise noted).

Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb 3000 (type K)
Purge:	11 min. @ 40mL/min. @ ambient temperature
Dry Purge:	1 min. @ 40mL/min.
Desorb Preheat:	245°C
Desorb:	250°C for 2 min., flow 10mL/min.
Bake:	260°C for 8 min.
Interface:	transfer line 0.53mm ID Silcosteel MXT tubing
Oven Program:	40°C (hold 10 min.) to 100°C @ 6°C/min. (hold 1 min.) to 210°C @ 30°C/min. (hold 7 min.)
Carrier Gas:	helium @ ~1.3mL/min. constant flow Adjust dichlorodifluoromethane to a retention time of 4.1 min. @ 40°C.
Detector:	Agilent 5973 MS, scan range 35 to 300 AMU

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. carbon disulfide
8. ethanol (2500ppb)
9. 1,1-dichloroethene
10. methylene chloride
11. allyl chloride
12. trans-1,2-dichloroethene
13. *tert*-butyl alcohol (100ppb)
14. methyl *tert*-butyl ether
15. allyl alcohol (250ppb)
16. diisopropyl ether
17. propargyl alcohol (500ppb)
18. 1,1-dichloroethane
19. ethyl-*tert*-buyl ether
20. chloroform
21. dibromofluoromethane
22. *cis*-1,2-dichloroethene
23. bromochloromethane
24. carbon tetrachloride
25. acetone
26. 2,2-dichloropropane
27. 1,1,1-trichloroethane
28. vinyl acetate
29. 1,1-dichloropropene
30. isobutyl alcohol (500ppb)
31. acrylonitrile
32. benzene
33. *tert*-amyl-methyl ether
34. tetrahydrofuran
35. 1,2-dichloroethane
36. trichloroethene
37. bromodichloromethane
38. methyl acrylate
39. dibromomethane
40. ethyl acetate
41. 2-butanone
42. 1,4-difluorobenzene
43. pentafluorobenzene
44. 1,2-dichloropropane
45. isopropyl acetate
46. *cis*-1,3-dichloropropene
47. dibromochloromethane
48. methyl methacrylate
49. toluene
50. toluene-d8
51. tetrachloroethene
52. 1,1,2-trichloroethane
53. *n*-propyl acetate
54. *trans*-1,3-dichloropropene
55. 1,2-dibromoethane
56. pyridine (250ppb)
57. 1,3-dichloropropane
58. ethyl methacrylate
59. bromoform
60. 1,1,1,2-tetrachloroethane
61. ethylbenzene
62. 4-methyl-2-penanone
63. chlorobenzene
64. chlorobenzene-D5
65. *m*-xylene
66. 2-picoline (250ppb)
67. *p*-xylene
68. *n*-butyl acetate
69. *o*-xylene
70. stryrene
71. 2-hexanone
72. isopropylbenzene
73. 1,1,2,2-tetrachloroethane
74. *n*-propylbenzene
75. bromobenzene
76. 4-bromo-1-fluorobenzene
77. 1,3,5-trimethylbenzene
78. pentachloroethane
79. 2-chlorotoluene
80. 1,2,3-trichloropropane
81. 4-chlorotoluene
82. *tert*-butylbenzene
83. 1,2,4-trimethylbenzene
84. *sec*-butylbenzene
85. *p*-isopropyltoluene
86. 1,3-dichlorobenzene
87. 1,2-dichlorobenzene-d4
88. 1,4-dichlorobenzene
89. *n*-butylbenzene
90. 1,2-dichlorobenzene
91. 1,2-dibromo-3-chloropropan
92. hexachlorobutadiene
93. 1,2,3-trichlorobenzene
94. naphthalene
95. 1,2,4-trichlorobenzene
96. nitrobenzene (250ppb)

Figure 3

An extracted ion chromatogram shows the Rtx®-200 column is the best column choice to separate m/p-xylene isomers.



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Interface:	transfer line 0.53mm ID Silcosteel MXT tubing
Oven Program:	40°C (hold 10 min.) to 100°C @ 6°C/min. (hold 1 min.) to 210°C @ 30°C/min. (hold 7 min.)
Carrier Gas:	helium @ ~1.3mL/min. constant flow. Adjust dichlorodifluoromethane to a retention time of 4.1 min. @ 40°C.
Detector:	Agilent 5973 MS, scan range 35 to 300 AMU

Product Listing:

Coiled Silcosteel® Tubing

Silcosteel®-Treated Welded/Drawn Grade 304 Stainless Steel Tubing
Sold by the foot—5 ft. minimum.

cat.#	ID, OD
20590	0.011" ID (0.28mm ID), 0.022" OD (0.56mm OD)
20591	0.021" ID (0.53mm ID), 0.029" OD (0.74mm OD)
20592	0.010" ID (0.25mm ID), 1/16" OD (1.59mm OD)
20593	0.020" ID (0.51mm ID), 1/16" OD (1.59mm OD)
20594	0.030" ID (0.76mm ID), 1/16" OD (1.59mm OD)
20595	0.040" ID (1.02mm ID), 1/16" OD (1.59mm OD)
0.020" wall:	
20596	0.085" ID (2.16mm ID), 1/8" OD (3.18mm OD)
0.020" wall:	
20597	0.210" ID (5.33mm ID), 1/4" OD (6.35mm OD)

Silcosteel®-Treated Seamless 316 Grade Stainless Steel Tubing
Sold by the foot—5 ft. minimum.

cat.#	ID, OD
0.035" wall:	
20598	0.055" ID (1.40mm ID), 1/8" OD (3.18mm OD)
0.035" wall:	
20599	0.180" ID (4.57mm ID), 1/4" OD (6.35mm OD)

Call for availability of lengths greater than 1000 ft.

***Metric conversion:** 6 ft. (1.8m), 25 ft. (7.6m), 50 ft. (15.2m), 200 ft. (61m), >400 ft. (>122m)

Other lengths and sizes of Silcosteel® tubing are available on a custom basis!

Straight Silcosteel® Tubing

0.085" ID (2.16mm), **1/8" OD** (3.18mm)

Length	Individual	5-Pack
18" (457mm)	20575	20576

0.210" ID (5.33mm), **1/4" OD** (6.35mm)

Length	Individual	5-Pack
18" (457mm)	20577	20578

Minimum Bend Radius
(dependent on OD)

OD	Min. Bend Radius
£1/16"	1"
1/8"	2"
1/4"	4"

Column product listing continued on back.

Product Listing:

Rtx®-200 (Fused Silica)

(Crossbond® trifluoropropylmethyl polysiloxane) Stable to 360°

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.10	-20 to 320/340°C	15005	15008	15011	
	0.25	-20 to 320/340°C	15020	15023	15026	15029
	0.50	-20 to 310/330°C	15035	15038	15041	15044
	1.00	-20 to 290/310°C	15050	15053	15056	15059
0.32mm	0.10	-20 to 320/340°C	15006	15009	15012	
	0.25	-20 to 320/340°C	15021	15024	15027	15030
	0.50	-20 to 310/330°C	15036	15039	15042	15045
	1.00	-20 to 290/310°C	15051	15054	15057	15060
	1.50	-20 to 280/300°C	15066	15069	15072	15075
0.53mm	0.10	-20 to 310/330°C	15007	15010	15013	
	0.25	-20 to 310/330°C	15022	15025	15028	
	0.50	-20 to 300/320°C	15037	15040	15043	
	1.00	-20 to 290/310°C	15052	15055	15058	
	1.50	-20 to 280/300°C	15067	15070	15073	
	3.00	-20 to 260/280°C	15082	15085	15088	15091
ID	**df (µm)**	**temp. limits**	**10-Meter**	**20-Meter**	**40-Meter**	
0.18mm	0.20	-20 to 310/330°C	45001	45002	45003	
	0.40	-20 to 310/330°C	45010	45011	45012	

MXT®-200 (Silcosteel®)

(Crossbond® trifluoropropylmethyl polysiloxane) Stable to 360°

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.25mm	0.50	-20 to 330°C	75035	75038	
	1.00	-20 to 310°C	75050	75053	
0.53mm	1.00	-20 to 290/310°C	75052	75055	75058
	1.50	-20 to 280/300°C	75067	75070	75073
	3.00	-20 to 260/280°C	75082	75085	75088
ID	**df (µm)**	**temp. limits***	**10-Meter**	**20-Meter**	**40-Meter**
0.18mm	0.20	-20 to 310/330°C	71881	71882	71883
	0.40	-20 to 310/330°C	71884	71885	71886

**The maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.*

Trademarks: *Stabilwax, Silcosteel, Rtx, Crossbond (Restek). Vocarb, (Supelco). Freon (E.I. du Pont de Nemours & Co., Inc.)*