

Reduce Helium Consumption by 68%

Using Nitrogen Purge Gas for VOCs in Water



- Save 490 mL of helium per sample by switching to nitrogen purge gas.
- Spend less money on lab gases and reduce your dependence on helium.
- Easily resolve critical Method 524.4 compounds using an Rtx[®]-VMS column.

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Reduce Helium Consumption by 68% Using Nitrogen Purge Gas for VOCs in Water

By now, everyone has felt the impact of the helium supply problem—even regulatory agencies. In order to help laboratories reduce helium use, EPA has revised Method 524.3 to allow for the use of nitrogen as a purge gas. Although labs have been doing this for years with other methods, substantial reductions in helium consumption can now be obtained when analyzing purgeable organic compounds in water by GC-MS using the revised method. By switching to nitrogen purge gas using Method 524.4, you can save an impressive 490 mL of helium per sample, which translates into a 68% reduction in helium consumption (Table I). Saving nearly 0.5 L of helium per sample quickly adds up to considerable cost savings and also insulates labs from the impact of fluctuating helium availability.

In addition to reducing helium consumption by using nitrogen purge gas, employing an Rtx®-VMS column for this analysis allows all Method 524.4 criteria to be easily met. The Rtx®-VMS column is recommended for purge-and-trap GC-MS analysis of VOCs by Method 524.4

because the selectivity of this column provides ample separation between all critical compounds. As shown in Figure 1, good resolution is obtained for target analytes including o-xylene and styrene, as well as 1,1,1-trichloroethane and carbon tetrachloride. The Rtx®-VMS column is listed in Method 524.4 and was used by the EPA to establish method performance [1]. No interference from overlapping peaks was observed and the small bore 0.25 mm column results in higher column efficiency and improved separations.

Labs analyzing purgeable organic compounds in water can save money and reduce helium dependence by using Method 524.4 with nitrogen purge gas and an Rtx®-VMS column. To compare results achieved with nitrogen purge gas to those obtained using helium, visit www.restek.com/524blog

References

[1] U.S. Environmental Protection Agency, Method 524.4, Measurement of Purgeable Organic Compounds in Water by Gas Chromatography/Mass Spectrometry Using Nitrogen Purge Gas, May 2013. <http://water.epa.gov/scitech/drinkingwater/labcert/upload/815R13002.pdf> (accessed December 19, 2013)

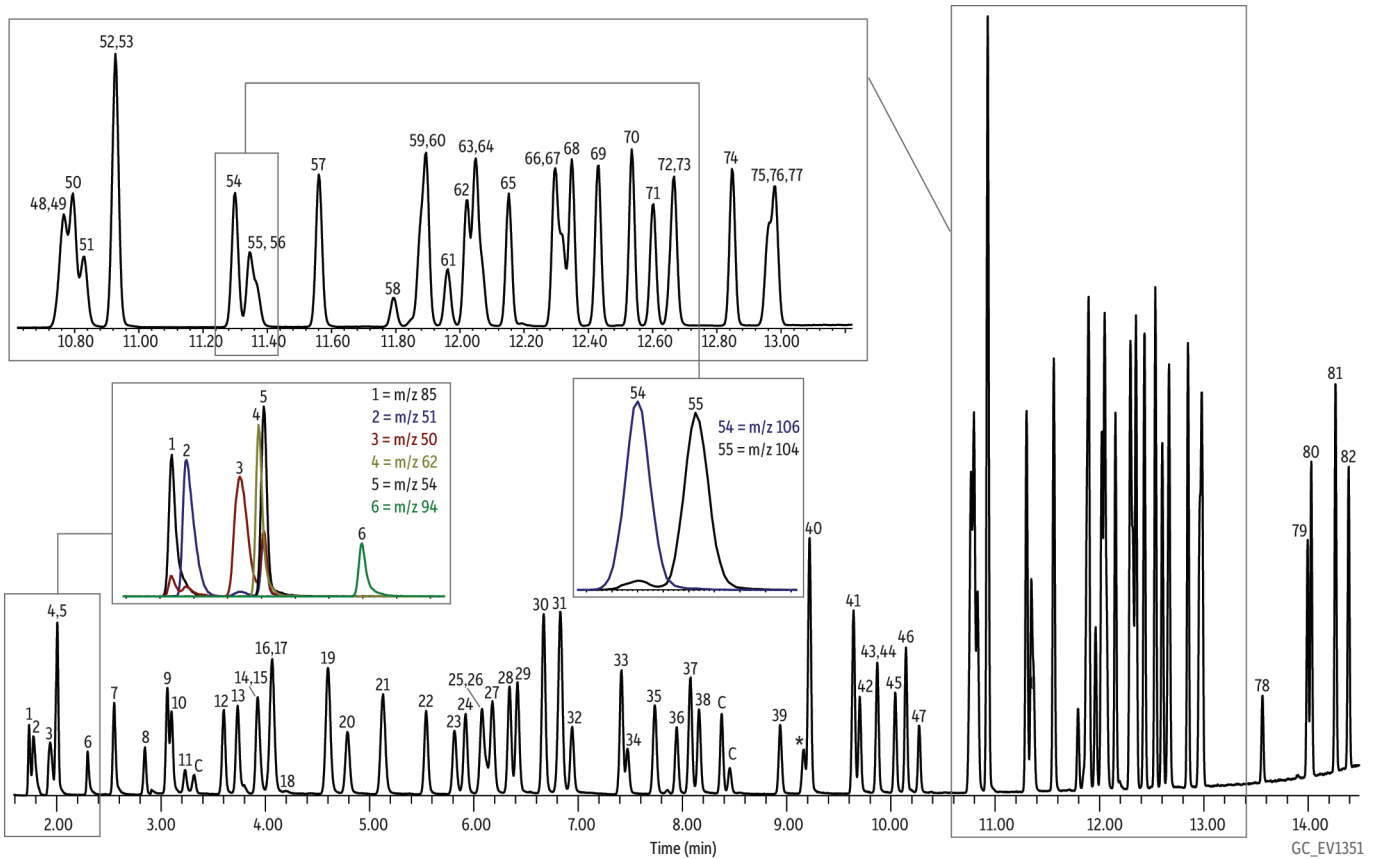
Table I: Using nitrogen purge gas allows labs to save reduce helium use by 68%.

Instrument	Analysis Step	He Volume	N ₂ Volume
P&T	Purge	—	440
	Dry Purge	—	50
	Desorb	30.9	—
GC-MS	Split Vent*	140	—
	Carrier	13.5	—
	Septum Purge	45	—
		Total Gas Volume	719.4
		Volume of Helium Saved	490
		Percent of Helium Saved	68%

*Gas saver is turned to 10 mL/min @ 1 min.

Figure 1: The Rtx®-VMS column is specifically designed for separating purgeable organic compounds, which means it provides excellent resolution of the Method 524.4 VOCs analyzed here using nitrogen purge gas.

- Peaks**
- | | | | | |
|--------------------------------------|---|--|-------------------------------|---|
| 1. Dichlorodifluoromethane | 17. MTBE | 34. 1,4-Difluorobenzene | 51. 1,1,1,2-Tetrachloroethane | 68. 1,2,4-Trimethylbenzene |
| 2. Chlorodifluoromethane | 18. <i>tert</i> -Butyl alcohol (TBA) | 35. <i>tert</i> -Amyl ethyl ether (TAEF) | 52. <i>m</i> -Xylene | 69. <i>sec</i> -Butylbenzene |
| 3. Chloromethane | 19. Diisopropyl ether (DIPE) | 36. Dibromomethane | 53. <i>p</i> -Xylene | 70. 4-Isopropyltoluene |
| 4. Vinyl chloride | 20. 1,1-Dichloroethane | 37. 1,2-Dichloropropane | 54. <i>o</i> -Xylene | 71. 1,3-Dichlorobenzene |
| 5. 1,3-Butadiene | 21. <i>tert</i> -Butyl ethyl ether (ETBE) | 38. Bromodichloromethane | 55. Styrene | 72. 1,4-Dichlorobenzene-d4 |
| 6. Bromomethane | 22. <i>cis</i> -1,2-Dichloroethane | 39. <i>cis</i> -1,3-Dichloropropene | 56. Bromoform | 73. 1,4-Dichlorobenzene |
| 7. Trichlorofluoromethane | 23. Bromochloromethane | 40. Toluene | 57. Isopropylbenzene | 74. <i>n</i> -Butylbenzene |
| 8. Diethyl ether | 24. Chloroform | 41. Tetrachloroethane | 58. 4-Bromofluorobenzene (SS) | 75. Hexachloroethane |
| 9. 1,1-Dichloroethene | 25. Carbon tetrachloride | 42. <i>trans</i> -1,3-Dichloropropene | 59. Bromobenzene | 76. 1,2-Dichlorobenzene-d4 (SS) |
| 10. Carbon disulfide | 26. Tetrahydrofuran | 43. 1,1,2-Trichloroethane | 60. <i>n</i> -Propylbenzene | 77. 1,2-Dichlorobenzene |
| 11. Methyl iodide | 27. 1,1,1-Trichloroethane | 44. Ethyl methacrylate | 61. 1,1,2,2-Tetrachloroethane | 78. 1,2-Dibromo-3-chloropropane |
| 12. Allyl chloride | 28. 1,1-Dichloropropene | 45. Dibromochloromethane | 62. 2-Chlorotoluene | 79. Hexachlorobutadiene |
| 13. Methylene chloride | 29. 1-Chlorobutane | 46. 1,3-Dichloropropane | 63. 1,3,5-Trimethylbenzene | 80. 1,2,4-Trichlorobenzene |
| 14. <i>trans</i> -1,2-Dichloroethene | 30. Benzene | 47. 1,2-Dibromoethane | 64. 1,2,3-Trichloropropane | 81. Naphthalene |
| 15. Methyl acetate | 31. <i>tert</i> -Amyl methyl ether (TAME) | 48. Chlorobenzene-d5 | 65. 4-Chlorotoluene | 82. 1,2,3-Trichlorobenzene |
| 16. MTBE-d3 (SS) | 32. 1,2-Dichloroethane | 49. Chlorobenzene | 66. <i>tert</i> -Butylbenzene | C. Contamination from nitrogen gas line; * Toluene-d8 |
| | 33. Trichloroethene | 50. Ethylbenzene | 67. Pentachloroethane | |



Column Rtx®-VMS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 19915)
Sample 524.3 Internal standard/surrogate mix (cat.# 30017)
 524.3 Gas calibration mix (cat.# 30014)
 524.3 VOA MegaMix® standard (cat.# 30013)
Diluent: RO water
Conc.: 40 ng/mL (5 mL sample)
Injection purge and trap split (split ratio 30:1)
Liner: Restek Premium 1.0 mm ID straight inlet liner (cat.# 23333.1)
Inj. Temp.: 200 °C
Purge and Trap
Instrument: EST Encon Evolution
Trap Type: Vocabr 3000
Purge: 11 min, flow 40 mL/min
Dry Purge: 1 min, flow 50 mL/min
Desorb: 1 min @ 260 °C, flow 30.9 mL/min
Bake: 8 min @ 265 °C
Interface
Connection: injection port
Transfer Line Temp.: 150 °C
Oven
Oven Temp.: 45 °C (hold 4.5 min) to 100 °C at 12 °C/min to 240 °C at 25 °C/min (hold 1.32 min)

Carrier Gas He, constant flow
Flow Rate: 0.9 mL/min
Detector MS
Mode: Scan
Scan Program:

Group	Start Time (min)	Scan Range (amu)	Scan Rate (scans/sec)
1	1.5	47-300	5.4
2	2.9	35-300	5.19

Transfer
Line Temp.: 240 °C
Analyzer Type: Quadrupole
Source Temp.: 230 °C
Quad Temp.: 150 °C
Electron Energy: 70 eV
Solvent
Delay Time: 1.5 min
Tune Type: BFB
Ionization Mode: EI
Instrument Agilent 7890A GC & 5975C MSD
Notes Nitrogen was used as the purge gas for the EST Encon Evolution.

For EPA 524.4 analysis, Restek recommends...



524.3 VOA MegaMix® Standard (69 components)

Allyl chloride (3-chloropropene) (107-05-1)
tert-Amyl ethyl ether (TAE) (919-94-8)
tert-Amyl methyl ether (TAME) (994-05-8)
 Benzene (71-43-2)
 Bromobenzene (108-86-1)
 Bromochloromethane (74-97-5)
 Bromodichloromethane (75-27-4)
 Bromoform (75-25-2)
tert-Butanol (TBA) (75-65-0)
n-Butylbenzene (104-51-8)
sec-Butylbenzene (135-98-8)
tert-Butylbenzene (98-06-6)
 Carbon disulfide (75-15-0)
 Carbon tetrachloride (56-23-5)
 Chlorobenzene (108-90-7)
 Chloroform (67-66-3)
 1-Chlorobutane (butyl chloride) (109-69-3)
 2-Chlorotoluene (95-49-8)
 4-Chlorotoluene (106-43-4)
 Dibromochloromethane (124-48-1)
 1,2-Dibromo-3-chloropropane (96-12-8)
 Dibromomethane (74-95-3)
 1,2-Dibromoethane (EDB) (106-93-4)
 1,2-Dichlorobenzene (95-50-1)
 1,3-Dichlorobenzene (541-73-1)
 1,4-Dichlorobenzene (106-46-7)
 1,1-Dichloroethane (75-34-3)
 1,2-Dichloroethane (107-06-2)
 1,1-Dichloroethene (75-35-4)
cis-1,2-Dichloroethene (156-59-2)
trans-1,2-Dichloroethene (156-60-5)
 1,2-Dichloropropane (78-87-5)
 1,3-Dichloropropane (142-28-9)
 1,1-Dichloropropene (563-58-6)
cis-1,3-Dichloropropene (10061-01-5)
trans-1,3-Dichloropropene (10061-02-6)
 Diethyl ether (ethyl ether) (60-29-7)
 Diisopropyl ether (DIPE) (108-20-3)
 Ethylbenzene (100-41-4)
 Ethyl-*tert*-butyl ether (ETBE) (637-92-3)
 Ethyl methacrylate (97-63-2)
 Hexachlorobutadiene (87-68-3)
 Hexachloroethane (67-72-1)
 Iodomethane (methyl iodide) (74-88-4)
 Isopropylbenzene (cumene) (98-82-8)
 4-Isopropyltoluene (*p*-cymene) (99-87-6)
 Methyl acetate (79-20-9)
 Methyl-*tert*-butyl ether (MTBE) (1634-04-4)
 Methylene chloride (dichloromethane) (75-09-2)
 Naphthalene (91-20-3)
 Pentachloroethane (76-01-7)
n-Propylbenzene (103-65-1)
 Styrene (100-42-5)
 Tetrachloroethene (127-18-4)
 1,1,1,2-Tetrachloroethane (630-20-6)
 1,1,2,2-Tetrachloroethane (79-34-5)
 Tetrahydrofuran (109-99-9)
 Toluene (108-88-3)
 1,2,3-Trichlorobenzene (87-61-6)
 1,2,4-Trichlorobenzene (120-82-1)
 1,1,1-Trichloroethane (71-55-6)
 1,1,2-Trichloroethane (79-00-5)
 Trichloroethene (79-01-6)
 1,2,3-Trichloropropane (96-18-4)
 1,2,4-Trimethylbenzene (95-63-6)
 1,3,5-Trimethylbenzene (108-67-8)
m-Xylene (108-38-3)
o-Xylene (95-47-6)
p-Xylene (106-42-3)

2,000 µg/mL each in P&T methanol, 1 mL/ampul

cat.# 30013 (ea.)



Rtx®-VMS Columns

(fused silica) (proprietary Crossbond® phase)

- Application-specific columns for volatile organic pollutants by GC-MS.
- Stable to 260 °C.
- Complete separation of U.S. EPA Method 8260B compounds in less than 10 minutes.
- Proprietary Restek® phase.

ID	df	temp. limits	30-Meter	60-Meter
0.25 mm	1.40 µm	-40 to 240/260 °C	19915	19916



Parker Balston® Nitrogen Gas Generators

- Produces ultra-pure nitrogen (up to 99.9999%).
- Require only a compressed air source and 110 volt AC power.
- Typical applications include GC carrier gas, make-up gas, and low flow sample concentrators.
- Maintenance kits include replacement filters.

CE

	Model #	qty.	cat.#
Nitrogen Generator	HPN2-1100 (ultra-high purity)	ea.	21653
Maintenance Kits	Model #	qty.	cat.#
Annual Maintenance Kit Includes: 1st and 2nd stage prefilters (1 each) and 1 final filter	for HPN2-1100, 76-92	kit	21649
Annual Maintenance Kit Includes: 1st, 2nd, and 3rd stage prefilters (1 each) and 1 final filter	for UHPN2-1100, 76-94, 76-96	kit	21655

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