

Simplify GC/MS Calibration for Volatile Organic Compounds

Rtx®-VMS Column Reduces Analysis Time, Provides Key Separations

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- ✓ Fast turnaround: **analysis completed in 11 minutes** on an Rtx®-VMS column.
- ✓ 84 Method 524.2 target compounds in only 3 mixes—new MegaMix™ includes 73 compounds.
- ✓ Gases and ketones in separate mixes.

According to methodology established by most regulatory agencies, volatile organic compounds and surrogates with low solubility in water (purgeable organics) are extracted from the sample matrix by a purge and trap procedure, then are analyzed by GC/MS. The US Environmental Protection Agency developed Method 524.2 for analysis of purgeable organic compounds in drinking water. The EPA method is applicable to a wide range of organic compounds, including four trihalomethane disinfection by-products that have suitable volatility/water solubility characteristics (bromodichloromethane, bromoform, chlorodibromomethane, chloroform).

Our new calibration mix for volatiles, Drinking Water VOA MegaMix™ 524.2 Rev 4.2, consists of 73 target compounds in EPA Method 524.2. The only compounds listed in Method 524.2 not included in

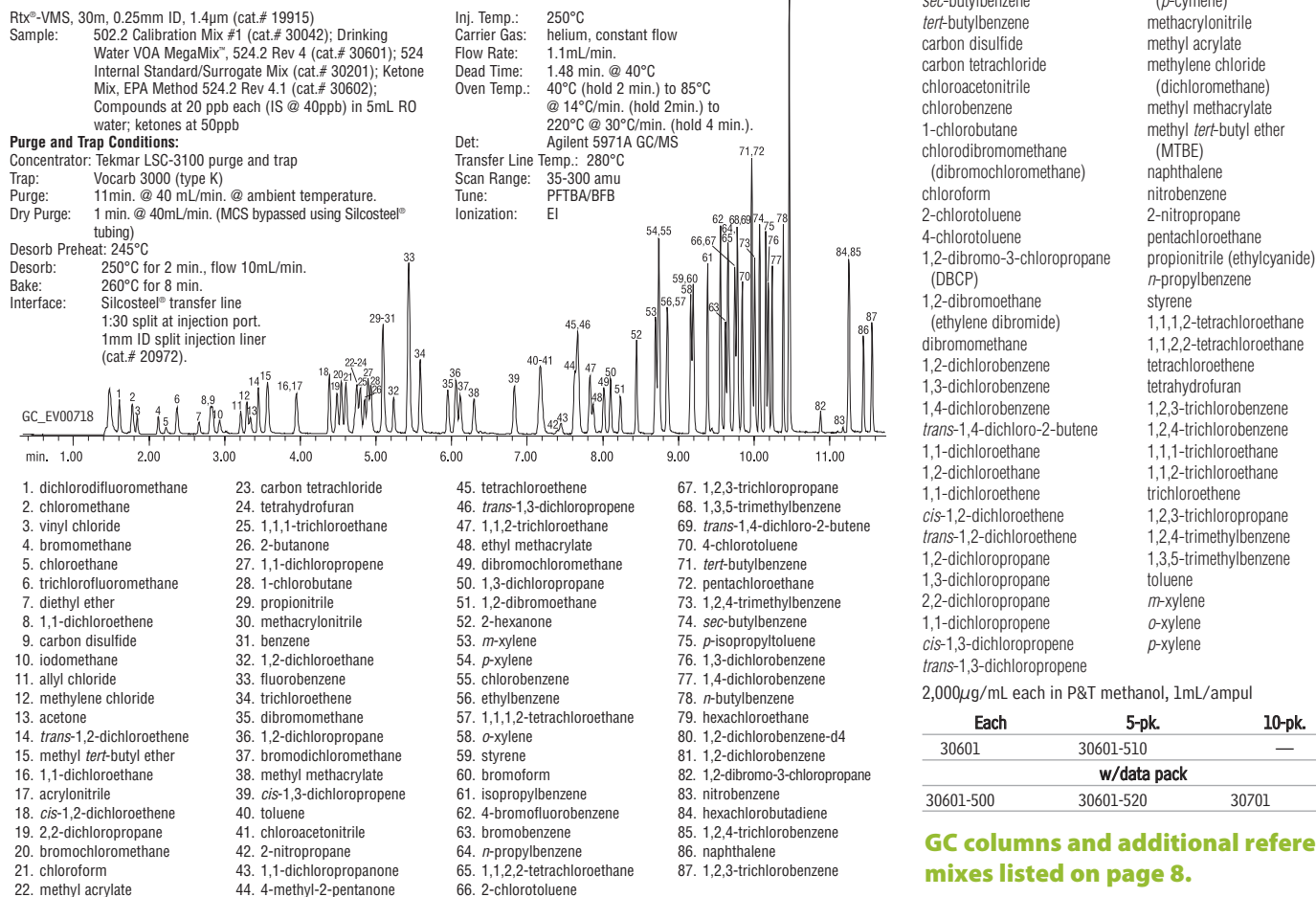
the MegaMix™ are the six compounds that are gases under standard pressure and temperature conditions, and five ketones that present long-term stability problems. We have offered a calibration mix of the six gases for some time (cat.# 30042), and now introduce a mix of the five ketones, in a purge & trap methanol/water (90:10) solution. Our research showed that this solvent system protects the keto groups and prevents acetal formation, a potential problem with ketones in 100% methanol. These optimizations assure accurate analytical results. We also offer the additional oxygenates specified by the State of California. Our fortification solution (cat.# 30201) completes the selection of reference materials needed for the method.

Productivity is very important to environmental laboratories, and in volatiles analysis purge and trap

cycle time and oven cycle time are the limiting parameters. A 30m x 0.25mm ID x 1.4µm Rtx®-VMS column (cat.# 19915) is an excellent choice for analyzing the 84 volatile organic compounds listed in EPA Method 524.2 (Figure 1). The narrow bore column rapidly separates the target compounds and surrogates and improves resolution of traditionally coeluting compounds, such as carbon tetrachloride / 1,1,1-trichloroethane (peaks 23 and 25). Rtx®-VMS columns were designed specifically to be compatible with higher starting temperatures: an initial temperature of 40°C shortens analysis time, compared to the 35°C initial temperature dictated by most columns, and the six early eluting gases still are resolved to the baseline (Figure 1).

Additional resolution between these pairs is possible with a slower initial ramp rate. Analysis time is less than 12 minutes, and cycle time is 16 minutes, a total well below the cycle time for a standard purge and trap system. This allows the fastest analyses currently available using a Tekmar 3100 purge and trap unit coupled with a single GC. (Continued on pg. 8.)

Figure 1 11-minute separation of the volatile organic target compounds in US EPA Method 524.2, with improved resolution, using an Rtx®-VMS column.



Drinking Water VOA MegaMix™, 524.2 Rev. 4.1

acrylonitrile	diethyl ether (ethyl ether)
allyl chloride	ethylbenzene
benzene	ethyl methacrylate
bromobenzene	hexachlorobutadiene
bromochloromethane	hexachloroethane
bromodichloromethane	iodomethane (methyl iodide)
bromoform	isopropylbenzene (cumene)
<i>n</i> -butylbenzene	4-isopropyltoluene
<i>sec</i> -butylbenzene	(<i>p</i> -cymene)
<i>tert</i> -butylbenzene	methacrylonitrile
carbon disulfide	methyl acrylate
carbon tetrachloride	methylene chloride
chloroacetonitrile	(dichloromethane)
chlorobenzene	methyl methacrylate
1-chlorobutane	methyl <i>tert</i> -butyl ether
chlorodibromomethane	(MTBE)
(dibromochloromethane)	naphthalene
chloroform	nitrobenzene
2-chlorotoluene	2-nitropropane
4-chlorotoluene	pentachloroethane
1,2-dibromo-3-chloropropane	propionitrile (ethylcyanide)
(DBCP)	<i>n</i> -propylbenzene
1,2-dibromoethane	styrene
(ethylene dibromide)	1,1,1,2-tetrachloroethane
dibromomethane	1,1,2,2-tetrachloroethane
1,2-dichlorobenzene	tetrachloroethene
1,3-dichlorobenzene	tetrahydrofuran
1,4-dichlorobenzene	1,2,3-trichlorobenzene
<i>trans</i> -1,4-dichloro-2-butene	1,2,4-trichlorobenzene
1,1-dichloroethane	1,1,1-trichloroethane
1,2-dichloroethane	1,1,2-trichloroethane
1,1-dichloroethene	trichloroethene
<i>cis</i> -1,2-dichloroethene	1,2,3-trichloropropane
<i>trans</i> -1,2-dichloroethene	1,2,4-trimethylbenzene
1,2-dichloropropane	1,3,5-trimethylbenzene
1,3-dichloropropane	toluene
2,2-dichloropropane	<i>m</i> -xylene
1,1-dichloropropene	<i>o</i> -xylene
<i>cis</i> -1,3-dichloropropene	<i>p</i> -xylene
<i>trans</i> -1,3-dichloropropene	

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

Each	5-pk.	10-pk.
30601	30601-510	—
w/data pack		
30601-500	30601-520	30701

GC columns and additional reference mixes listed on page 8.

(cont. from p. 7) Laboratories using either dual purge and trap technology or newer purge and trap units with rapid cycle times are encouraged to use our 20m x 0.18mm x 1.0µm Rtx®-VMS capillary column, for sub-10 minute runtimes.^{1,2} The 30m x 0.25mm ID column is our recommendation for the best gas reso-

lution. Even at a concentration of 20ppb in 5mL of reverse osmosis water the gases are better than 90% resolved with a 40°C starting temperature (Figure 1). For best gas resolution using a Tekmar 3100 unit we optimized the method by using an alternate packing material in the trap, as described in the conditions for Figure 1 (pg. 7).

Rtx®-VMS Columns (fused silica)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	
0.32mm	1.80	-40 to 240/260°C	19919	19920	
0.45mm	2.55	-40 to 240/260°C	19908	19909	
0.53mm	3.00	-40 to 240/260°C	19985	19988	19974
ID	df (µm)	temp. limits	20-Meter	40-Meter	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

Ketones Mix, 524.2 Rev. 4.1

acetone
2-butanone (MEK)
1,1-dichloro-2-propanone
5,000µg/mL each in P&T methanol:water (90:10), 1mL/ampul

Each	5-pk.	10-pk.
30602	30602-510	—
w/data pack		
30602-500	30602-520	30702

524 Internal Standard/Surrogate Mix fortification solution

4-bromofluorobenzene
1,2-dichlorobenzene-d4

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

Each	5-pk.	10-pk.
30201	30201-510	—
w/data pack		
30201-500	30201-520	30301



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If you are analyzing volatile environmental target analytes, Restek offers the columns, reference materials, accessories, and technical knowledge you need to get your system running quickly and accurately.

References

- Butler J.C., E. Phillips, M. Conoley *A Quick, Sensitive Solution for Meeting Short Holding Times for VOAs in Drinking Water* Application Note AN9197, Thermo Electron Corporation, 2215 Grand Avenue Parkway, Austin, TX (2003).
- Hilling, A.L. and G. Smith *Environmental Testing & Analysis* 10 (3),15-19 (2001).

502.2 Calibration Mix #1 (gases)

bromomethane
chloroethane
chloromethane

dichlorodifluoromethane
trichlorofluoromethane
vinyl chloride

Each	5-pk.	10-pk.
2,000µg/mL each in P&T methanol, 1mL/ampul		
30042	30042-510	—
30042-500*	30042-520*	30142*
200µg/mL each in P&T methanol, 1mL/ampul		
30439	30439-510	—
30439-500*	30439-520*	30539*

*w/data pack

Fluorobenzene Mix

internal standard

fluorobenzene

2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30030	30030-510	—
w/data pack		
30030-500	30030-520	30130