Application Note SI-02408

Evaluation of EPA Method 524.3: a New Draft Method for the Analysis of VOCs in Drinking Water using GC/MS and V:Results™ GC/MS Software

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Introduction

The U.S. Environmental Protection Agency (EPA) developed a draft method for the measurement of volatile organic compounds (VOCs) in drinking water[1]. The new method, known as EPA 524.3, is a direct update of the earlier version of EPA Method 524.2. Key differences from 524.2 include the use of solid acid preservatives, a revised target list, an option for use of selected ion monitoring (SIM), and more flexible guidance for optimizing purge-and-trap parameters. The application laboratory at Varian, Inc. participates in what EPA calls a Secondary Laboratory Demonstration study, to help the Agency evaluate the new method. The study allows commercial laboratories and other stakeholders to provide data and comments to EPA. The study requires participants to:

a. Analyze an acceptable calibration in the range specified in the method
b. Provide precision and accuracy data, known as a Demonstration of Laboratory Capability (DOC)
c. Establish Lowest Concentration Minimum Reporting Levels (LCMRLs) for select regulated compounds

In this note, data taken for the Secondary Laboratory Demonstration is presented using a Varian GC/MS system with a Teledyne Tekmar™ Purge and Trap Concentrator (Figure 1). Commentary is also provided on the proposed changes in EPA 524.3 relative to EPA 524.2.

Instrumentation

- Varian 240-MS ion trap mass spectrometer
- 431-GC gas chromatograph
- Varian 1177 Split/Splitless injector with 2 mm ID open liner
- Varian Archon™ Purge & Trap AutoSampler Teledyne Tekmar Stratum Purge and Trap Concentrator with #9 Trap
- V:Results GC/MS software

Figure 1. Varian Archon Purge and Trap AutoSampler with Tekmar Stratum Purge and Trap Concentrator (top) and 240-MS Ion Trap GC/MS (bottom). and Varian 240-MS Ion Trap with Varian 431-GC (bottom).

Methods and Materials

Purge and Trap and GC conditions

Column:	FactorFour™ VF-624ms, 20 m × 0.15 mm x 0.84 µm (Part no. CP9100)
GC:	35 °C for 2 min, to 200 °C at 10 °C/min, hold 0 min, to 240 °C at 50 °C/min, hold 3 min
Purge Volume:	5 mL at 40 mL/min
Desorb Time:	1 min
Desorb Temp:	260 °C
Split Ratio:	1:100

General MS Conditions

Scan Range:	m/z 35-300 (gas segment 47-150)
Scans Averaged:	2 µscans
Max Ion Time:	25,000 µsec
Emission Current:	15-20 µA
Manifold Temp:	60 °C

Transfer Line Temp: 220 °C
Ion Trap Temp: 190 °C

Results and Discussion

Comparison of EPA 524.3 and EPA 524.2

The basic differences between the old and new method versions are described in Table 1. In general, there was much greater flexibility in the new method, especially in the purge and trap (PAT) concentrator conditions. The new method also mandated the use of a limited sample purge volume of 5 mL and currently requires a chilled autosampler system to keep the samples at 10 °C during analysis (the Archon Purge and Trap AutoSampler has a chiller option to meet this requirement).

Table 1. Major differences between new and old EPA Method 524.

Requirement/Recommendation	
Former EPA Method 524.2	New EPA Method 524.3
Unrestricted purge volume	Purge volume restricted to 5 mL
No dry purge allowed, desorb time must be 4 minutes	Dry purge allowed, with general flexibility in all PAT concentrator settings
Full scan only	Selected Ion Monitoring allowed for select compounds
HCl preservative	Maleic acid and ascorbic acid preservatives
Long capillary column, typically 60 m; cryogenic cooling of GC oven permitted	Short 30 m column recommended, no cryogenics needed
3-component sorbent trap of silica gel, charcoal, and Tenax™	Can use VoCarb™ and other, less water adsorbing trap materials
No clear defined calibration procedure	Calibration range recommended with a QC procedure to check calibration curve
Some compounds with poor purge and trap efficiencies	Some poor performing compounds removed, new ethers and other compounds added
Injection of bromofluorobenzene (BFB) tuning compound every 8 hours	BFB requirement only for each new initial calibration or after major maintenance of MS
Method detection limits (MDLs) required, based on Student t statistical test	MDLs optional; must establish a minimum reporting limit (MRL) using a specific procedure
No matrix spikes required in analytical run	Matrix spikes required in QC section
No chilled autosampler compartment required	Chilled autosampler compartment needed

Initial Calibration

EPA Method 524.3 recommends the following calibration levels to be used for full scan analysis: 0.5, 1.0, 2.0, 5.0, 10.0, 20.0 and 40.0 ppb. There are no minimum response factors for selected compounds in the method. The %RSD (relative standard deviation) of the initial calibration for each analyte is not specified by the method, and in fact, the data presented in the EPA method are calculated using curves with a quadratic fit and weighting at the low end of the calibration curves. The only requirement for acceptance is that each calibration standard point, when calculated against the curve, must have a percent recovery in the range of 70-130%, except for the lowest calibration standard, which must have a recovery between 50-150%.

The method contains approximately 82 compounds with the new surrogate and internal standards. All compounds are included in the calibration mixtures and so it is important for the analyst to ensure that either the column is capable of resolving compounds that have similar quantitation ions, or that there is sufficient sensitivity to choose other quantitation ions of lower intensity. Figure 2 is a typical chromatogram showing all of the target compounds in the method.

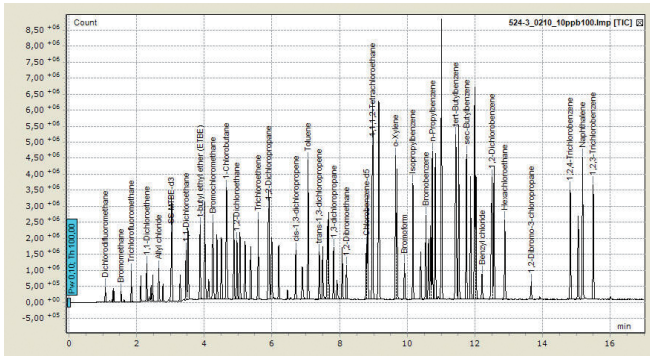

Figure 2. TIC for all target compounds. Excellent peak shape, resolution and sensitivity were easily obtained with the VF-624ms column and 240-MS GC/MS.

Table 2 contains some general calibration information. All calibration standards passed the percent recovery quality-control check. All compounds showed excellent calibration coefficient and relative standard deviation at a concentration range from 0.5 to 40 ppb. The average r^2 and %RSD of all compounds were 0.998 and 9.94%, respectively (Table 2). An example calibration curve is shown in Figure 3 for t-butyl alcohol.

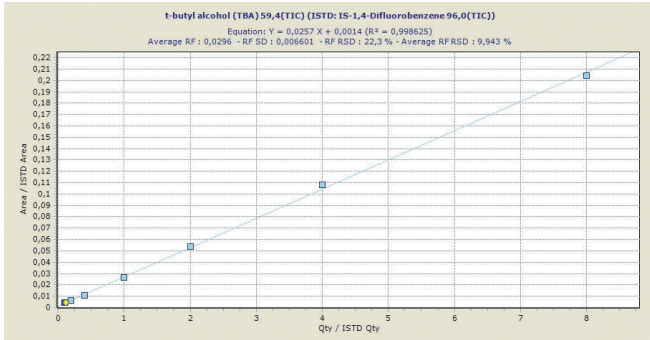

Figure 3. Calibration curve for t-butyl alcohol (TBA) from 0.5 to 40 ppb. Although typically a very poor purge-and-trap compound, TBA shows excellent response on the 240-MS GC/MS.MS.

Table 2. Calibration data for EPA 524.3 target compounds from 0.5 to 40 ppb, 5 mL purge volume.

Compound name	Correlation coefficient (r^2)	Average RF	%RSD	Method calibration requirement
Dichlorodifluoromethane	0.9964	0.0826	14.04	PASS
Chlorodifluoromethane	0.9906	0.0721	17.76	PASS
Chloromethane	0.9990	0.0344	83.27	PASS
Vinyl chloride	0.9985	0.0977	23.57	PASS
1,3-Butadiene	0.9848	0.0683	12.94	PASS
Bromomethane	0.9971	0.0479	5.05	PASS
Trichlorofluoromethane	0.9981	0.2743	10.62	PASS
Diethyl ether	0.9916	0.1070	10.50	PASS
1,1-Dichloroethene	0.9998	0.3771	7.71	PASS
Methyl iodide	0.9976	0.1661	18.97	PASS
Carbon disulfide	0.9989	0.3342	6.46	PASS
Allyl chloride	0.9990	0.2589	4.71	PASS
Methylene chloride	0.9963	0.1958	11.72	PASS
t-Butyl alcohol (TBA)	0.9986	0.0296	22.30	PASS
SS-MTBE-d3	-	0.4313	2.15	PASS
trans-1,2-Dichloroethene	0.9992	0.4107	4.77	PASS
Methyl acetate	0.9985	0.0193	8.19	PASS
MTBE	0.9998	0.3670	2.20	PASS
1,1-Dichloroethane	0.9948	0.5349	16.22	PASS
Diisopropyl ether (DIPE)	0.9980	0.6911	6.55	PASS
t-Butyl ethyl ether (ETBE)	0.9984	0.9093	7.15	PASS
cis-1,2-Dichloroethene	0.9999	0.7513	1.63	PASS
Tetrahydrofuran	0.9951	0.0089	11.16	PASS
Bromochloromethane	0.9994	0.2997	2.21	PASS
Chloroform	0.9991	0.4498	4.35	PASS
1,1,1-Trichloroethane	0.9996	0.6724	3.56	PASS
1-Chlorobutane	0.9989	0.4084	3.92	PASS
Carbon tetrachloride	0.9989	0.3536	7.81	PASS
1,1-Dichloropropene	0.9992	0.1667	6.87	PASS
Benzene	0.9999	0.5658	3.19	PASS
1,2-Dichloroethane	0.9949	0.5703	15.25	PASS
t-Amyl methyl ether (TAME)	0.9991	0.3494	13.42	PASS
Trichloroethene	0.9998	0.2494	4.38	PASS
1,2-Dichloropropane	0.9993	0.2531	4.50	PASS
t-Amyl ethyl ether (TAEE)	0.9959	0.7344	18.91	PASS
Dibromomethane	0.9997	0.3241	2.55	PASS
Bromodichloromethane	0.9998	0.3502	3.42	PASS
cis-1,3-Dichloropropene	0.9997	0.2093	5.33	PASS
Toluene	0.9996	1.6577	7.09	PASS
trans-1,3-Dichloropropene	0.9995	0.2050	6.60	PASS
Ethyl methacrylate	0.9942	0.2837	32.07	PASS
1,1,2-Trichloroethane	0.9989	0.3119	2.60	PASS
Tetrachloroethene	0.9996	0.2981	3.27	PASS
1,3-Dichloropropane	0.9999	0.2464	4.07	PASS
Dibromochloromethane	0.9988	0.4489	7.22	PASS
1,2-Dibromoethane	0.9992	0.3762	7.33	PASS
Chlorobenzene	0.9996	0.9664	1.78	PASS
1,1,1,2-Tetrachloroethane	0.9997	0.3864	4.57	PASS
Ethylbenzene	0.9997	1.6240	8.02	PASS
m,p-Xylene	0.9979	3.0508	11.20	PASS
o-Xylene	0.9984	1.5158	12.33	PASS
Styrene	0.9996	0.9237	16.61	PASS
Bromoform	0.9999	0.2899	4.75	PASS
Isopropylbenzene	0.9989	1.2787	16.88	PASS
SS-4-Bromofluorobenzene	-	0.8441	3.90	PASS
Bromobenzene	0.9996	0.8280	3.28	PASS
1,1,2,2-Tetrachloroethane	0.9995	0.9251	4.08	PASS
1,2,3-Trichloropropane	0.9998	0.7890	4.16	PASS
n-Propylbenzene	0.9992	4.2845	13.62	PASS
2-Chlorotoluene	0.9994	0.8041	6.48	PASS
4-Chlorotoluene	0.9988	0.7972	5.26	PASS
1,3,5-Trimethylbenzene	0.9989	3.4694	13.56	PASS
tert-Butylbenzene	0.9995	2.9540	13.93	PASS
Pentachloroethane	0.9993	0.6249	7.23	PASS
1,2,4-Trimethylbenzene	0.9990	3.5831	18.26	PASS
sec-Butylbenzene	0.9994	3.6829	17.81	PASS
1,3-Dichlorobenzene	0.9995	1.8812	3.28	PASS
p-Isopropyltoluene	0.9996	2.8969	20.21	PASS
Benzyl chloride	0.9938	0.7484	15.60	PASS
1,4-Dichlorobenzene	0.9993	1.9625	3.42	PASS
SS-1,2-Dichlorobenzene-d4	-	0.9351	2.25	PASS
1,2-Dichlorobenzene	0.9997	1.8328	2.73	PASS
n-Butylbenzene	0.9994	2.7817	16.81	PASS
Hexachloroethane	0.9994	0.7001	3.55	PASS
1,2-Dibromo-3-chloropropane	0.9982	0.4923	7.97	PASS
1,2,4-Trichlorobenzene	0.9983	1.2828	7.26	PASS
Hexachlorobutadiene	0.9998	0.8135	4.00	PASS
Naphthalene	0.9976	3.5956	25.29	PASS
1,2,3-Trichlorobenzene	0.9997	1.3321	5.90	PASS
Average	0.9983		9.94	

Table 3 shows the data obtained for 1,2-dibromoethane when the individual calibration points were calculated against the calibration curve. This shows passing data for the curve based on the recovery limits presented in the table.

Table 3. Example results for 1,2-dibromoethane (EDB) where each calibration point is calculated against the calibration curve and compared to recovery criteria in the method.

Compound Name	Target concentration (ppb)	Results	%Recovery	Method limit
1,2-Dibromomo-ethane	0.5	0.56	111	50-150%
	1.0	1.03	103	70-130%
	2.0	1.85	92	70-130%
	5.0	4.71	94	70-130%
	10.0	9.67	97	70-130%
	20.0	20.26	101	70-130%
	40.0	40.42	101	70-130%

Precision and Accuracy

Precision and accuracy are determined after the completion of a valid calibration. Laboratories perform a Demonstration of Capability (DOC), in which a minimum of seven replicates in reagent water are analyzed. Table 4 provides a summary of the results. Note that the limits for the %RSD and %Recovery are 20% for each compound. All compounds easily met the requirements, with an average %Recovery of 102.6% and average %RSD of 4.67%.

Table 4. Precision and accuracy data for EPA Method 524.3 compounds.

Compound name	True value	DOC-A	DOC-B	DOC-C	DOC-D	DOC-E	DOC-F	DOC-G	Qty %RSD	Average %Recovery	DOC
Dichlorodifluoromethane	10	9.28	8.82	8.06	8.24	9.89	9.21	8.59	8.78	88.70	PASS
Chlorodifluoromethane	10	10.86	10.52	10.38	9.5	11.4	10.43	10.97	5.62	105.80	PASS
Chloromethane	10	8.01	6.95	8.53	8.08	10.04	9.53	7.35	13.31	83.56	PASS
Vinyl chloride	10	9.12	9.58	8.99	9.55	9.33	10.05	9.33	3.70	94.21	PASS
1,3-Butadiene	10	12.45	11.68	12.9	10.56	13.36	12	13.58	8.50	123.61	PASS
Bromomethane	10	9.19	9.36	9.03	8.72	9.03	8.63	9.18	2.93	90.20	PASS
Trichlorofluoromethane	10	9.18	9.28	8.52	9.29	9.99	9.59	8.72	5.40	92.24	PASS
Diethyl ether	10	12.76	12.85	12.74	12.93	13.1	12.87	12.88	0.93	128.76	PASS
1,1-Dichloroethene	10	9.31	9.47	9.11	9.51	9.81	9.78	9.06	3.15	94.36	PASS
Methyl iodide	10	6.33	6.7	6.76	7.07	7.71	7.65	7.42	7.38	70.91	PASS
Carbon disulfide	10	7.97	8.26	7.9	8.23	8.42	8.27	8.05	2.27	81.57	PASS
Allyl chloride	10	11.54	11.33	11.72	11.47	11.72	11.66	11.64	1.25	115.83	PASS
Methylene choride	10	12.21	12.78	12.71	12.53	13.34	12.76	13.3	3.14	128.04	PASS
t-Butyl alcohol (TBA)	10	15.07	16.39	16.33	16.61	16.26	16.66	17.96	5.15	164.69	PASS
trans-1,2-Dichloroethane	10	11.07	11.76	11.21	11.24	11.57	11.6	11.16	2.33	113.73	PASS
Methyl acetate	10	9.34	10.75	9.29	11.23	8.19	10.86	10.5	10.97	100.23	PASS
MTBE	10	10.19	10.5	10.4	10.69	10.31	10.6	10.85	2.16	105.06	PASS
1,1-Dichloroethane	10	10.12	10.32	10.21	10.16	10.2	10.66	10.28	1.78	102.79	PASS
Diisopropyl ether (DIPE)	10	11.69	11.28	12.3	11.57	12.2	11.87	12.57	3.81	119.26	PASS
t-Butyl ethyl ether (ETBE)	10	11.51	11.74	12.29	11.52	12.07	11.37	12.27	3.24	118.24	PASS
cis-1,2-Dichloroethene	10	11.53	11.33	11.63	11.47	11.77	12.01	11.93	2.13	116.67	PASS
Tetrahydrofuran	10	11.75	9.53	8.01	11.29	12.67	10.78	12.33	15.08	109.09	PASS
Bromochloromethane	10	11.13	11.54	11.84	11.59	11.83	11.99	11.86	2.50	116.83	PASS
Chloroform	10	9.91	10.13	10.07	10.14	10.72	10.38	10.48	2.72	102.61	PASS
1,1,1-Trichloroethane	10	9.99	10	9.9	10.26	10.29	10.66	9.86	2.78	101.37	PASS
1-Chlorobutane	10	9.59	9.83	9.77	9.97	10.23	9.98	9.51	2.50	98.40	PASS
Carbon tetrachloride	10	9.42	9.76	9.3	9.67	10.33	10.72	8.97	6.21	97.39	PASS
1,1-Dichloropropene	10	10.07	10.04	9.89	10.34	11.1	10.67	10.05	4.20	103.09	PASS
Benzene	10	10.18	10.36	10.71	10.5	10.86	10.88	10.55	2.45	105.77	PASS
1,2-Dichloroethane	10	11.27	11.39	11.39	11.18	11.04	11.35	11.37	1.16	112.84	PASS
t-Amyl methyl ether (TAME)	10	11.16	11.23	11.53	11.16	11.48	11.12	12.1	3.07	113.97	PASS
Trichloroethene	10	10.21	10.23	10.52	10.35	10.79	11.1	10.78	3.16	105.69	PASS
1,2-Dichloropropane	10	11.23	11.84	11.85	11.75	12.1	12.46	11.92	3.13	118.79	PASS
t-Amyl ethyl ether (TAEE)	10	12.91	12.48	13.72	12.87	13.48	12.95	13.66	3.57	131.53	PASS
Dibromomethane	10	10.97	11.2	10.85	11.18	10.97	11.31	11.47	1.96	111.36	PASS

Compound name	True value	DOC-A	DOC-B	DOC-C	DOC-D	DOC-E	DOC-F	DOC-G	Qty %RSD	Average %Recovery	DOC
Bromodichloromethane	10	9.96	10.71	10.64	10.57	10.42	10.73	10.72	2.62	105.36	PASS
cis-1,3-Dichloropropene	10	11.37	11.57	12	11.49	11.87	11.84	11.64	1.94	116.83	PASS
Toluene	10	9.7	9.6	9.66	9.72	9.9	10.55	9.41	3.72	97.91	PASS
trans-1, 3-Dichloropropene	10	10.81	10.82	11.26	10.67	10.72	10.89	10.83	1.76	108.57	PASS
Ethyl methacrylate	10	12.36	12.18	12.29	12.03	12.11	12.36	12.563	1.38	122.66	PASS
1,1,2-Trichloroethane	10	9.78	9.59	9.56	9.66	9.83	9.95	9.89	1.56	97.51	PASS
Tetrachloroethene	10	9.98	9.71	9.36	9.47	10.68	10.43	9.17	5.72	98.29	PASS
1,3-Dichloropropane	10	9.59	9.46	9.71	9.82	9.96	9.83	9.41	2.13	96.83	PASS
Dibromochloromethane	10	9.78	9.35	9.4	9.55	9.95	9.54	9.8	2.32	96.24	PASS
1,2-Dibromoethane	10	9.18	9.13	8.84	9.32	9.04	9.25	9.12	1.73	91.26	PASS
Chlorobenzene	10	9.95	9.55	9.9	9.52	10.2	10.19	9.67	2.87	98.54	PASS
1,1,1,2-Tetrachloroethane	10	10.43	9.83	9.84	10.1	10.41	10.02	9.53	3.25	100.23	PASS
Ethylbenzene	10	9.8	9.5	9.74	9.84	10.28	10.16	9.28	3.57	98.00	PASS
m,p-Xylene	10	9.85	9.29	9.69	9.55	10.29	10.26	9.37	4.11	97.57	PASS
o-Xylene	10	9.85	9.95	9.79	10.21	10.67	10.45	9.77	3.50	100.99	PASS
Styrene	10	9.43	9.38	9.6	9.6	9.13	9.87	9.43	2.44	94.91	PASS
Bromoform	10	11.23	10.74	11.04	11.04	11.2	11.13	11	1.49	110.54	PASS
Isopropylbenzene	10	10.14	9.74	10.29	10.46	10.92	10.7	9.42	5.13	100.16	PASS
Bromobenzene	10	9.53	9.1	10.22	9.94	10.34	10.9	10.08	5.79	100.16	PASS
1,1,2,2-Tetrachloroethane	10	9.24	8.99	9.57	9.65	9.68	10.27	10.06	4.55	96.37	PASS
1,2,3-Trichloropropane	10	10.69	10.16	10.85	10.7	10.91	11.18	11.24	3.33	108.19	PASS
n-Propylbenzene	10	9.31	8.68	9.49	9.51	10.07	10.49	9.49	5.99	95.77	PASS
2-Chlorotoluene	10	9.01	8.26	9.41	9.63	9.94	10.45	9.29	7.39	94.27	PASS
4-Chlorotoluene	10	9.09	8.57	9.18	9.53	9.21	10.58	9.01	6.77	93.10	PASS
1,3,5-Trimethylbenzene	10	8.98	8.4	9.13	9.47	9.71	10.3	9.05	6.52	92.91	PASS
tert-Butylbenzene	10	9.62	8.92	9.97	10.11	10.69	11.07	9.83	7.01	100.30	PASS
Pentachloroethane	10	8.14	7.79	8.75	8.38	9.01	9.69	8.4	7.24	85.94	PASS
1,2,4-Trimethylbenzene	10	8.41	7.86	8.99	9.15	9.41	9.91	8.87	7.44	89.43	PASS
sec-Butylbenzene	10	9.11	8.49	9.04	9.56	10.48	10.68	8.95	8.68	94.73	PASS
1,3-Dichlorobenzene	10	8.87	8.37	9.29	9.22	9.59	10.3	9.14	6.48	92.54	PASS
p-Isopropyltoluene	10	8.8	8.03	8.8	9.09	9.86	10.32	8.86	8.32	91.09	PASS
Benzyl chloride	10	16.15	14.44	15.7	14.34	14.68	14.21	14.48	5.07	148.57	PASS
1,4-Dichlorobenzene	10	8.71	8.06	9.1	9.02	9.43	9.97	9.1	6.52	90.56	PASS
1,2-Dichlorobenzene	10	9.05	8.6	9.31	9.19	10.01	10.6	9.49	6.96	94.64	PASS
n-Butylbenzene	10	9.22	8.39	9.3	9.63	10.15	10.54	8.89	7.77	94.46	PASS
Hexachloroethane	10	8.28	7.84	8.43	8.77	9.17	9.94	8.82	7.73	87.50	PASS
1,2-Dibromo-3-chloropropane	10	6.58	6.26	6.69	6.6	6.79	7.16	7.07	4.57	67.36	PASS
1,2,4-Trichlorobenzene	10	9.6	8.97	9.94	10.04	10.45	10.69	9.6	5.82	98.99	PASS
Hexachlorobutadiene	10	9.44	8.44	9.37	10.45	10.77	10.86	9.84	8.85	98.81	PASS
Naphthalene	10	8.6	7.88	8.66	8.74	8.57	9.4	8.4	5.25	86.07	PASS
1,2,4-Trichlorobenzene	10	8.79	8.33	9.39	9.29	9.83	10.31	9.21	6.97	93.07	PASS
Average									4.67	102.59	

Detection Limits

In EPA Method 524.3, the standard method detection limit (MDL) calculation based on Student's t value for the 99% confidence level with n-1 degrees of freedom (Section 9.2 of the method) is now optional. For the Secondary Laboratory Demonstration, the Agency asked participating laboratories to perform a Lowest Concentration Minimum Reporting Level (LCMRL) experiment. The LCMRL is described as the true concentration for which future recovery is predicted to fall with high confidence (99%) between 50 and 150% recovery[2]. This gives the user more accurate information on the reporting limit and true detection limit for analytes in the method.

The procedure involves taking multiple concentration replicate data and processing it through a downloadable LCMRL calculator as measured concentrations versus the true concentrations[3]. The LCMRL calculator was developed through a collaboration of Shaw Environmental and Infrastructure, Inc. and EPA contract statisticians.

For the experiments on the 240-MS, a special calibration curve was prepared at concentrations of 10, 50, 100, 500, 1000, 2000, 5000, 10,000 and 20,000 ng/L (ppt). Units of ng/L or ppt were required for data entry into the LCMRL calculator. After the calibration curves were completed, four replicates at seven different concentration levels near the low-end of the calibration curves were analyzed. The levels were 10, 25, 50, 100, 200, 500 and 700 ng/L. The calculated results of these replicate injections along with the true or target values were entered into the LCMRL calculator. Figure 4 is an example of the output information generated by the calculator.

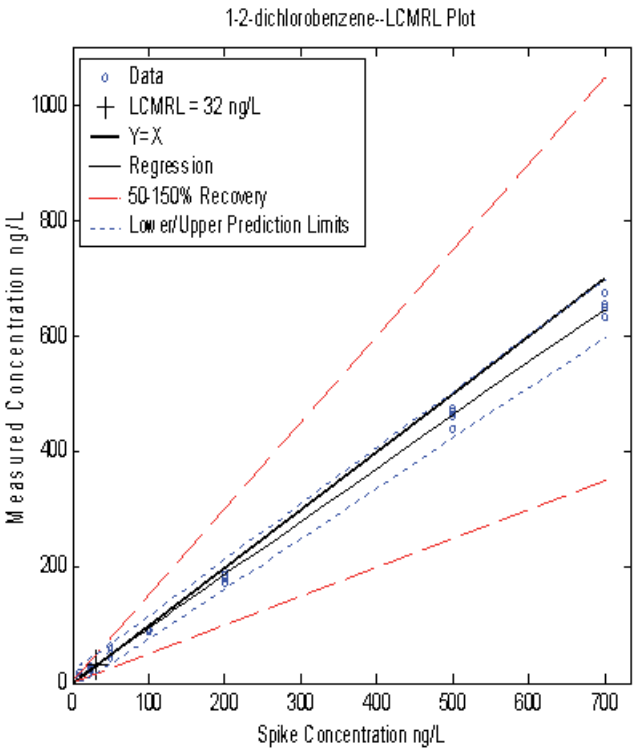

Figure 4a. Calibration curve regression graph for EDB obtained as output from the LCMRL calculator.

The information from the calculator provides improved statistical significance of the reporting limits. Table 5 lists the LCMRLs and detection limits (DLs) for the regulated compounds (and some new target analytes as requested by the EPA study).

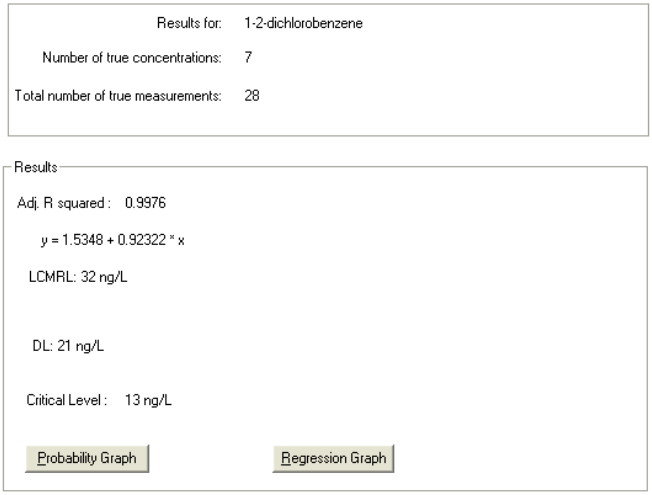

Figure 4b. Results for EDB. LCMRL is calculated to be 22 ng/L, with a predicted detection limit (DL) of 10 ng/L.

Table 5. LCMRLs and DLs from the EPA calculator based on test data from the 240-MS.

Compound name	LCMRL (ng/L)
Chlorodifluoromethane**	NC
Vinyl chloride	627
1,3-Butadiene**	NC
1,1-Dichloroethene	38
Methylene chloride	NC
trans-1,2-Dichloroethene	80
cis-1,2-Dichloroethene	37
Chloroform	27
1,1,1-Trichloroethane	13
Carbon tetrachloride	12
Benzene	51
1,2-Dichloroethane	76
Trichloroethene	39
1,2-Dichloropropane	68
Bromodichloromethane	81
Toluene	14
1,1,2-trichloroethane	46
Tetrachloroethene	101
Dibromochloromethane	117
1,2-Dibromoethane	20
Chlorobenzene	11
Ethylbenzene	22
m.p-Xylene	27
o-Xylene	10
Styrene	19
Bromoform	100
1,4-Dichlorobenzene	52
1,2-Dichlorobenzene	56
1,2-Dibromo-3-chloropropane	37
1,2,4-Trichlorobenzene	36

*NC = not calculated **New analyte added to EPA Method 524.3.

Three of the compounds register as not calculated (NC) for the LCMRL result: chlorodifluoromethane, 1,3-butadiene and methylene chloride. The LCMRL calculator requires the proper selection of calibration levels and replicates in order to obtain valid results. Therefore, it is often necessary to repeat the analysis for some compounds, since analytes will have different responses in the GC/MS.

Conclusions

The Varian 240-MS GC/MS with Archon Purge & Trap AutoSampler and Tekmar Stratum Purge & Trap Concentrator provided excellent results for the new EPA 524. draft method. The FactorFour™ VF-624ms column demonstrated excellent peak shape and resolution for the method analytes. All quality control criteria for initial calibration, as well as accuracy and precision, were easily met.

The new method improved procedures for determining the detection and reporting limits for the target compounds. Calculated LCMRLs were the same or lower than data presented in the new draft method.

Data from this work and comments on the method procedures were submitted to the EPA's Second Laboratory Demonstration study. The Varian 240-MS GC/MS system will also be used to gather low-level full scan data on the select SIM target analytes in EPA 524.3.

References

1. Prakash, B., Zaffiro, A.D., Zimmerman, M., Munch, D.J. and Pepich, B.V. (2009) Method 524.3: Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Version 1.0, June 2009. EPA Document # EPA 815-B-09-009. United States Environmental Protection Agency, Washington DC, USA.

2. Anon (2004) Statistical Protocol for the Determination of the Single-Laboratory Lowest Concentration Minimum Reporting Level (LCMRL) and Validation of Laboratory Performance at or Below the Minimum Reporting Level (MRL). EPA Document # 815-R-05-006 United States Environmental Protection Agency, Washington DC, USA.

3. Downloadable LCMRL calculator available at: http://www.epa.gov/ogwdw000/methods/analyticalmethods_ogwdw.html

These data represent typical results.
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