

# US EPA Method 524.3 – Atomx XYZ and Agilent 7890B GC/5977A MS

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## Abstract

US EPA Method 524.3 was used to determine the concentration of volatile organic compounds (VOCs) in drinking water matrices. The Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with an Agilent 7890B Gas Chromatograph (GC)/5977A Mass Spectrometer (MS) was used to create a working linear regression ( $r^2$ ) calibration curve, method detection limits (MDLs), and minimum reporting level (MRL) confirmation for target compounds.

## Introduction

The Atomx XYZ is Teledyne Tekmar's most advanced P&T system and is based on the time-tested Atomx instrument platform. The concentrator's efficient trap cooling design reduces sample cycle time by as much as 14% over the previous model. Combined with its 84-position soil and water autosampler, the result is more samples tested per 12-hour period. An innovative moisture control system (MCS) improves water vapor removal by as much as 60%, thereby reducing peak interference and increasing GC column lifespan. In addition to other refinements, the Atomx XYZ incorporates a precision-machined valve manifold block to reduce potential leak sources and ensure the system is both reliable and robust.

## Sample preparation

Calibration working standards in concentrations of 10, 25, and 50 ppm were prepared in methanol from the following Restek® standards: 524.3 VOA Mega Mix and 524.3 Gas Calibration Mix. In total, the standards contained 75 compounds.

A nine-point linear regression ( $r^2$ ) calibration curve was prepared from 0.2 ppb to 50 ppb for all compounds with regression value ( $r^2$ )  $\geq 0.995$ . The 10 ppm calibration working standard was diluted to create 0.2, 0.5, and 1 ppb concentrations. The 25 ppm calibration working standard was diluted to create 2 and 5 ppb concentrations. The 50 ppm calibration working standard was diluted to create 10, 20, 30, and 50 ppb concentrations. The relative response factor (RF) was calculated for each compound using three internal standards: 1,4-Difluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Surrogate standards consisted of: Methyl-*tert*-Butyl Ether-d3, 4-Bromofluorobenzene and

1,2-Dichlorobenzene-d4. Internal and surrogate standards were prepared in methanol from Restek standards at a concentration of 12.5 ppm, after which 5  $\mu$ L was then mixed with each 5 mL sample for a resulting concentration of 12.5 ppb.

Seven 1 ppb standards were prepared to calculate the MDL and accuracy and precision calculations. Seven 10 ppb standards were prepared for the MRL confirmation. All calibration, MDL, accuracy and precision, and MRL standards were analyzed with the Atomx XYZ conditions in Table 1. GC-MS conditions are shown in Table 2.

## Experimental Instrument Conditions

**Table 1.** Teledyne Tekmar Atomx XYZ Water Method Conditions.

Purge	Variable	Desorb	Variable
Valve Oven Temp	140 °C	Methanol Needle Rinse	Off
Transfer Line Temp	140 °C	Methanol Needle Rinse Volume	0.00 mL
Sample Mount Temp	90 °C	Water Needle Rinse Volume	7.00 mL
Water Heater Temp	90 °C	Sweep Needle Time	0.25 min
Sample Vial Temp	20 °C	Desorb Preheat Temp	245 °C
Soil Valve Temp	100 °C	GC Start Signal	Begin Desorb
Standby Flow	10 mL/min	Desorb Time	2.00 min
Purge Ready Temp	40 °C	Drain Flow	300 mL/min
		Desorb Temp	250 °C
Purge	Variable	Bake	Variable
Sample Equilibrate Time	0.00 min	Methanol Glass Rinse	Off
Pre-sweep Time	0.25 min	Number of Methanol Glass Rinses	0
Prime Sample Fill Volume	3.00 mL	Methanol Glass Rinse Volume	0.00 mL
Sample Volume	5.00 mL	Water Bake Rinses	1
Sweep Sample Time	0.25 min	Water Bake Rinse Volume	7.00 mL
Sweep Sample Flow	100 mL/min	Bake Rinse Sweep Time	0.25 min
Spurge Vessel Heater	Off	Bake Rinse Sweep Flow	100 mL/min
Spurge Vessel Temp	40 °C	Bake Rinse Drain Time	0.40 min
Pre-purge Time	0.00 min	Bake Time	2.00 min
Pre-purge Flow	0 mL/min	Bake Flow	200 mL/min
Purge Time	5.00 min	Bake Temp	280 °C
Purge Flow	80 mL/min	Condensate Bake Temp	180 °C
Purge Temp	20 °C		
Condensate Purge Temp	20 °C		
Dry Purge Time	0.00 min	Trap	9
Dry Purge Flow	0 mL/min	Chiller Tray	On
Dry Purge Temp	20 °C	Purge Gas	Helium

**Table 2.** Agilent 7890B GC and 5977A MSD System Conditions.

Agilent 7890B GC Conditions	
Column	Rtx®-VMS, 20m x 0.18 mm, 1 $\mu$ m Film, Helium – 1 mL/min
Oven Profile	35 °C, 2 min, 16 °C/min to 85 °C, 30 °C/min to 225 °C, 1 min hold, Run Time 10.79 min
Inlet	180 °C, 120:1 Split, 19.752 psi
Agilent 5977A MSD Conditions	
Temp	Transfer Line 225 °C; Source 230 °C; Quad 150 °C
Scan	Range 35 m/z to 260 m/z, Solvent Delay 0.50 min, Normal Scanning
Gain	Gain Factor 10.00, Autotune

## Results

The linear correlation coefficient of the calibration curve ( $r^2$ ), MDL, accuracy and precision, and MRL confirmation data are shown in Table 3. Figure 1 displays a 10 ppb standard, indicating excellent peak resolution with minimal water interference for all VOCs.

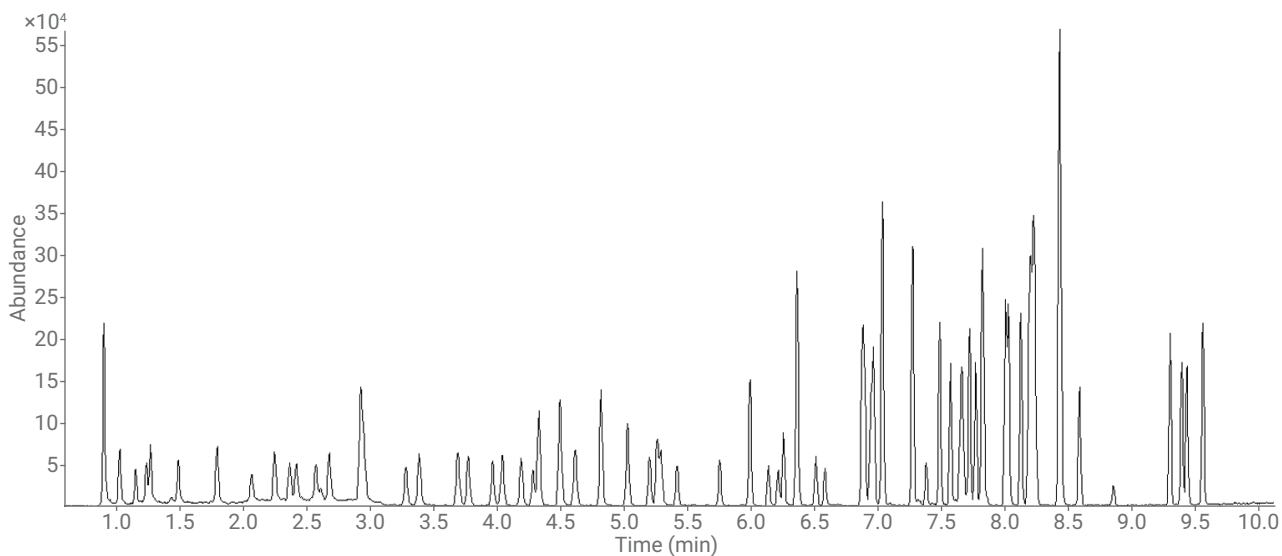
**Table 3.** Method 524.3 Calibration, Accuracy and Precision Data.

Compound	Calibration		Accuracy and Precision (n = 7, 1 ppb) <sup>1</sup>			MRL Confirmation (n = 7, 10 ppb) <sup>2</sup>	
	Linearity ( $r^2 \geq 0.995$ )	MDL (ppb) <sup>1</sup>	Avg. Conc. (ppb)	Accuracy ( $\pm 20\%$ )	Precision ( $\leq 20\%$ )	LPIR ( $\geq 50\%$ )	UPIR ( $\leq 150\%$ )
1,4-Difluorobenzene (IS)							
Dichlorodifluoromethane	0.998	0.28	0.98	98	9.2	61	112
Chlorodifluoromethane	0.995	0.27	0.92	92	9.5	58	110
Chloromethane	0.997	0.13	0.96	96	4.4	64	102
Vinyl chloride	0.997	0.57	0.91	91	20.0	53	122
1,3-Butadiene	0.999	0.36	1.00	100	11.5	64	108
Bromomethane	0.996	0.24	1.13	113	6.6	82	122
Trichlorofluoromethane	0.996	0.27	1.11	111	7.8	88	123
Diethyl ether	0.996	0.17	1.00	100	5.5	69	133
1,1-Dichloroethene	0.996	0.44	0.99	99	14.0	76	124
Carbon Disulfide	0.998	0.26	1.00	100	8.2	89	116
Methyl iodide	0.996	0.27	0.90	90	9.7	91	120
Allyl chloride	0.998	0.27	0.97	97	8.9	74	124
Methyl acetate	0.999	0.20	1.06	106	6.0	90	114
Methylene chloride	0.999	0.27	1.00	100	8.7	69	124
<i>trans</i> -1,2-Dichloroethene	0.999	0.33	0.94	94	11.2	61	134
Methyl- <i>tert</i> -butyl ether-d3 (SURR)			12.7	102	10.1		
Methyl- <i>tert</i> -butyl ether	0.997	0.43	1.04	104	13.2	60	150
<i>tert</i> -Butyl alcohol	0.997	0.38	1.10	110	11.2	76	125
1,1-Dichloroethane	0.996	0.29	0.89	89	10.4	78	118
Diisopropyl ether	0.998	0.38	0.95	95	12.8	70	114
<i>tert</i> -Butyl ethyl ether	0.998	0.26	0.96	96	8.5	86	114
<i>cis</i> -1,2-Dichloroethene	0.997	0.27	0.90	90	9.6	71	116
Bromochloromethane	0.995	0.32	0.87	87	11.6	67	131
Chloroform	0.996	0.28	1.00	100	8.9	83	118
Carbon Tetrachloride	0.999	0.52	0.95	95	17.3	85	115
Tetrahydrofuran	0.998	0.24	1.00	100	7.6	91	116
1,1,1-Trichloroethane	0.999	0.44	0.97	97	14.3	87	112
1,1-Dichloropropene	0.999	0.51	1.02	102	16.0	77	122
1-Chlorobutane	0.999	0.34	0.90	90	11.9	71	114
Benzene	0.998	0.35	0.97	97	11.3	78	121
<i>tert</i> -Amyl methyl ether	0.999	0.33	0.89	89	11.9	76	113
1,2-Dichloroethane	0.999	0.23	0.90	90	8.0	71	116
Trichloroethene	0.998	0.29	1.10	110	8.3	91	112
<i>tert</i> -Amyl ethyl ether	0.999	0.33	0.99	99	10.6	79	117

Compound	Calibration		Accuracy and Precision (n = 7, 1 ppb) <sup>1</sup>			MRL Confirmation (n = 7, 10 ppb) <sup>2</sup>	
	Linearity (r <sup>2</sup> ≥0.995)	MDL (ppb) <sup>1</sup>	Avg. Conc. (ppb)	Accuracy (±20%)	Precision (≤20%)	LPIR (≥50%)	UPIR (≤150%)
Dibromomethane	0.999	0.49	1.07	107	14.7	98	114
1,2-Dichloropropane	0.996	0.47	1.02	102	14.7	70	110
Bromodichloromethane	0.998	0.20	0.87	87	7.4	73	117
<i>cis</i> -1,3-Dichloropropene	0.998	0.46	0.92	92	16.1	89	112
Chlorobenzene-d5 (IS)							
Toluene	0.999	0.56	0.95	95	18.6	78	124
Tetrachloroethene	0.996	0.37	1.01	101	11.6	73	128
<i>trans</i> -1,3-Dichloropropene	0.997	0.41	0.90	90	14.5	93	112
Ethyl methacrylate	0.996	0.44	0.97	97	14.4	83	118
1,1,2-Trichloroethane	0.999	0.39	0.95	95	13.1	62	119
Dibromochloromethane	0.995	0.25	1.06	106	7.6	70	131
1,3-Dichloropropane	0.999	0.39	0.97	97	12.7	78	128
1,2-Dibromoethane	0.999	0.22	1.00	100	7.0	81	120
Chlorobenzene	0.999	0.29	1.00	100	9.3	82	125
Ethylbenzene	0.999	0.43	0.94	94	14.8	76	121
1,1,1,2-Tetrachloroethane	0.999	0.51	1.00	100	16.1	83	126
<i>m,p</i> -Xylene	0.999	0.73	1.78	89	13.0	88	120
<i>o</i> -Xylene	0.998	0.48	1.03	103	15.0	73	132
Styrene	0.998	0.22	0.88	88	7.9	80	120
Bromoform	1.000	0.38	0.83	83	14.5	87	119
Isopropylbenzene	0.999	0.38	0.92	92	13.2	78	125
1,4-Dichlorobenzene-d4 (IS)							
4-Bromofluorobenzene (SURR)			12.7	102	3.5		
Bromobenzene	0.998	0.34	0.94	94	11.6	72	119
<i>n</i> -Propylbenzene	0.996	0.20	0.96	96	6.5	77	120
1,1,1,2-Tetrachloroethane	0.998	0.37	0.91	91	12.9	72	124
2-Chlorotoluene	0.996	0.28	1.02	102	8.6	62	131
1,3,5-Trimethylbenzene	0.997	0.28	0.92	92	9.6	75	121
1,2,3-Trichloropropane	0.995	0.31	1.17	117	8.4	93	116
4-Chlorotoluene	0.996	0.28	0.93	93	9.7	76	121
<i>tert</i> -Butylbenzene	0.995	0.23	0.89	89	8.1	72	124
Pentachloroethane	0.996	0.36	0.84	84	13.9	54	118
1,2,4-Trimethylbenzene	0.998	0.23	0.94	94	7.9	73	131
<i>sec</i> -Butylbenzene	0.998	0.41	0.89	89	14.6	67	131
4-Isopropyltoluene	0.997	0.35	0.95	95	11.7	76	126
1,3-Dichlorobenzene	0.998	0.42	1.06	106	12.5	73	126
1,4-Dichlorobenzene	0.997	0.29	1.04	104	8.9	77	127
<i>n</i> -Butylbenzene	0.997	0.29	0.98	98	9.3	81	123
Hexachloroethane	0.996	0.59	0.95	95	19.9	65	111
1,2-Dichlorobenzene-d4 (SURR)			12.3	98	2.1		
1,2-Dichlorobenzene	0.998	0.37	1.05	105	11.3	70	124
1,2-Dibromo-3-chloropropane	0.999	0.42	0.83	83	15.9	76	131
Hexachlorobutadiene	0.996	0.43	0.99	99	14.0	67	134
1,2,4-Trichlorobenzene	0.999	0.60	0.97	97	19.6	63	129
Napthalene	0.997	0.22	0.92	92	7.6	67	129
1,2,3-Trichlorobenzene	0.997	0.21	0.98	98	6.7	66	133

<sup>1</sup> Data from seven 1 ppb samples.

<sup>2</sup> Data from seven 10 ppb samples.



**Figure 1.** Total Ion Chromatogram of a 10 ppb VOC Standard Indicating Consistent Peak Shapes for all Compounds with No Water Interference.

## Conclusion

This study demonstrates the capability of the Teledyne Tekmar Atomx XYZ P&T system to process VOCs in drinking water samples following US EPA Method 524.3 with detection by an Agilent 7890B GC/5977A MS. The linearity of the calibration curve from 0.2 ppb to 50 ppb passed all method requirements with no interference from excessive water. The MDL, accuracy and precision for seven 1 ppb standards and MRL confirmation for seven 10 ppb standards, also indicated no interference from excessive water.

Furthermore, the Atomx XYZ and GC-MS conditions displayed in Table 1 and Table 2 allow for up to four samples to run within one hour. By making additional, appropriate changes to the GC oven temperature program, the GC/MS cycle time may also be reduced, increasing laboratory throughput in a 12-hour period.

## Reference

1. Munch, J. W. Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry; *US EPA Method 524.3* - Revision 1.0, June **2009**.

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