APPLICATION NOTE: AN1802
TECHNOLOGY: P+T VOC
INDUSTRY: ENV

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

*Amy Nutter, Applications Chemist; Teledyne Tekmar*



## 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 ($r^2$) ≥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 µL 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 I. GC-MS conditions are shown in Table II.



**Sales/Support**: 800-874-2004 • **Main**: 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
**www.teledynetekmar.com**

## Experimental Instrument Conditions

| Table I Teledyne Tekmar Atomx XYZ Water Method Conditions | | | |
|---|---|---|---|
| **Purge** | **Variable** | **Desorb** | **Variable** |
| Valve Oven Temp | 140 ºC | Methanol Needle Rinse | Off |
| Transfer Line Temp | 140 ºC | Methanol Needle Rinse Volume | 0.00 mL |
| Sample Mount Temp | 90 ºC | Water Needle Rinse Volume | 7.00 mL |
| Water Heater Temp | 90 ºC | Sweep Needle Time | 0.25 min |
| Sample Vial Temp | 20 ºC | Desorb Preheat Temp | 245 ºC |
| Soil Valve Temp | 100 ºC | GC Start Signal | Begin Desorb |
| Standby Flow | 10 mL/min | Desorb Time | 2.00 min |
| Purge Ready Temp | 40 ºC | Drain Flow | 300 mL/min |
| | | Desorb Temp | 250 ºC |
| **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 |
| Sparge Vessel Heater | Off | Bake Rinse Sweep Flow | 100 mL/min |
| Sparge Vessel Temp | 40 ºC | 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 ºC |
| Purge Flow | 80 mL/min | Condensate Bake Temp | 180 ºC |
| Purge Temp | 20 ºC | | |
| Condensate Purge Temp | 20 ºC | | |
| Dry Purge Time | 0.00 min | **Trap** | 9 |
| Dry Purge Flow | 0 mL/min | **Chiller Tray** | On |
| Dry Purge Temp | 20 ºC | **Purge Gas** | Helium |



**Sales/Support:** 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
**www.teledynetekmar.com**

| Table II Agilent 7890B GC and 5977A MSD System Conditions | |
|---|---|
| **Agilent 7890B GC Conditions** | |
| Column | Rtx®-VMS, 20m x 0.18 mm, 1µm Film, Helium – 1 mL/min |
| Oven Profile | 35 ºC, 2 min, 16 ºC/min to 85 ºC, 30 ºC/min to 22 5 ºC, 1 min hold, Run Time 10.79 min |
| Inlet | 180 ºC, 120:1 Split, 19.752 psi |
| **Agilent 5977A MSD Conditions** | |
| Temp | Transfer Line 225 ºC; Source 230 ºC; Quad 150 ºC |
| 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 III. Figure 1 displays a 10 ppb standard, indicating excellent peak resolution with minimal water interference for all VOCs.

**Table III Method 524.3 Calibration, Accuracy and Precision Data**

| Compound | Calibration | | Accuracy and Precision (n=7, 1 ppb)[1] | | | MRL Confirmation (n=7, 10 ppb)[2] | |
|---|---|---|---|---|---|---|---|
| | Linearity ($r^2$ ≥0.995) | MDL (ppb)[1] | Avg. Conc. (ppb) | Accuracy (±20%) | Precision (≤20%) | LPIR (≥50%) | UPIR (≤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 |

| Table III Method 524.3 Calibration, Accuracy and Precision Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Compound | Calibration | | Accuracy and Precision (n=7, 1 ppb)[1] | | | MRL Confirmation (n=7, 10 ppb)[2] | |
| | Linearity ($r^2$ ≥0.995) | MDL (ppb)[1] | Avg. Conc. (ppb) | Accuracy (±20%) | Precision (≤20%) | LPIR (≥50%) | UPIR (≤150%) |
| 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 |
| Trans-1,2-Dichloroethene | 0.999 | 0.33 | 0.94 | 94 | 11.2 | 61 | 134 |
| Methyl-tert-Butyl Ether-d3 (SURR) | | | 12.7 | 102 | 10.1 | | |
| Methyl-tert-Butyl Ether | 0.997 | 0.43 | 1.04 | 104 | 13.2 | 60 | 150 |
| tert-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 |
| tert-Butyl Ethyl Ether | 0.998 | 0.26 | 0.96 | 96 | 8.5 | 86 | 114 |
| cis-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-Trichloroehane | 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 |
| tert-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 |
| tert-Amyl Ethyl Ether | 0.999 | 0.33 | 0.99 | 99 | 10.6 | 79 | 117 |
| 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 |
| cis-1,3-Dichloropropene | 0.998 | 0.46 | 0.92 | 92 | 16.1 | 89 | 112 |

| Table III Method 524.3 Calibration, Accuracy and Precision Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Compound | Calibration | | Accuracy and Precision (n=7, 1 ppb)[1] | | | MRL Confirmation (n=7, 10 ppb)[2] | |
| | Linearity ($r^2$ ≥0.995) | MDL (ppb)[1] | Avg. Conc. (ppb) | Accuracy (±20%) | Precision (≤20%) | LPIR (≥50%) | UPIR (≤150%) |
| 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 |
| trans-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 |
| m,p-Xylene | 0.999 | 0.73 | 1.78 | 89 | 13.0 | 88 | 120 |
| o-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 |
| n-Propylbenzene | 0.996 | 0.20 | 0.96 | 96 | 6.5 | 77 | 120 |
| 1,1,2,2-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 |

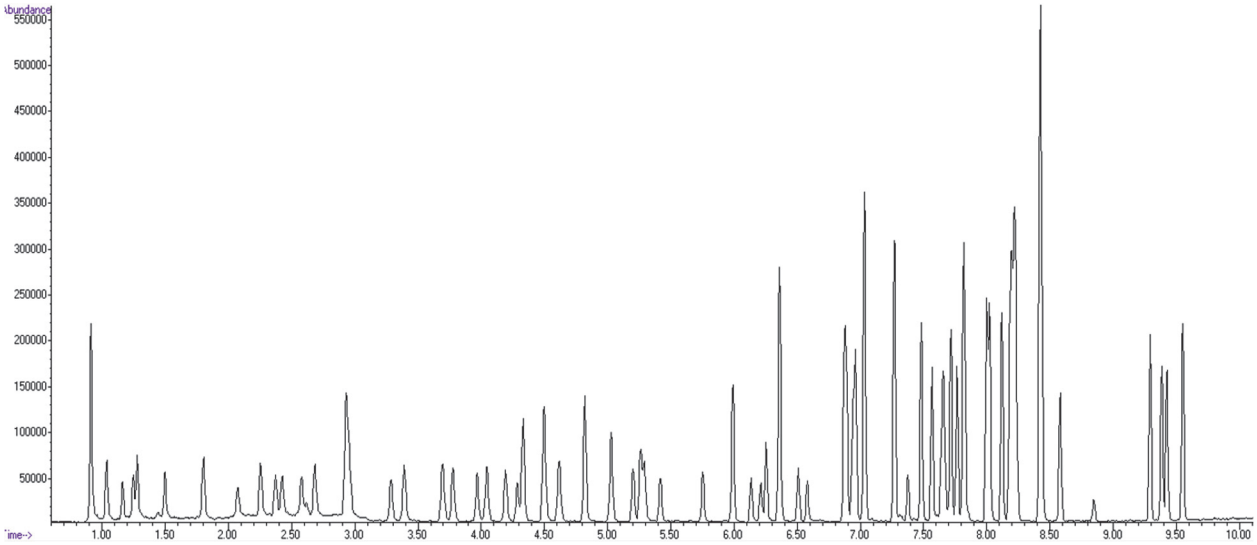
| Table III Method 524.3 Calibration, Accuracy and Precision Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Compound | Calibration | | Accuracy and Precision (n=7, 1 ppb)[1] | | | MRL Confirmation (n=7, 10 ppb)[2] | |
| | Linearity ($r^2$ ≥0.995) | MDL (ppb)[1] | Avg. Conc. (ppb) | Accuracy (±20%) | Precision (≤20%) | LPIR (≥50%) | UPIR (≤150%) |
| tert-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 |
| sec-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 |
| n-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 |

1. Data from seven 1 ppb samples.
2. Data from seven 10 ppb samples.



**Sales/Support:** 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
**www.teledynetekmar.com**

**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 I and Table II 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.

## References

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.



**Sales/Support:** 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
**www.teledynetekmar.com**