APPLICATION NOTE: AN1903
TECHNOLOGY: P+T VOC
INDUSTRY: ENV

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Analysis of 1,4-Dioxane in Drinking Water Using the Teledyne Tekmar Lumin P & T Concentrator, AQUATek LVA and Agilent 7890B GC/5977A MSD

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Abstract

Drinking water analysis of Volatile Organic Compounds (VOCs) is normally performed by purge and trap (P&T) concentration, using standard US EPA methods. This application manipulates P&T and Gas Chromatograph/Mass Spectrometer (GC/MS) parameters to create an efficient method in order to detect 1,4-Dioxane at the part-per-billion level (ppb), despite its poor purge efficiency. This study uses a Teledyne Tekmar Lumin P&T concentrator with an AQUATek LVA liquid autosampler in conjunction with an Agilent 7890B Gas Chromatograph (GC)/5977A Mass Spectrometer (MS) in selected ion monitoring (SIM) mode to create a working linear calibration curve and method detection limits (MDLs) for 1,4-Dioxane.

Introduction

The AQUATek LVA is Teledyne Tekmar's most advanced water-only P&T autosampler and is based on the proven Atomx XYZ platform. The AQUATek LVA includes whisper-quiet XYZ automation, two standard addition vessels and an optional pH meter. Combined with its 84-position chiller-enabled sample tray, the result is simple and reliable sample preparation and handling. In addition to other refinements, the AQUATek LVA incorporates a precision-machined valve manifold block to reduce potential leak sources and ensure the system is both reliable and robust. By pairing the AQUATek LVA with the Lumin's innovative moisture control system (MCS), water vapor removed is improved by as much as 60%, thereby reducing peak interference and increasing GC column lifespan.

Sample Preparation

A 25 ppm calibration working standard was prepared in methanol from a Restek® 1,4-Dioxane standard. The calibration curve was prepared from 0.2 ppb to 50 ppb. The relative response factor (RF) was calculated using a 1,4-Dioxane-d8 internal standard. The internal standard was prepared in methanol from a Restek standard at a concentration of 25 ppm, after which 5 µL was then mixed with each 5 mL sample for a resulting concentration of 25 ppb.

Seven 0.5 ppb standards were prepared to calculate MDL and accuracy and precision calculations. All calibration and MDL standards were analyzed with the Lumin P&T concentrator and AQUATek LVA conditions in Table I. GC/MS conditions are shown in Table II.

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Experimental Instrument Conditions

Table I Teledyne Tekmar Lumin/AQUATek LVA Water Method Conditions			
Standby	Variable	Desorb	Variable
Valve Oven Temp	140 °C	Desorb Preheat Temp	245 °C
Transfer Line Temp	140 °C	Desorb Temp	250 °C
Sample Mount Temp	90 °C	Desorb Time	1.00 min
Standby Flow	10 mL/min	Drain Flow	300 mL/min
Purge Ready Temp	40 °C	GC Start Signal	Begin Desorb
MCS Purge Temp	20 °C		
Purge	Variable	Bake	Variable
Purge Temp	20 °C	Bake Time	2.00 min
Purge Time	5.00 min	Bake Temp	280 °C
Purge Flow	100 mL/min	Bake Flow	400 mL/min
Dry Purge Temp	40 °C	MCS Bake Temp	175 °C
Dry Purge Time	1.00 min		
Dry Purge Flow	200 mL/min	AQUATek LVA	Variable
Pre-Purge Time	0.50 min	Sample Loop Time	0.35 min
Pre-Purge Flow	40 mL/min	Sample Transfer Time	0.35 min
Preheat Time	1.00 min	Rinse Loop Time	0.30 min
Sample Heater Enable	On	Sweep Needle Time	0.30 min
Sample Temp	60 °C	Presweep Time	0.25 min
Trap	K	Water Temp	90 °C
Chiller Tray	Off	Bake Rinse Cycles	1
Purge Gas	Helium	Bake Rinse Drain Time	0.35 min

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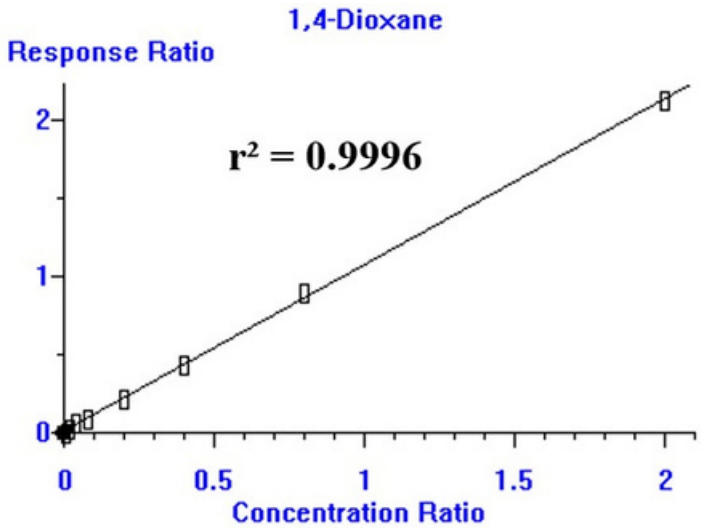
Table II Agilent 7890B GC and 5977A MSD System Conditions	
Agilent 7890B GC Conditions	
Column	Rtx®-VMS, 20 m x 0.18 mm, 1µm Film, Helium – 0.8 mL/min
Oven Profile	35 °C, 4 min, 15 °C/min to 85 °C, 30 °C/min to 225 °C, 2 min Hold, Run Time 14 min
Inlet	180 °C, 80:1 Split
Agilent 5977A MSD Conditions	
Temp	Transfer Line 225 °C; Source 230 °C; Quad 150 °C
Sim Ions/Dwell Time	1,4-Dioxane: 88, 1,4-Dioxane-d8: 96. Dwell Time: 150 msec per ion
Gain	Gain Factor 10.00, Autotune

Calibration/Results

The linear calibration curve for 1,4-Dioxane is displayed in Figure 1. 1,4-Dioxane had a 0.9996 coefficient of determination (r^2) over the range of 0.2 to 50 ppb.

Figure 1 Calibration Curve 0.2-50 ppb for 1,4-Dioxane.



To verify that the 0.5 ppb calibration point is precise and accurate, a reproducibility test was performed. Seven duplicate samples were prepared at 0.5 ppb. An example of a SIM scan for 1,4-Dioxane at 0.5 ppb is displayed in Figure 2. These seven duplicate samples were analyzed to establish a minimum detection limit (MDL) for 1,4-Dioxane. The reproducibility and MDL results can be found in Table III.

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Figure 2 Selective Ion Monitoring (SIM) Scan of 0.5 ppb 1,4-Dioxane Standard (Retention Time 6.92 min).

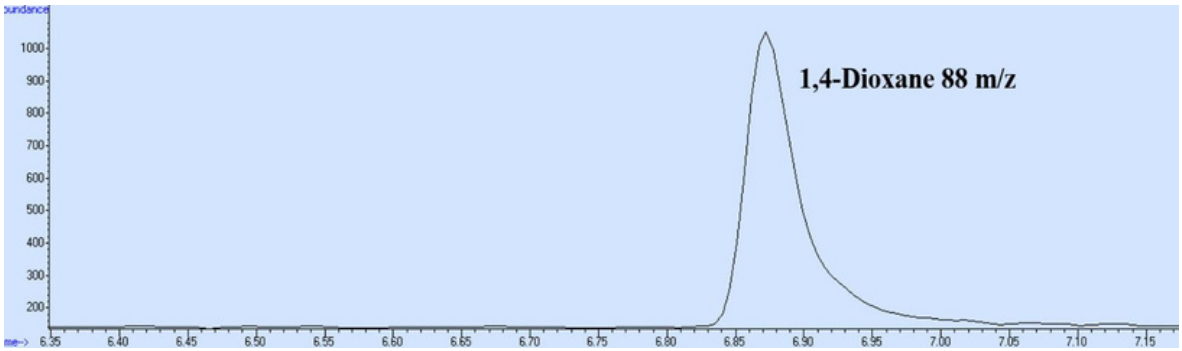

Table III Reproducibility and MDL for 1,4-Dioxane		
Sample Rep	Area	Concentration (ppb)
1	599	0.54
2	598	0.56
3	561	0.55
4	587	0.54
5	558	0.56
6	524	0.52
7	564	0.55
Average	0.55	
%RSD	2.56	
MDL	0.04	

Conclusion

Using the GC/MS and purge and trap parameters defined in this application note, 1,4-Dioxane can be detected as low as 0.2 ppb. As the detection limits of 1,4-Dioxane continue to get lower, it is important to modify the method parameters established in this application, to satisfy changing requirements. This study demonstrates the capability of the Teledyne Tekmar Lumin P&T concentrator and AQUATek LVA to process 1,4-Dioxane in water samples with detection by an Agilent 7890B GC/5977A MS. The %RSD of the calibration curve passed all method requirements. Furthermore, MDL and accuracy and precision for seven 0.5 ppb standards showed no interference from excessive water.

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, without compromising sensitivity.

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