TELEDYNE TEKMAR
A Teledyne Technologies Company

Reducing Cycle Time for Analysis of 1,4-Dioxane using an Atomx Automated Sample Prep System

Application Note

Abstract

With advances in instrumentation and without regulatory method constraints, 1,4-dioxane can be detected at the part-per-billion (ppb) level, despite its poor purge efficiency. This application will manipulate purge and trap, as well as GC/MS parameters to create a more efficient method to reduce cycle times. This study will utilize a Teledyne Tekmar Atomx Automated Sample Prep System in conjunction with an Agilent 7890/5975 GC/MS. A linear calibration and method detection limits (MDLs) for 1,4-dioxane will be established using this method.

Introduction

1,4-Dioxane is commonly analyzed in water using Purge and Trap concentration to prepare the sample for evaluation by GC/MS. Due to the poor purge efficiency of the analyte, normal purge and trap methods must be modified to detect 1,4-dioxane at the part-per-billion level. This method also utilizes an Agilent 7890A/5975C with Triple Axis Detector in Selected Ion Monitoring (SIM) mode to provide the increased sensitivity required by this low-level analysis.

For this study, an Atomx Automated Sample Prep System was used in conjunction with a GC/MS. This "all-in-one" autosampler allows for complete automation of sample preparation for the analysis of soil and liquid samples for purge and trap. Through the features the Atomx provides, such as the 80-position sample tray and standard addition vessels, efficiency and throughput can be greatly increased, leading to cost and time savings.

Utilizing an Agilent 7890/5975 GC/MS, a linear calibration was performed and percent Relative Standard Deviation (%RSD) and MDLs were determined for 1,4-dioxane, using a 5mL purge volume.

Experimental-Instrument Conditions

The Atomx Automated Sample Prep System was coupled to an Agilent 7890A/5975 GC/MS with Triple Axis Detector for analysis. Teledyne Tekmar's proprietary #9 trap was the analytical trap utilized. The GC was configured with an Agilent DB-624 20m x 0.18mm x 1.0µm column. The GC/MS parameters are outlined in Tables 1 and 2. Table 3 outlines the Atomx conditions.

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GC Parameters		MSD Parameters	
GC:	Agilent 7890A	MSD:	Agilent 5975C with Triple Axis Detector
Column:	Agilent DB-624 20m x 0.18mm x 1.0µm	Source:	230°C
Oven Program:	35°C for 3 min; 12°C/min to 100°C for 0 min; 50°C/min to 240°C for 0.78 min; 12 min runtime	Quad:	150°C
Inlet:	220°C	Solvent Delay:	0.5 min
Column Flow:	0.9 mL/min	SIM Ions:	88, 58, 96, 64
Split:	30:1	Dwell Time:	100 msec dwell per ion
Pressure:	17.984 psig		

Tables 1 & 2: GC and MSD Parameters

Atomx Water Parameters			
Variable	**Value**	**Variable**	**Value**
Valve oven Temp	140°C	Dry Purge Flow	200mL/min
Transfer Line Temp	140°C	Dry Purge Temp	40°C
Sample Mount Temp	90°C	Methanol Needle Rinse	Off
Water Heater Temp	90°C	Methanol Needle Rinse Volume	3.0mL
Sample Vial Temp	30°C	Water Needle Rinse Volume	7.0mL
Sample Equilibrate Time	0.00 min	Sweep Needle Time	0.25min
Soil Valve Temp	50°C	Desorb Preheat Time	245°C
Standby Flow	10mL/min	GC Start Signal	Start of Desorb
Purge Ready Temp	45°C	Desorb Time	1.00 min
Condensate Ready Temp	40°C	Drain Flow	300mL/min
Presweep Time	0.25 min	Desorb Temp	250°C
Prime Sample Fill Volume	3.0mL	Methanol Glass rinse	Off
Sample Volume	5.0mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	3.0mL
Sweep Sample Flow	100mL/min	Number of Bake Rinses	1
Sparge Vessel Heater	On	Water Bake Rinse Volume	7.0mL
Sparge Vessel Temp	60°C	Bake Rinse Sweep Time	0.25 min
Prepurge Time	0.50 min	Bake Rinse Sweep Flow	100mL/min
Prepurge Flow	40mL/min	Bake Rinse Drain Time	0.40 min
Purge Time	5.00 min	Bake Time	2.00 min
Purge Flow	100mL/min	Bake Flow	400mL/min
Purge Temp	20°C	Bake Temp	280°C
Condensate Purge Temp	20°C	Condensate Bake Temp	200°C
Dry Purge Time	1.00 min		

Table 3: Atomx Parameters (items in yellow were not used)

Calibration Data

A 50ppm working calibration standard was prepared in methanol. Calibration standards were prepared in 50mL volumetric flasks filled to volume with de-ionized water over a range of 1 to 500 ppb. Samples were transferred to headspace free 40mL vials for analysis. The Internal Standard (IS) was prepared in methanol at a 50ppm concentration and added to each sample, bringing the final concentration of 50ppb. Agilent Chemstation software was used to process the calibration data. Relative response factors were

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evaluated for linearity and %RSD, which were 0.9998 and 5.12 respectively. Method detection limits were also established for 1,4-dioxane by analyzing seven replicates at a concentration of 5ppb. The MDL for 1,4-dioxane was 0.74. An Extracted Ion Chromatogram for 10ppb 1,4-dioxane standard can be found in Figure 1.

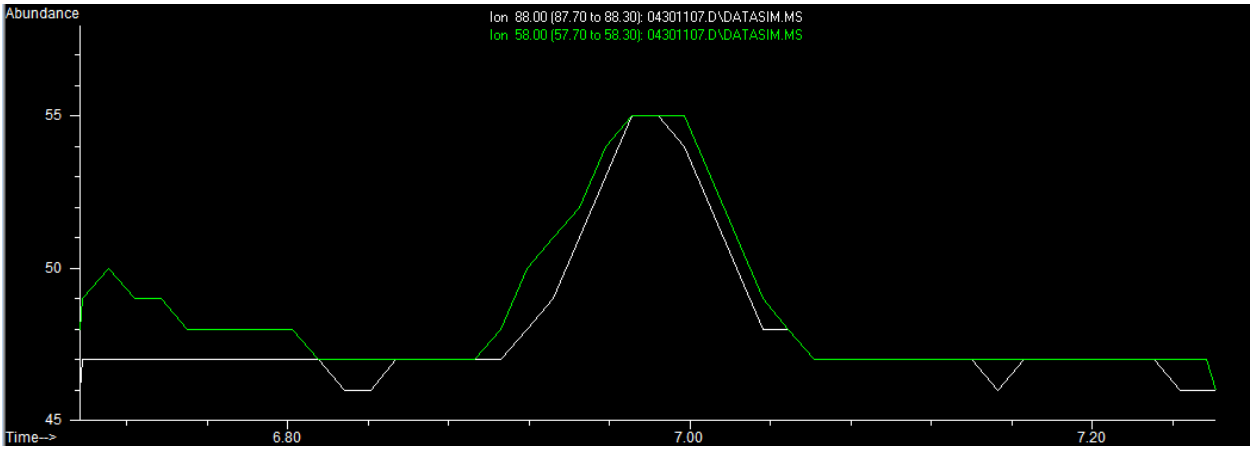

Figure 1: Extracted Ion Chromatogram (EIC) of a 10 ppb 1,4-Dioxane Standard

Conclusions

Throughput is a major factor in lab efficiency, and therefore, profitability. Automation and optimized purge and trap methods can greatly reduce sample prep and overall cycle times. This study demonstrates the capabilities of the Teledyne Tekmar Atomx Automated Sample Prep System coupled with an Agilent 7890/5975 GC/MS, to detect 1,4-dioxane at the part-per-billion level. By optimizing the purge parameters, without compromising sensitivity, throughput can be greatly increased, leading to cost and time savings.

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