Analysis of 1,4-Dioxane Using the Stratum PTC and the SOLATek 72

Application Note

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Abstract

Low level detection of 1,4-dioxane is required in many labs. The detection limits change with regulatory requirements and can vary from state to state and country to country. As instrument sensitivity improves, it is possible to detect compounds at lower and lower levels. This application note investigates the detection of 1,4-dioxane at a 1ppb level.

Introduction

A common technique for the determination 1,4-Dioxane water is Purge and Trap (P&T) concentration in conjunction with Gas Chromatography and Mass Spectrometry (GC/MS). However, due to the poor purge efficiency of 1,4-Dioxane, it is necessary to modify traditional P&T and GC/MS techniques in order to detect 1,4-Dioxane at low levels.

In this study, P&T conditions were optimized in order to achieve a 1ppb level of detection. The GC/MS system used for this analysis was an Agilent 7890A GC and 5975C inert XL MS run Selected Ion Monitoring (SIM) mode. A 25mL purge volume was utilized and the P&T conditions are outlined in this paper. The Stratum PTC was configured with Teledyne Tekmar's proprietary #9 trap in order to aid in moisture control as the samples required heating.

Experimental-Instrument Conditions

A Stratum PTC and SOLATek 72 were configured with the Agilent 7890A GC and the 5975 inert XL MSD. Tables 1 and 2 display the GC and MSD conditions. Several different purge flows, purge times, and sample temperatures were evaluated. The Stratum PTC/SOLATek 72 conditions, outlined in Table 3, provided the optimum compound response.

GC Parameters	
GC:	Agilent 7890A
Column:	J&W Scientific DB-624 20m x 0.180mm x1.0um
Oven Program:	45°C for 4.5 min; 12°C/min.to 100°C for 0 min; 25°C /min to 240 °C for 1.32min; 16.0 min runtime
Inlet:	220°C
Column Flow:	1.5 mL/min
Gas:	Helium
Split:	30:1
Pressure:	28.64psig

Table 1: GC Parameters

MSD Parameters	
MSD:	Agilent 5975C inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Column Flow:	1.5mL/min
SIM Ions:	Primary ions 88 and 76 Secondary ions 58 and 57
Dwell Time:	100 msec dwell per ion

Table 2: MSD Parameters

Stratum PTC and SOLATek 72 Parameters Water Parameters			
Variable	**Value**	**Variable**	**Value**
Rinse Water Temp	90°C	Sample Preheat Time	5.00 min
Sample Cup Temp:	30°C	Sample Temp	60°C
Sample Needle Temp	30°C	Purge Time	5.00
Transfer Line Temp	125°C	Purge Temp	0°C
Soil Valve Temp	125°C	Purge Flow	100mL/min
Sample Sweep Time	0.50 min	Dry Purge Time	1.00 min
Needle Rinse Volume	7mL	Dry Purge Temp	40°C
Needle Sweep Time	0.50 min	Dry Purge Flow	200mL/min
Bake Rinse Volume	25mL	GC Start	Start of Desorb
Bake Sweep Time	0.50 min	Desorb Preheat Temp	245°C
Bake Drain Time	0.50 min	Desorb Drain	On
Number of Bake Rinses	2	Desorb Time	1.00 min
Valve Oven Temp	140°C	Desorb Temp	250°C
Transfer Line Temp	140°C	Desorb Flow	300mL/min
Sample Mount Temp	90°C	Bake Time	2.00 min
Purge ready Temp	45°C	Bake Temp	270°C
Condenser Ready Temp	40°C	Bake Flow	400mL/min
Condenser Purge Temp	20°C	Condenser Bake Temp	175°C
Standby Flow	10mL/min		
Pre-Purge Time	0.50 min		
Pre-Purge Flow	40mL/min		
Sample Heater	On		

Table 3: Stratum PTC and SOLATek 72 Parameters

Stratum PTC Parameters are indicated in blue

Standard Preparation

A 1000ppm 1,4-dioxane working standard was prepared:

STD	Cat#	Name	Batch#	Concentration	Amt. Used	Vol.	Final Conc.
Sigma	34857	1,4-dioxane	12184TD	$\geq$99.5% pure	10µL	10mL	1000ppm

A 50ppm stock standard was prepared from the 1000ppm working standard as follows:

Amt. Used	Vol.	Final Conc.
500µL	10mL	50ppm

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Curve Preparation

The 50ppm stock standard was used to prepare the following calibration points in a 50mL volumetric flask filled to volume with de-ionized water.

Amount Used	Final Volume	Final Concentration
1.0µL	50mL	1.0ppb
5.0µL	50mL	5.0ppb
10.0µL	50mL	10.0ppb
25.0µL	50mL	25.0ppb
50.0µL	50mL	50.0ppb
100.0µL	50mL	100.0ppb

The standards were transferred to headspace free 40mL vials for analysis. A 25mL purge volume was used. A 25ppm Internal Standard (IS) of the MTBE-d3 was prepared in methanol and transferred to the standard vessel on the SOLATek 72. The SOLATek 72 then transferred the IS in 10µL aliquots to the samples in order to hold the IS concentration at a constant 10ppb.

Results

The calibration data was analyzed using Agilent Chemstation software. The relative response factor of 1,4-dioxane was 7.93. A statistical determination of the MDL's was determined for 1,4-dioxane by analyzing seven replicate standards of a 1ppb calibration standard. Finally, seven replicate standards of a 10ppb calibration standard were analyzed in order to determine the precision and accuracy of the experimental conditions. As established in Table 4, using the Stratum PTC and SOLATek 72 and the outlined experimental conditions, 1,4-dioxane may be detected down to the 1ppb level.

Compound	Spike	MDL	Spike Level	Accuracy	Precision
MTBE-d3 (IS)	10.00	N/A	10.00	3.33	N/A
1,4-Dioxane	1.00	0.275	10.00	10.76	102.3

Table 4: Experimental Results

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	MTBE-d3 Response	1,4-Dioxane Response
P&A 1	7860914	23241
P&A 2	7934985	25237
P&A 3	7138921	21491
P&A 4	7517331	19688
P&A 5	7637458	26855
P&A 6	7437355	20167
P&A 7	7732026	26194
P&A 8	7465293	22473
Ave	7590535	23168
Std Dev	257025	2711
%RSD	3.39	11.70

Table 5: Precision and Accuracy Compound Response

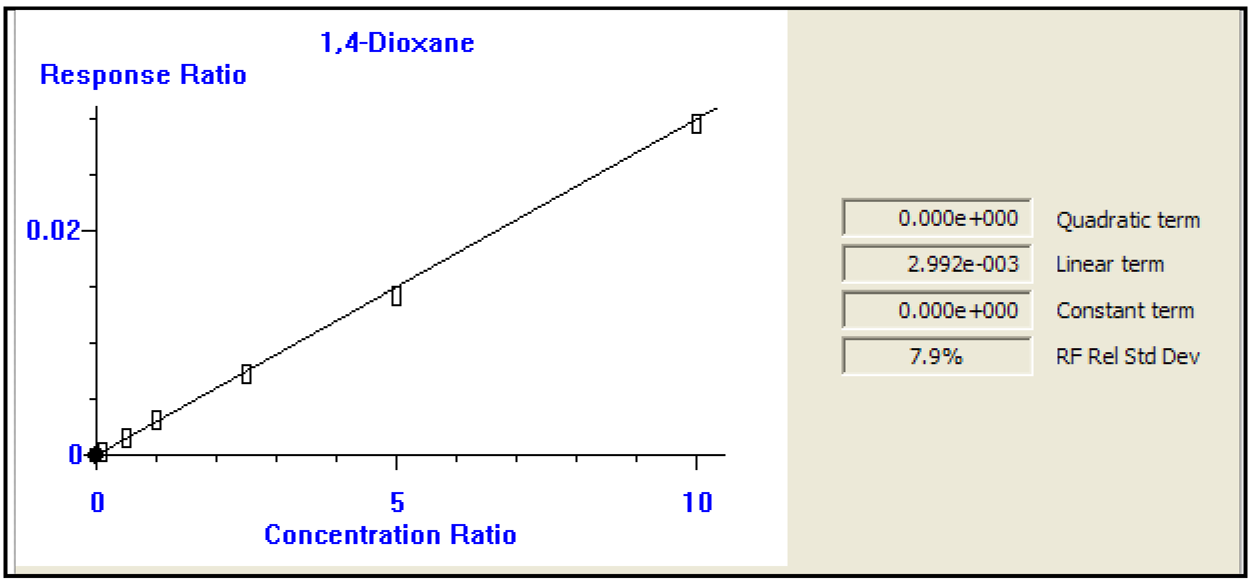

Figure 1: Calibration Curve of 1,4-Dioxane

Conclusions

As the detection limits of 1,4-Dioxane change it is important to be able to satisfy these requirements. Using the parameters outlined in this paper, 1,4-Dioxane can be detected at 1ppb. Due to the poor purge efficiency of 1,4-Dioxane, it was necessary to heat the samples and moisture control was very important. The Stratum PTC comes equipped with an innovative U-shaped condensate trap. The geometry of this trap enables more efficient water removal, thus making the Stratum PTC an excellent choice for this analysis.