Determination of 1,4-Dioxane in Water

AN_0001 Introduction

1,4-Dioxane is a synthetic industrial chemical stabiliser used for many different purposes such as paint strippers, greases and waxes. Historically, 90% of all 1,4-dioxane was used for the transport of chlorinated hydrocarbons in aluminium containers[1].

This highly soluble clear ether does not readily bind to soils therefore it can easily leach into groundwater resulting in contamination of drinking water. Contamination of water supplies is vast in the USA with dioxane levels ranging from 2ppb to over 11,000ppb being detected at 67 different sites[1]. Critically, over 34 of these different locations have been deemed a national priority for the release of the contaminant/hazardous substance into the drinking water supply[2]. Due to being resistant in nature, 1,4-dioxane does not undergo natural bio-degradation processes.

The United States Environmental Protection Agency (US EPA) classified 1,4-dioxane as a likely carcinogenic to humans with common side effects including irritation to eyes, nose and throat with longer effects being liver and kidney damage[1].

Federal screening levels and state health-based drinking water guidance values have been established however as of yet, no federal law has been determined. The guideline range is from

0.25ppb to 77ppb, with the average contamination ranging between 3-6ppb[1].

Experimental

Instrumentation: Teledyne Tekmar Atomx Purge and Trap couple with a SCION Single Quad GC-MS
Software: SCION Mass Spectrometry Work Station

The analytical conditions of the GC-MS can be found in Figure 1.

Table 1. Analytical conditions of GC-MS

Conditions	
Injector	S/SL at 200°C Split 50mL/min
Columns	30m x 0.25mm x 1.4µm (Scion624-MS)
Oven Conditions	40°C Hold 2 Minutes 100°C at 10°C 200°C at 30°C Hold 4 minutes
Carrier	Helium 1mL/min
MS	SIM Method
Peak Identification	1,4- Dioxane: m/z 88

High purity water was gravimetrically spiked with low level concentrations of 1,4-Dioxane. Standards were prepared with a concentration range of 0.3ppb to 6ppb with the addition of fluorobenzene as an internal standard. Samples were prepared using the Atomx Sparge 5mL vessel with no heating unit installed on the purge and trap system.

Single Ion Monitoring (SIM) was used to identify m/z 88, the quantifier ion of 1,4-Dioxane.

Results

Table 2 shows the expected and actual values, peak area and associated levels of the spiked calibrators.

Table 2. Expected/actual values, peak area and associated calibrator level

Expected Value (ppb)	Actual Value (ppb)	Area	Level
0.30	0.292	57027	6
0.50	0.521	90774	5
1.0	1.217	152159	4
2.0	2.437	303620	3
3.0	3.120	332391	2
6.0	6.143	642293	1

The values shown in Table 2 were used to generate a calibration curve for 1,4-Dioxane. The R^2 value of the 1,4-Dioxane was 0.9995 and was calculated using peak area vs ratio of standard to internal standard.

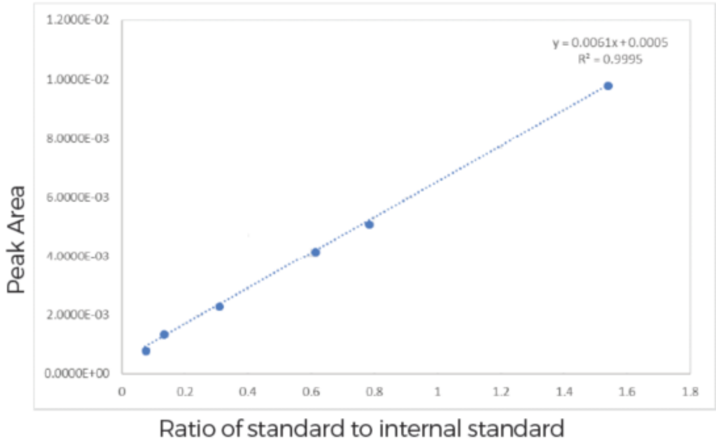

Figure 1. Calibration curve of 1,4-Dioxane

Fluoroebenzene, the internal standard used and 1,4-Dioxane had retention times of 5.94 minutes and 6.75 minutes, respectively, making this application extremely time efficient. Figure 2 shows the extracted ion chromatogram of 1,4-Dioxane at 0.3ppb, using m/z 88.

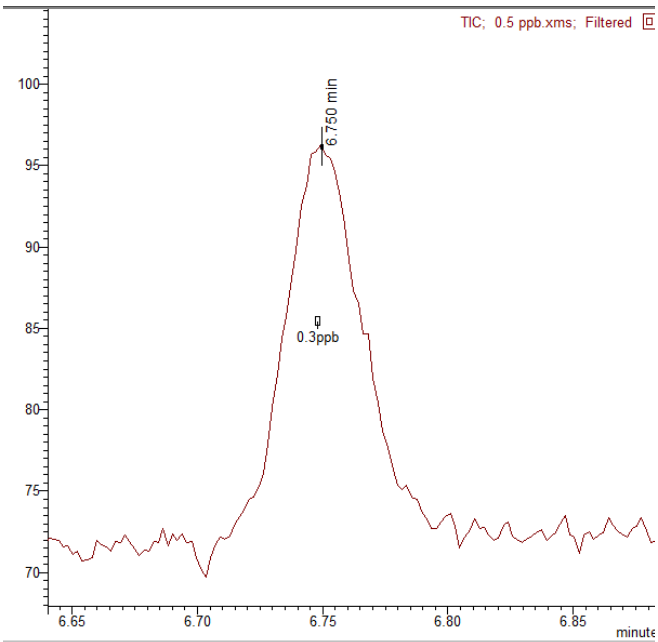

Figure 2. 1,4-Dioxane standard at 0.3ppb

Although the limit of detection (LOD) was not tested, the lowest standard of 0.3ppb yielded a S/N (RMS) of 11, this is indicating that the LOD would be considerably lower.

Conclusion

Using the highly sensitive, automated SCION GC-MS (SQ) coupled with the Teledyne Tekmar Atomx Purge and Trap, it was possible to detect low level concentrations of 1,4-Dioxane in drinking water. The simple yet reliable method demonstrates excellent sensitivity with low detection limits of 0.3ppb being easily quantified and distinguishable from the baseline. It is possible to increase the sensitivity of the system through the addition of a heating unit on the Atomx.