Application Note

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Analysis of Epichlorohydrin in Drinking Water

Using the Tekmar Atomx XYZ P&T, Agilent 8890 GC, and Agilent 5977C GC/MSD

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Abstract

Epichlorohydrin (ECH) is a versatile starting material in the production of drugs and polymers, and also serves as an insect fumigant and solvent for organic synthesis reactions. ECH-based polymer pipes are widely employed in drinking water production. Due to its extreme reactivity and toxicity, many nations have started imposing limits on the amount of ECH allowable in drinking water, including the new European Union Directive 2020/2184 requiring a limit of 100 parts per trillion (ppt). Many European countries go beyond this, recommending compliance at a third of this limit, indicating a 30 ppt minimum detection limit (MDL).

In the United States, drinking water analysis of volatile organic compounds (VOCs) is performed by purge and trap (P&T) concentration, using standard US Environmental Protection Agency (EPA) methods. In Europe, most drinking water detection limits are achieved with static headspace, but to reach the desired 30 ppt MDL, P&T will be used in this application. Drinking water samples were prepared using various EPA methods, with modifications to the matrix and method parameters. Sample preparation was performed using the Teledyne LABS Tekmar Atomx XYZ P&T concentrator, with analysis performed by an Agilent 8890 Gas Chromatograph (GC) and an Agilent 5977C GC/MSD. Calibration data, MDL, mid-point calibration check, and accuracy and precision of n = 40, 30 ppt ECH samples are also presented.

Introduction

The Tekmar Atomx XYZ is Teledyne LABS' 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, thereby reducing peak interference and increasing GC column life span. 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.

Experimental

Sample preparation

Two working calibration standards were prepared in methanol at the concentrations of 100 parts per billion (ppb) and 500 ppb from a commercially available ECH standard.

An eight-point linear calibration curve ($R^2 \geq 0.995$) was prepared from 10 to 500 ppt. The relative response factor (RRF) was calculated for ECH using the internal standard: 1,4-difluorobenzene. The internal standard was prepared in methanol from a commercially available 1,4-difluorobenzene standard at a concentration of 200 ppb. Then, 5 µL was mixed with each 5 mL sample for a resulting concentration of 200 ppt.

Seven 30 ppt standards were prepared to calculate the MDL. Also, seven 100 ppt standards were prepared for the accuracy and precision calculations of the mid-point calibration check.

All calibration, MDL, and mid-point calibration check standards were analyzed with the Tekmar Atomx XYZ conditions in Table 1. GC/MS conditions are shown in Table 2.

Instrument conditions

Table 1. Tekmar Atomx XYZ P&T water method conditions.

Parameter	Value
Standby	
Valve Oven Temperature	140 °C
Transfer Line Temperature	140 °C
Sample Mount Temperature	90 °C
Water Heater Temperature	90 °C
Sample Cup Temperature	20 °C
Soil Valve Temperature	50 °C
Standby Flow	10 mL/min
Purge Ready Temperature	40 °C
Desorb	
Methanol Needle Rinse	Off
Water Needle Rinse Volume	7.00 mL
Sweep Needle Time	0.25 min
Desorb Preheat Temperature	245 °C
GC Start Signal	Begin desorb
Desorb Time	1.00 min
Drain Flow	300 mL/min
Desorb Temperature	250 °C
Purge	
Sample Equilibrate Time	0.00 min
Pre-sweep Time	0.25 min
Prime Sample Fill Volume	3.00 mL
Sample Volume	5.00 mL
Sweep Sample Time	0.25 min
Sweep Sample Flow	100 mL/min
Sparge Vessel Heater	Off
Purge Time	11.00 min
Purge Flow	40 mL/min
Purge Temperature	20 °C
MCS Purge Temperature	20 °C
Dry Purge Time	0.50 min
Dry Purge Flow	100 mL/min
Dry Purge Temperature	20 °C

Parameter	Value
Bake	
Methanol Glass Rinse	Off
Water Bake Rinses	2
Water Bake Rinse Volume	7.00 mL
Bake Rinse Sweep Time	0.25 min
Bake Rinse Sweep Flow	100 mL/min
Bake Rinse Drain Time	0.40 min
Bake Time	2.00 min
Bake Flow	200 mL/min
Bake Temperature	260 °C
MCS Bake Temperature	200 °C
Trap	
Vocarb 3000 (K)	
Chiller Tray	
On, 10 °C	
Purge Gas	
Nitrogen	

Results and discussion

The linear calibration curve, MDL, and mid-point calibration check standard data are shown in Table 3. Figure 1 displays the calibration curve for ECH. Additionally, a long-term, 40-sample, 30 ppt low-point calibration check standard study was performed with the data shown in Table 4.

Table 2. Agilent 8890 GC and Agilent 5977C GC/MSD system conditions.

Parameter	Value
8890 GC Conditions	
Column	Agilent DB-624 UI, 20 m × 0.18 mm, 1 µm film, column flow: 0.8 mL/min
Oven Profile	40 °C, 1 min 12 °C/min to 130 °C 40 °C/min to 220 °C, 1 min Run time: 11.75 min
Inlet	200 °C, 30:1 split Septum purge flow: 0.5 mL/min, 16.53 psi Carrier gas: helium
5977C GC/MSD Conditions	
Temperature	Transfer line: 250 °C Source: 230 °C Quadrupole: 150 °C
SIM	1,4-Difluorobenzene ions: 114 Epichlorohydrin ions: 57,49,62 Solvent delay: 3.50 min Dwell time: 100
Current	Gain factor 15, BFB auto tune

Table 3. Epichlorohydrin calibration, method detection limit (MDL), and mid-point calibration check standard data.

Compound	Calibration (10 to 500 ppt)				Method Detection Limits (n = 7, 30 ppt)			Mid-Point Calibration Check (n = 7, 100 ppt)	
	RT (min)	Confirm. Ion	Cal. Type	Linearity ($R^2 \geq 0.995$)	MDL (ppt)	Precision (≤ 20%)	Accuracy (± 30%)	Precision (≤ 20%)	Accuracy (± 30%)
1,4-Difluorobenzene (IS)	4.44	114							
Epichlorohydrin	5.43	57	Linear	0.9997	4.4	4.7	99	3.0	93

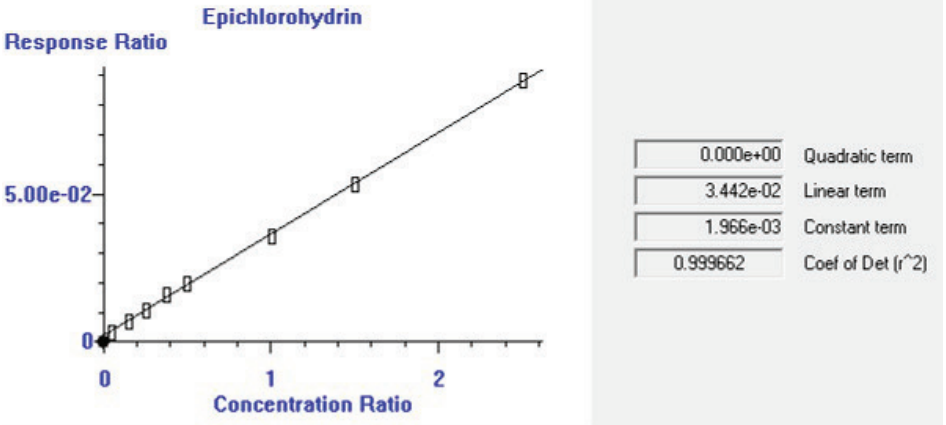

Figure 1. Epichlorohydrin calibration curve fit from 10 to 500 ppt, showing coefficient of determination ($R^2 \geq 0.995$).

Table 4. Epichlorohydrin long-term calibration check data.

Compound	Long-Term Calibration Check (n = 40, 30 ppt)	
	Precision (≤ 20%)	Accuracy (± 20%)
Fluorobenzene (IS)		
Epichlorohydrin	4.3	92

Figure 2 displays a 30 ppt ECH standard in water in SIM mode with confirmation ion 57 *m/z* and two secondary ions 62 and 49 *m/z*. Figure 3 displays a 100 ppt ECH standard in water in SIM mode with confirmation ion 57 *m/z* and two secondary ions 62 and 49 *m/z*. Figure 4 displays the results of the long-term calibration check standard study for ECH.

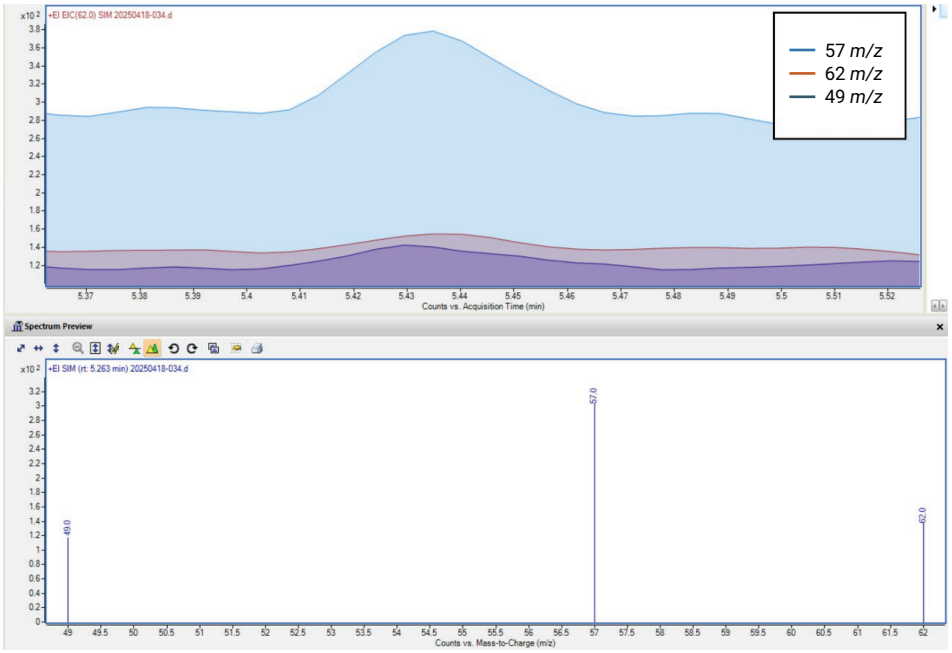

Figure 2. Total ion chromatogram of 30 ppt epichlorohydrin standard in a drinking water sample with confirming ion (57 *m/z*) and two secondary ions (62 and 49 *m/z*).

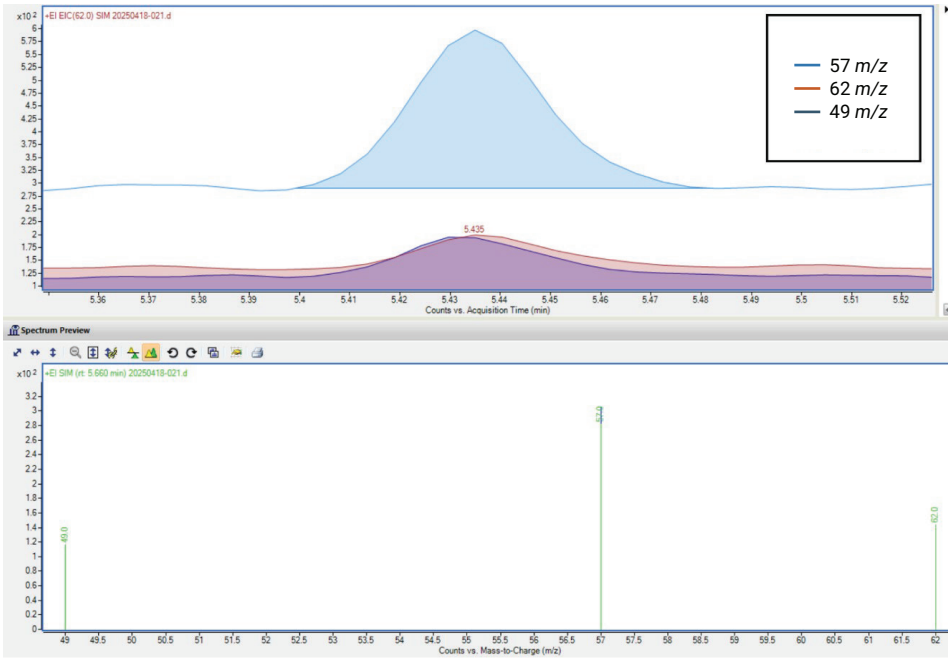

Figure 3. Total ion chromatogram of 100 ppt epichlorohydrin standard in a drinking water sample with confirming ion (57 *m/z*) and two secondary ions (62 and 49 *m/z*).

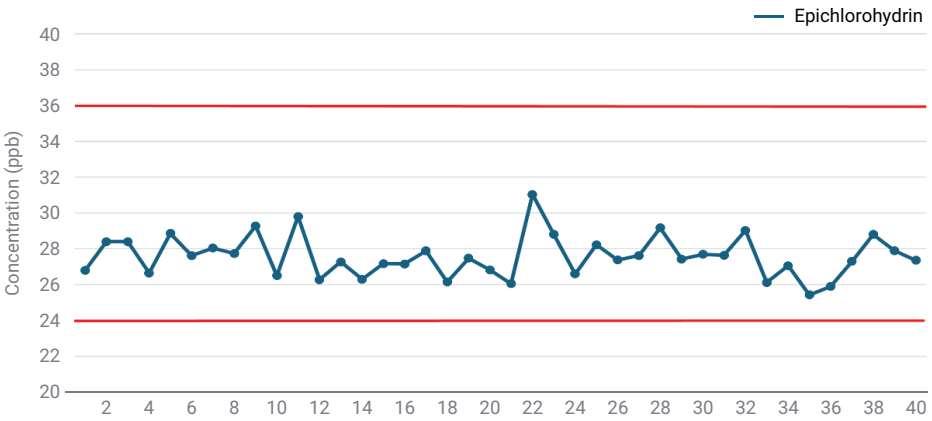

Figure 4. Results of the long-term study, n = 40, 30 ppt epichlorohydrin standard in drinking water samples. Red lines represent ±20% accuracy method requirement for continuing calibration check standards.

Conclusion

This study demonstrates the capability of the Teledyne LABS Tekmar Atomx XYZ P&T concentrator to process low-level ECH in drinking water samples, with detection performed using an Agilent 8890 GC and Agilent 5977C GC/MSD. The linearity of the calibration curve from 10 to 500 ppt passed method requirements, including verification of the initial calibration curve. The 10 ppt standard passed the lower limit of quantitation (LLOQ) within ± 50% of its true value, while all other calibration levels (> LLOQ) passed within ± 30%. The blank following the highest point in the calibration curve passed method carryover requirements by remaining below half pf the LLOQ. Furthermore, the application proved robust during a long-term study with 40 samples of a 30 ppt ECH standard, achieving 4.3% precision and 92% accuracy of the recovery.

References

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