Chromatography
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Analysis of Epichlorohydrin in Drinking Water Using the Tekmar Lumin P&T and the Agilent 8890 GC and 5977C MS

Amy Nutter, Technical Product Specialist; Teledyne LABS

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Abstract

Epichlorohydrin (ECH) is a versatile starting material in the production of drugs and polymers and is also used as an insect fumigant and solvent for organic synthesis reactions. ECH-based polymer pipes are widely employed in the production of drinking water. Due to its extreme reactivity and toxicity, many nations have begun 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 1/3 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 United States Environmental Protection Agency (EPA) methods. In Europe, most drinking water detection limits are achieved with static headspace, but in order to reach the desired 30 ppt MDL, P&T will be used in this application. Variations EPA drinking water methods, with modifications to the matrix and method parameters, will be made to prepare the drinking water samples by the Teledyne LABS Tekmar Lumin P&T concentrator combined with the AQUATek LVA autosampler with analysis performed by an Agilent 8890 Gas Chromatograph (GC) and 5977C Mass Spectrometer (MS) (GC/MS). Calibration data, MDL, mid-point calibration check, and accuracy and precision of n=40, 30 ppt ECH samples will also be presented.

Introduction

The Tekmar Lumin P&T has an innovative Moisture Control System (MCS) that improves water vapor removal, thereby reducing peak interference and increasing GC column lifespan. The AQUATek LVA autosampler has an 84-position chiller enabled sample tray and utilizes a fixed volume loop that transfers the liquid sample, internal standards, and surrogate standards to the Lumin P&T concentrator. It initiates a clean-up cycle where the sample loop and sparger are cleaned with 90 °C water, assuring method carryover compliance is met. 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.

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 correlation coefficient (r^2) calibration curve was prepared from 10 to 500 ppt with regression value (r^2) ≥0.995. 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 parts per billion (ppb), after which 5 µL was then 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 Teledyne LABS Tekmar Lumin P&T and AQUATek LVA conditions in Table I. GC/MS conditions are shown in Table II.

Experimental Instrument Conditions

Table I Tekmar Lumin P&T and 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	35 °C	GC Start Signal	Begin Desorb
MCS Purge Temp	20 °C	**Bake**	**Variable**
Purge	**Variable**	Bake Time	2.00 min
Purge Temp	20 °C	Bake Temp	260 °C
Purge Time	11.00 min	MCS Bake Temp	200 °C
Purge Flow	40 mL/min	Bake Flow	200 mL/min
Dry Purge Temp	20 °C	**AQUATek LVA**	**Variable**
Dry Purge Time	0.50 min	Sample Loop Time	0.35 min
Dry Purge Flow	100 mL/min	Sample Transfer Time	0.35 min
Sample Heater Enable	Off	Rinse Loop Time	0.30 min
		Sweep Needle Time	0.30 min
		Presweep Time	0.25 min
Trap	Vocarb 3000 (K)	Water Temp	90 °C
Chiller Tray	On, 10 °C	Bake Rinse Cycles	2
Purge Gas	Nitrogen	Bake Rinse Drain Time	0.35 min

Table II Agilent 8890 GC and 5977C MS System Conditions

Agilent 8890 GC Conditions	
Column	DB-624 UI, 20 m x 0.18 mm, 1µm 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
Agilent 5977C MS Conditions	
Temp	Transfer Line 250 °C; Source 230 °C; Quad 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

Results

The linear correlation coefficient of the calibration curve (r^2), MDL, and mid-point calibration check standard data are shown in Table III. In addition, a long-term, 40 sample, 30 ppt low-point calibration check standard study was performed with the data shown in Table IV. Figure 1 displays the average response factor calibration curve for ECH. 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 ECH long-term calibration check standard study.

Table III Epichlorohydrin Calibration, MDL, and Mid-Point Calibration Check Standard Data

Compound	Calibration (10-500 ppt)					Method Detection Limits (n=7, 30 ppt)		Mid-Point Calibration Check (n=7, 100 ppt)	
	Ret. Time	Confirm. Ion	Cal Type	Linearity (r^2 ≥0.995)	MDL (ppt)	Precision (≤20%)	Accuracy (±30%)	Precision (≤20%)	Accuracy (±30%)
1,4-Difluorobenzene (IS)	4.31	114							
Epichlorohydrin	5.43	57	LIN	0.9990	6.3	6.7	100	3.7	111

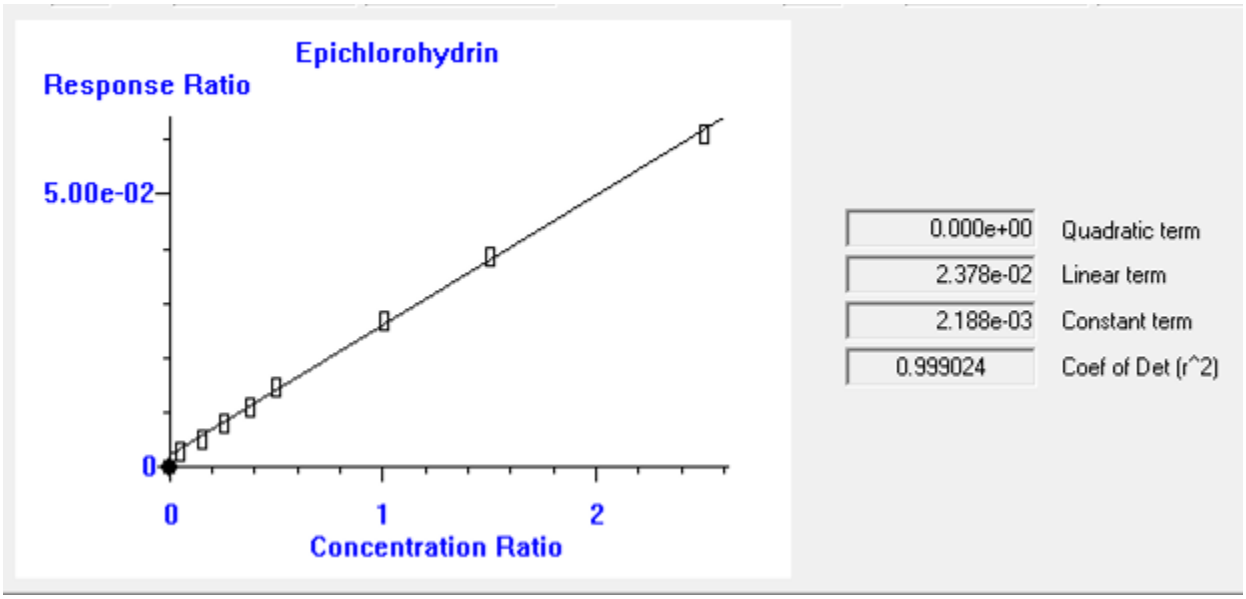

Figure 1 ECH linear correlation coefficient (r^2) calibration curve fit, 10-500 ppt.

Table IV **Epichlorohydrin Long-Term Calibration Check Data**		
Compound	Long-Term Calibration Check (n=40, 30 ppt)	
	Precision (≤20%)	Accuracy (±20%)
Fluorobenzene (IS)		
Epichlorohydrin	6.3	101

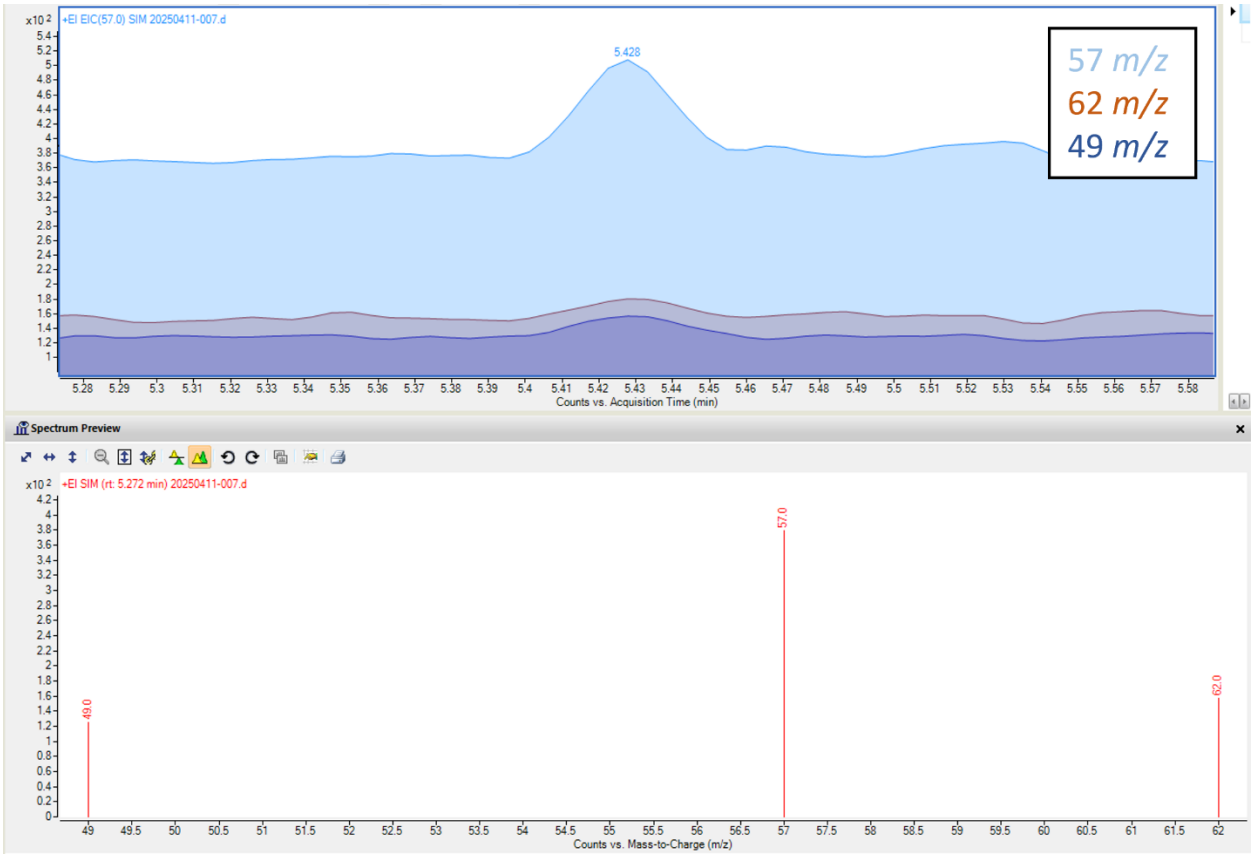

Figure 2 Total ion chromatogram (TIC) of 30 ppt ECH standard in a drinking water sample with confirming ion (57 *m/z*) and two secondary ions (62 and 49 *m/z*).

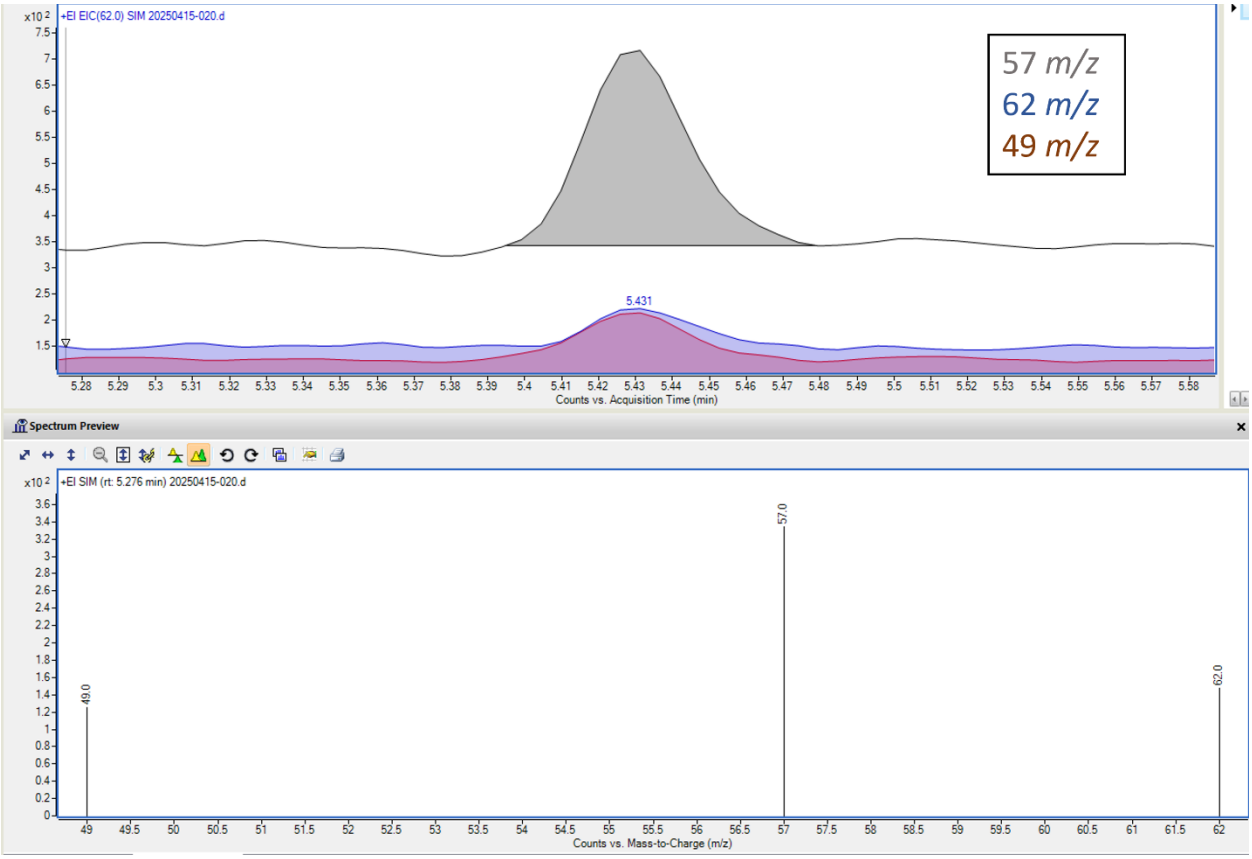

Figure 3 TIC of 100 ppt ECH standard in a drinking water sample with confirming ion (57 *m/z*) and two secondary ions (62 and 49 *m/z*).

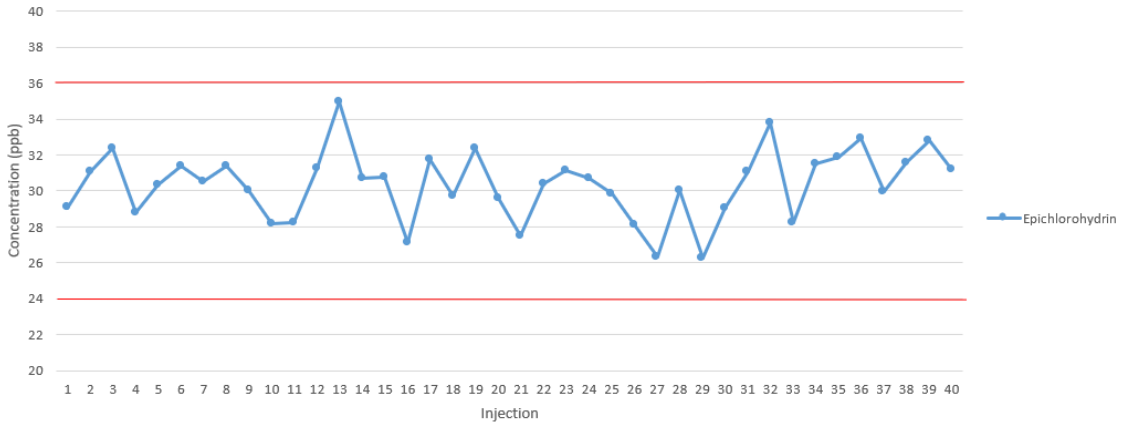

Figure 4 Results of long-term study, n=40, 30 ppt ECH standard in drinking water samples. Red lines represent ±20% accuracy method requirement for continuing calibration check standards.

Conclusion

This study demonstrates the capability of the Teledyne LABS Tekmar Lumin P&T and AQUATek LVA system to process low-level ECH in drinking water samples with detection by an Agilent 8890 GC and 5977C MS. The linearity of the calibration curve from 10 to 500 ppt passed method requirements, including the verification of the initial calibration curve with the 10 ppt passing the lower standard (LLOQ) recalculation within ±50% of its true value and the rest of the calibration curve (>LLOQ) passing with ±30% of their true value. The blank after the highest point in

the calibration curve passed method carryover requirements by remaining <1/2 the LLOQ. Furthermore, the application proved robust during a long-term study with 40 samples of a 30 ppt ECH standard with 6.3% precision and 101% accuracy of the recovery.

References

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