

# LC-MS/MS Analysis of Phosphonic Acid in Water using a Venusil® HILIC column

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## Overview

Phosphonic acid is a degradation product of the fungicide fosetyl aluminum (fosetyl-Al).

In this application note we show a fast and robust method for the LC-MS/MS determination and quantification of phosphonic acid in water.

## Experimental

The experiments were performed on an Agilent® 1290 Infinity I system connected to an Agilent 6495a triple quadrupole MS detector.

## MS Conditions

Table 1. Source Parameters

Parameter	Value(-)
Gas Temp. (°C)	200
Gas Flow (L/min)	14
Nebulizer (psi)	30
Sheath Gas Heater	270
Sheath Gas Flow	12
Capillary (V)	4000
VCharging	400

Table 2. MRM Transitions

	Precursor Ion	Product Ion	Collision Energy (V)
MRM1	81	79	15
MRM2	81	63	38

## LC Conditions

**Column:** Venusil HILIC 3 µm

**Dimension:** 100 x 2.1 mm

**Part No.:** VH931002-0

**Mobile Phase:** A = 0.1 % Formic acid in water  
B = 0.1 % Formic acid in acetonitrile

Gradient:	Time(min)	%B
	0	5
	1	5
	7	90
	10	90

**Flow Rate:** 0.4 mL/min

**Injection:** 20 µL

**Column Temperature:** 30 °C

**Detection:** MSD

To mimic water samples with different salt concentrations, several solutions were mixed following the recipes for the EPA AAP media.

Table 3. Composition of EPA AAP media.

Component	Concentration (mg/L)
NaNO <sub>3</sub>	25.5
MgSO <sub>4</sub> ×7H <sub>2</sub> O	14.7
K <sub>2</sub> HPO <sub>4</sub>	1.04
NaHCO <sub>3</sub>	15.0
MgCl <sub>2</sub> ×6H <sub>2</sub> O	12.16
CaCl <sub>2</sub> ×2H <sub>2</sub> O	4.41
FeCl <sub>3</sub> ×6H <sub>2</sub> O	0.160
Na <sub>2</sub> EDTA×2H <sub>2</sub> O	0.300
H <sub>3</sub> BO <sub>3</sub>	0.186
MnCl <sub>2</sub> ×4H <sub>2</sub> O	0.415
ZnCl <sub>2</sub>	0.00327
CoCl <sub>2</sub> ×6H <sub>2</sub> O	0.00143
CuCl <sub>2</sub> ×2H <sub>2</sub> O	0.000012
Na <sub>2</sub> MoO <sub>4</sub> ×2H <sub>2</sub> O	0.00726



## Chromatograms and Calibration Curve

Figure 1a. TIC of water sample containing 2.0 µg/L phosphonic acid

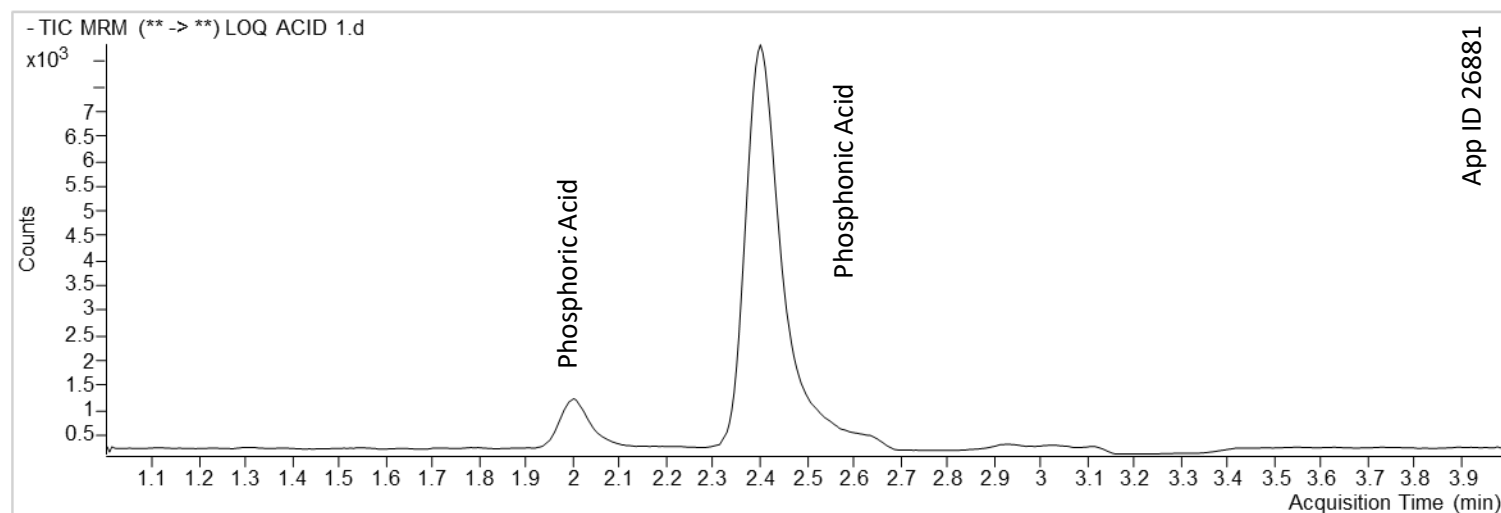


Figure 1b. MRM 81.0 -&gt; 79.0 of water sample

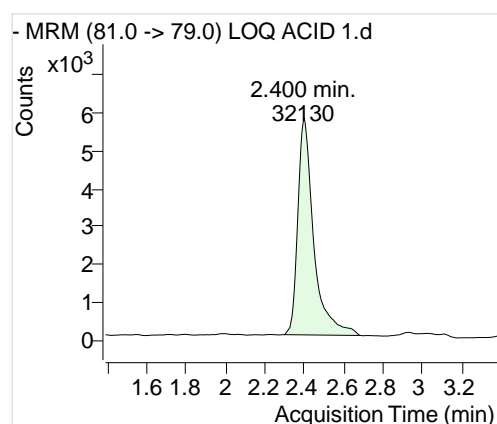


Figure 1c. Overlay MRM 81.0 -&gt; 79.0 and 81.0 -&gt; 63.0

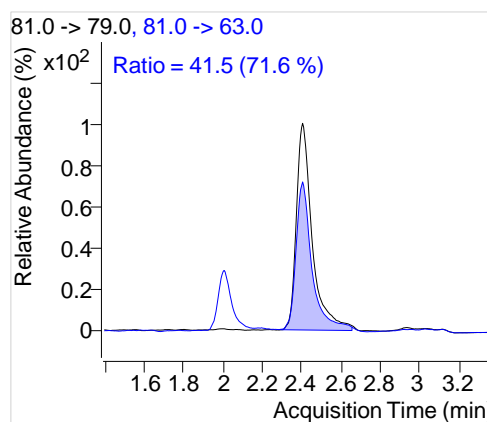
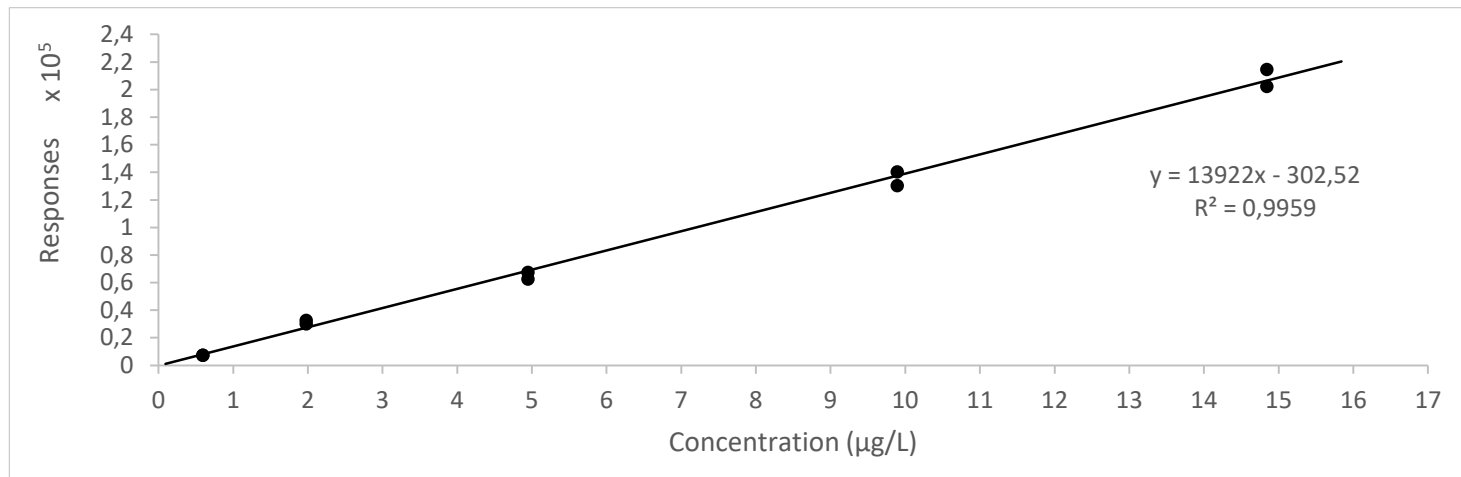


Table 4. Calibration Data

Sample	Analyte	Sample Type	R <sub>t</sub> (min)	Response	Concentration (µg/L)	Expected Concentration (µg/L)	Accuracy (%)
LIN 1 ACID	Phosphonic Acid	Calibration	2.405	7,375	0.5515	0.5937	92.89
LIN 2 ACID	Phosphonic Acid	Calibration	2.400	32,723	2.3721	1.9792	119.85
LIN 3 ACID	Phosphonic Acid	Calibration	2.405	67,494	4.8697	4.9479	98.42
LIN 4 ACID	Phosphonic Acid	Calibration	2.410	140,292	10.0986	9.8958	102.05
LIN 5 ACID	Phosphonic Acid	Calibration	2.410	214,575	15.4342	14.8437	103.98



Figure 2. Calibration Curve



### Conclusion

The Venusil® HILIC column was effective in retaining phosphonic acid under isocratic conditions (**Figure 1a-c**). The LOD for the analysis of phosphonic acid was 0.6 µg/L and the LOQ was 2.0 µg/L for water samples with high salt content. The linearity of quantification was confirmed for concentrations between 2.0 µg/L and 15.0 µg/L with a recovery between 92 and 120 % (**Table 4**).



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