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

Stefano Aversa[1] and Luigi Margarucci[2]
[1]BioTecnologie BT S.r.l., Frazione Pantalla, 06059 Todi (PG), Italy
[2]Phenomenex S.r.l., Via M. Serenari 15/D, 40013 Castel Maggiore (BO), Italy

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)
$NaNO_3$	25.5
$MgSO_4 \times 7H_2O$	14.7
K_2HPO_4	1.04
$NaHCO_3$	15.0
$MgCl_2 \times 6H_2O$	12.16
$CaCl_2 \times 2H_2O$	4.41
$FeCl_3 \times 6H_2O$	0.160
$Na_2EDTA \times 2H_2O$	0.300
H_3BO_3	0.186
$MnCl_2 \times 4H_2O$	0.415
$ZnCl_2$	0.00327
$CoCl_2 \times 6H_2O$	0.00143
$CuCl_2 \times 2H_2O$	0.000012
$Na_2MoO_4 \times 2H_2O$	0.00726

Chromatograms and Calibration Curve

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

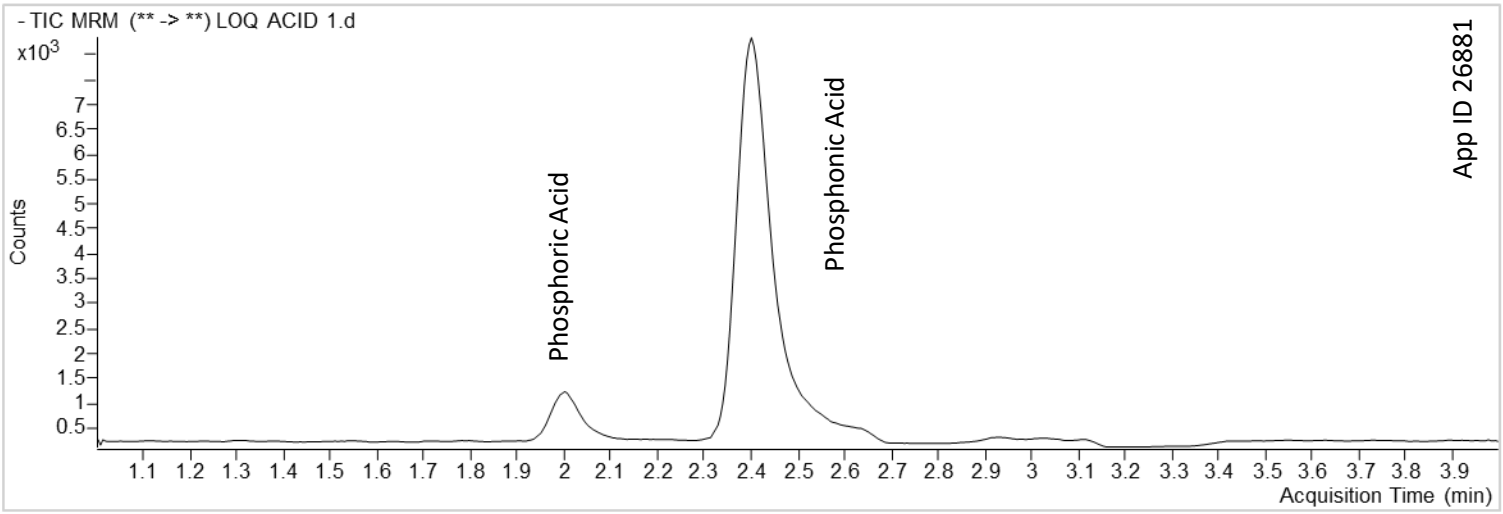


Figure 1b. MRM 81.0 -> 79.0 of water sample

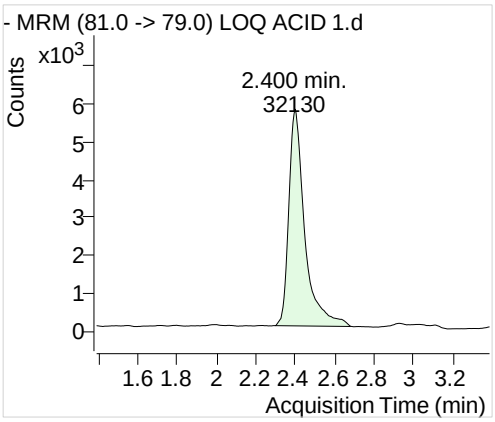


Figure 1c. Overlay MRM 81.0 -> 79.0 and 81.0 -> 63.0

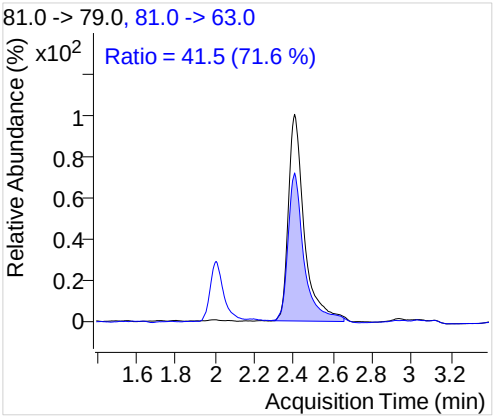
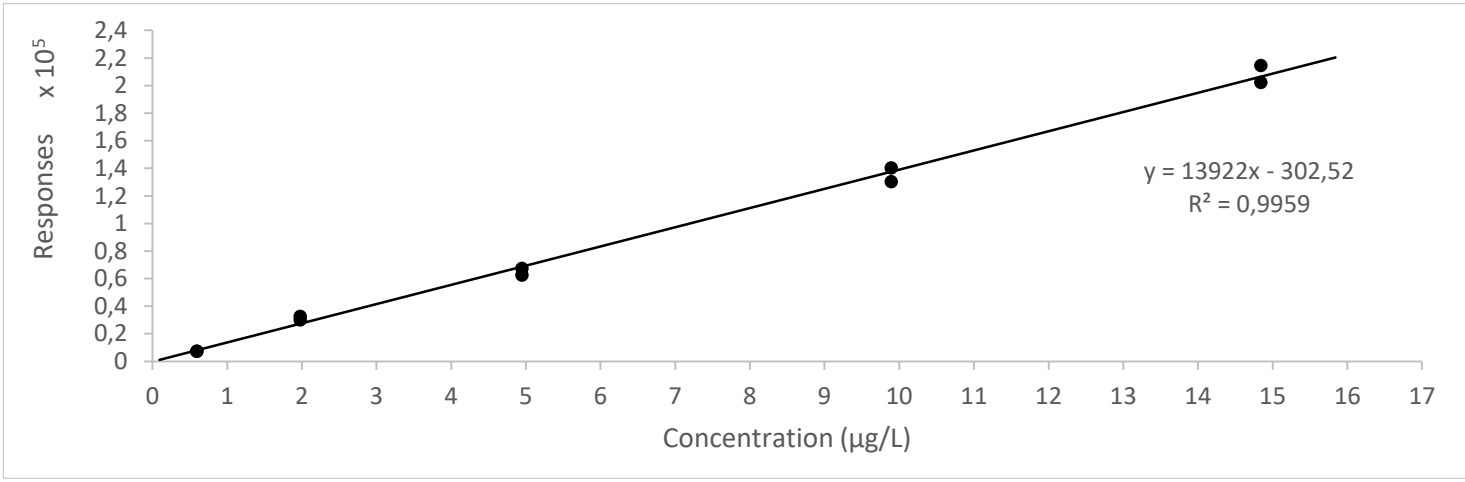


Table 4. Calibration Data

Sample	Analyte	Sample Type	R_t (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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Australia
t: +61 (0)2-9428-6444
auinfo@phenomenex.com

Austria
t: +43 (0)1-319-1301
anfrage@phenomenex.com

Belgium
t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
beinfo@phenomenex.com

Canada
t: +1 (800) 543-3681
info@phenomenex.com

China
t: +86 400-606-8099
cninfo@phenomenex.com

Czech Republic
t: +420 272 017 077
cz-info@phenomenex.com

Denmark
t: +45 4824 8048
nordicinfo@phenomenex.com

Finland
t: +358 (0)9 4789 0063
nordicinfo@phenomenex.com

France
t: +33 (0)1 30 09 21 10
franceinfo@phenomenex.com

Germany
t: +49 (0)6021-58830-0
anfrage@phenomenex.com

Hong Kong
t: +852 6012 8162
hkinfo@phenomenex.com

India
t: +91 (0)40-3012 2400
indiainfo@phenomenex.com

Indonesia
t: +62 21 5010 9707
indoinfo@phenomenex.com

Ireland
t: +353 (0)1 247 5405
eireinfo@phenomenex.com

Italy
t: +39 051 6327511
italiainfo@phenomenex.com

Japan
t: +81 (0) 120-149-262
jpinfo@phenomenex.com

Luxembourg
t: +31 (0)30-2418700
nlinfo@phenomenex.com

Mexico
t: 01-800-844-5226
tecnicomx@phenomenex.com

The Netherlands
t: +31 (0)30-2418700
nlinfo@phenomenex.com

New Zealand
t: +64 (0)9-4780951
nzinfo@phenomenex.com

Norway
t: +47 810 02 005
nordicinfo@phenomenex.com

Poland
t: +48 22 104 21 72
pl-info@phenomenex.com

Portugal
t: +351 221 450 488
ptinfo@phenomenex.com

Singapore
t: +65 800-852-3944
sginfo@phenomenex.com

Slovakia
t: +420 272 017 077
sk-info@phenomenex.com

Spain
t: +34 91-413-8613
espinfo@phenomenex.com

Sweden
t: +46 (0)8 611 6950
nordicinfo@phenomenex.com

Switzerland
t: +41 (0)61 692 20 20
swissinfo@phenomenex.com

Taiwan
t: +886 (0) 0801-49-1246
twinfo@phenomenex.com

Thailand
t: +66 (0) 2 566 0287
thaiinfo@phenomenex.com

United Kingdom
t: +44 (0)1625-501367
ukinfo@phenomenex.com

USA
t: +1 (310) 212-0555
info@phenomenex.com

All other countries/regions
Corporate Office USA
t: +1 (310) 212-0555
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