Application Note 01508

Quantitative Analysis of the Herbicide Imazaquin on the Varian 320-MS Triple Quadrupole LC/MS/MS

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Introduction

The imidazolinones belong to a class of herbicides, most of which are used to control weeds in legume, cereal crops, and peanuts.[1] These ALS-inhibiting herbicides are characterized by relatively low application rates, typically less than 100 g of active ingredient per hectare. Consequently, the expected environmental concentrations at which these analytes might be found makes their detection and analysis difficult compared to that of traditional herbicides.[2] It is important to monitor these compounds because they are known to persist in soil.[1] Accordingly, a highly sensitive, selective, and accurate method has been developed using the Varian 320-MS Triple Quadrupole LC/MS/MS to quantify the concentration of the imidazolinone herbicide imazaquin (Figure 1) at trace levels.

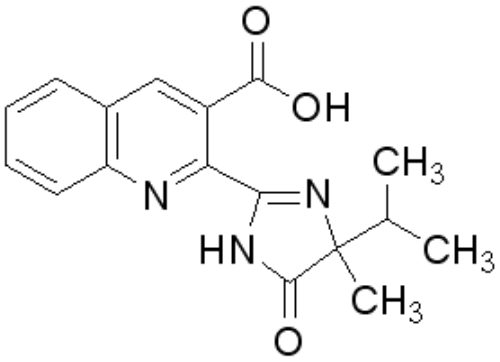

Figure 1. Molecular structure of the herbicide imazaquin.

In this analysis, the detection limit for imazaquin was first determined by serial dilution. Then, a calibration curve was generated from 0.05 ppb to 200 ppb.

Instrumentation

- Varian 320-MS Triple Quadrupole LC/MS/MS with ESI source
- Varian 212-LC Binary Solvent Delivery Modules
- Varian Prostar™ 410 AutoSampler
- Varian MetaTherm™ HPLC Column Heater (Varian Part No. A9542)

Sample Preparation

Solutions were prepared from a known standard at concentrations of 0.05, 0.2, 1, 5, 10, 50, and 200 ppb.

HPLC Conditions

Column:	Polaris™ C18-A, 3 µm, 50 x 2.0 mm ID (Varian Part No. A2001050X020)
Mobile Phase:	60:40 H_2O:CH_3OH with 0.1% formic acid
LC Program:	300 µL/min isocratic flow
Injection Volume:	20 µL
Column Temp:	30 °C

MS Parameters

Ionization Mode:	ESI Positive
API Drying Gas:	35 psi at 350 °C
API Nebulizing Gas:	50 psi

Table 1. MS Transitions

Compound	Precursor	Product	Dwell Time (ms)	CID Energy (V)
Imazaquin	312.0	267.0	20	20.0
	312.0	199.0	20	30.5

Results & Discussion

An RMS signal-to-noise ratio of 61 was obtained for a 20-µL injection of a 0.05 ppb standard. Figure 2 shows the chromatogram resulting from a 0.20 ppb injection.

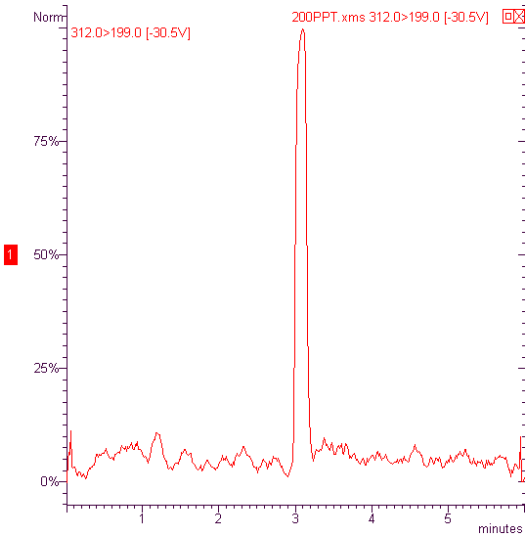

Figure 2. Mass chromatogram for a 0.20 ppb injection of the herbicide imazaquin.

Based on this information, a calibration curve was run from 0.05 ppb to 200 ppb. Figure 3 shows the resulting calibration curve with excellent r^2 value of 0.999.

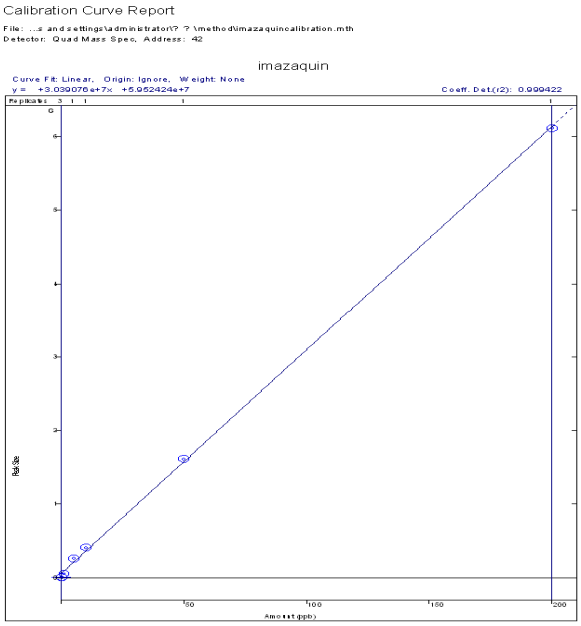

Figure 3. Calibration curve for imazaquin from 0.05 ppb to 200 ppb. r^2 value is 0.999.

Conclusion

The Varian 320-MS Triple Quadrupole Mass Spectrometer is able to detect and quantify the imidazolinone herbicide imazaquin from 0.05 ppb to 200 ppb. This analysis shows excellent sensitivity of the Varian 320-MS for quantitative analysis.

References

1. Anisuzzaman, A.K.M.; Amin, M.; Ogg, N.; Hoq, F.; Kanithi, M.R.; Jenkins, R.E.; "Synthesis of Dimethyl Derivatives of Imidazolinone Herbicides: Their Use in Efficient Gas Chromatographic Methods for the Determination of These Herbicides" *J. Agric. Food Chem.* **2000**, 48, 5893—5902.
2. Rodriguez, M.; Orescan, D.B.; "Confirmation and Quantitation of Selected Sulfonylurea, Imidazolinone, and Sulfonamide Herbicides in Surface Water Using Electrospray LC/MS" *Anal. Chem.* **1998,** 70, 2710-2717.

These data represent typical results.
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