

ENVIRONMENTAL ANALYSIS

TRACE LEVEL DETERMINATION OF AMITROLE IN SURFACE AND GROUND WATER BY DIRECT AQUEOUS INJECTION ON THE AGILENT 6495 LC/MS/MS

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ABSTRACT

This Solution note outlines the trace level analysis of the herbicide Amitrole in surface and ground water on the Agilent 6495 LC/MS/MS with a direct injection of 100 μ L sample.

INTRODUCTION

Amitrole is a triazole herbicide used to control grasses, broadleaf weeds and aquatic vegetation. It has high solubility in water with little adsorption on organic matter leading to potentially high residual levels in surface and ground waters. Monitoring of amitrole is carried out on raw waters used for drinking water production where regulatory compliance limits of 0.1 μ g/L will apply to the relevant metabolite 1,2,4-triazole, which can also be formed from other triazole herbicides.

Amitrole is a small highly polar molecule leading to a demanding analysis at trace levels. The very high sensitivity of the Agilent 6495 LC/MS/MS however enables reliable detection and measurement with a direct aqueous injection of only 100 μ L of sample and no external sample preparation.

Results are presented for Amitrole spiked into spring water and surface water at levels of down to 0.001 μ g/L. The HPLC gradient used was from 100% water to 100% methanol, both containing 0.1% formic acid. 15N-Amitrole was used as internal standard.



ANALYTICAL SYSTEM OVERVIEW

UHPLC-QQQ:

- Agilent 1290 Infinity Bin Pump (G4220A)
- Agilent 1290 Infinity Thermostat (G1330B)
- Agilent 1290 Infinity Sampler (G4226A), Extension Seat Capillary, 80 μ L (G4226-87303)
- Agilent 1290 Infinity TCC (G1316C)
- Agilent 6495 QQQ (G6495A), positive electrospray ionization , MRM
 - o Amitrole: m/z 85 \rightarrow m/z 43, collision energy 33 eV
 - o ^{15}N -Amitrole: m/z 86 \rightarrow m/z 43, collision energy 33 eV
- Column: Porous Graphite Carbon, 100 x 2.1 mm, 5 μ m
- Formic Acid, Water, Methanol

RESULTS

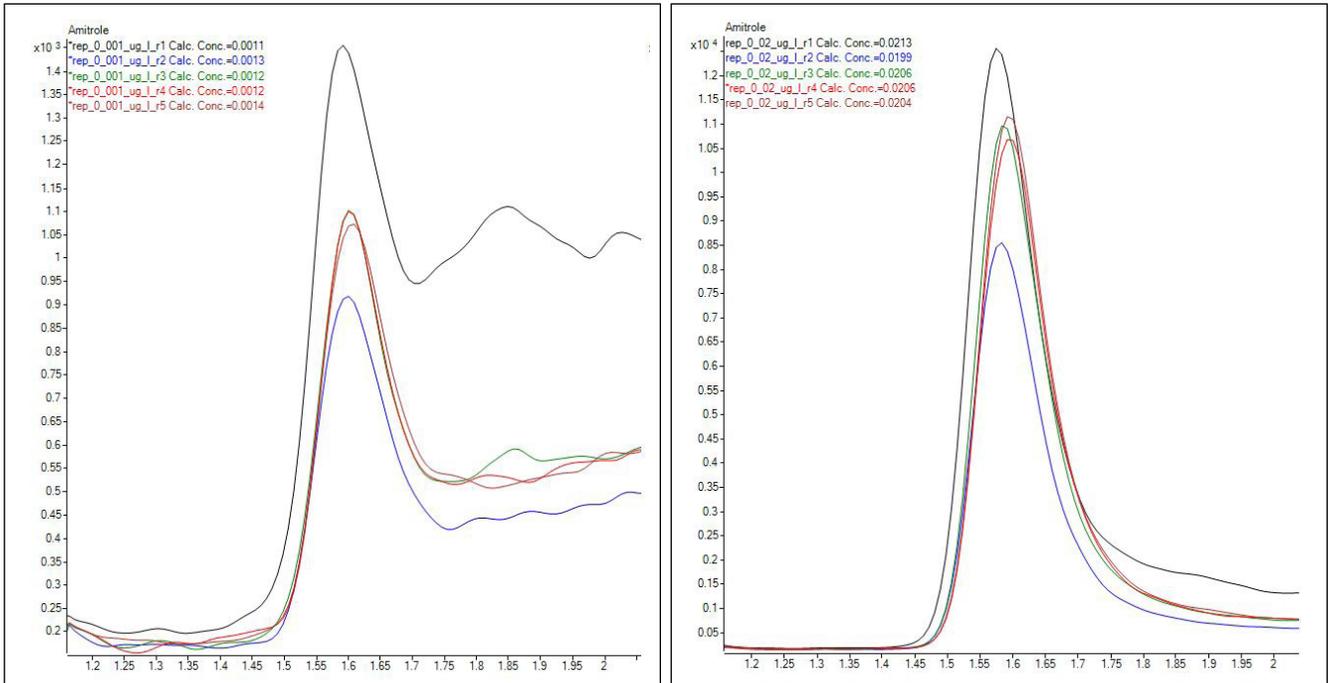


Figure 1: Surface water sample spiked with Amitrole at 0.001 µg/L (left) and 0.02 µg/L (right), overlay of 5 replicate samples. RSD (with internal standard) is 9.2% (0.001 µg/L) and 2.4% (0.02 µg/L).

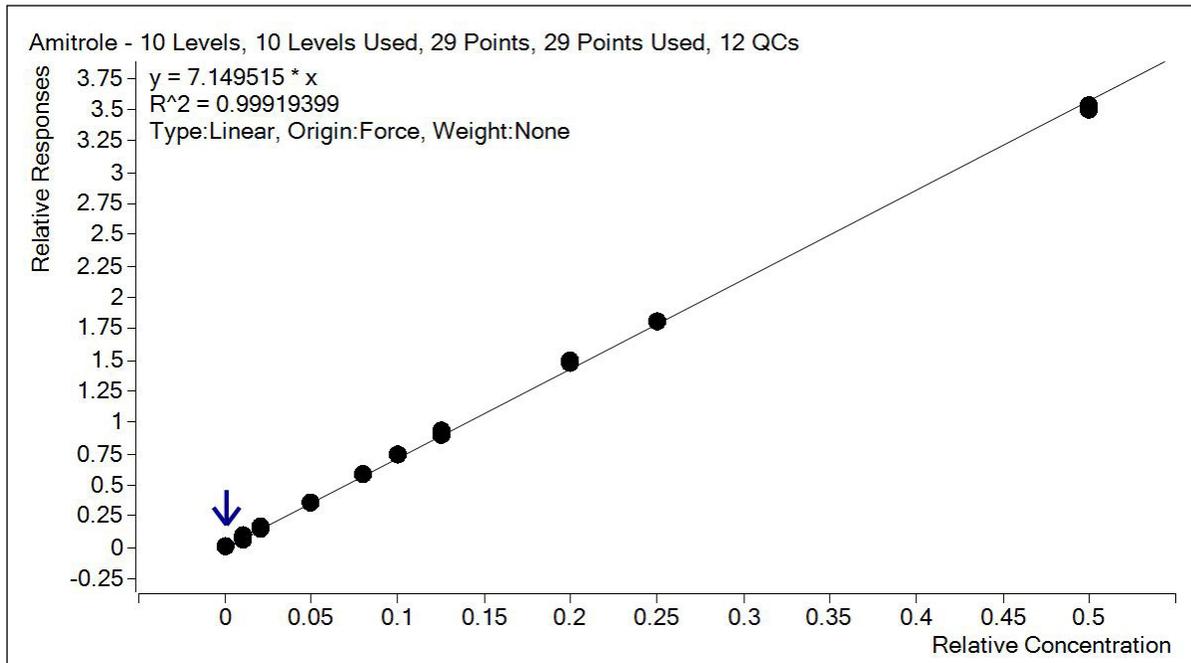


Figure 2: Linear Calibration of amitrole between 0.001 and 0.5 µg/L

SUMMARY:

This document outlines a robust direct injection method for the quantitative determination of Amitrole at trace levels in raw water samples which could also be applied to drinking water as part of a compliance monitoring program.



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