

**Title** LC/MS/MS detection of fungicide guazatine residues for quality assessment of commercial citrus fruit

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### Abstract

An analytical protocol was developed for investigating guazatine occurrence in citrus fruit with the aim of controlling the import of treated fruits in countries where the use of this fungicide is forbidden. The main constituents of guazatine mixture (GN, GG, GNG, GGN, GGG, GGGN and GGGG) and the internal standard (dodine) were separated by high performance liquid chromatography using a hydrophilic end-capped Aquasil C<sub>18</sub> column and detected by ESI/MS/MS of parent ions, operating in positive mode. Extraction from citrus peels was performed with 1% HCOOH in water/acetone (1:2 v/v). The analytical method was statistically validated on three of the main constituents (GG, GGN and GGG) representing more than 65% of the total content. The regression lines, ranging from 0.100 to 3.750 mg/L of total guazatine, showed  $r^2 > 0.990$ . Recoveries of about 81, 90 and 104% were obtained on average for the fortification level of 0.010, 0.035 and 0.060 mg/kg, respectively; the relative standard deviations ranged from 2 to 8% ( $n = 6$ ). The limit of detection was below 0.0050 mg/kg, while the limit of quantification did not exceed 0.0065 mg/kg. The method was successfully applied to 77 samples of extra-European citrus fruit collected in the Italian market during the summer 2007. The results demonstrated that 64% of the investigated citrus samples contained guazatine over the residue limit value of 0.010 mg/kg for not allowed pesticides, evidencing the alarming illicit employ of this fungicide in citrus post-harvest treatments.

<http://www.springerlink.com/content/18556361472r6w0l/fulltext.pdf>