

Pesticide Residue Research Group

European Union Reference Laboratory for Pesticide Residues in Fruits and Vegetables UNIVERSITY OF ALMERÍA, ALMERÍA, SPAIN



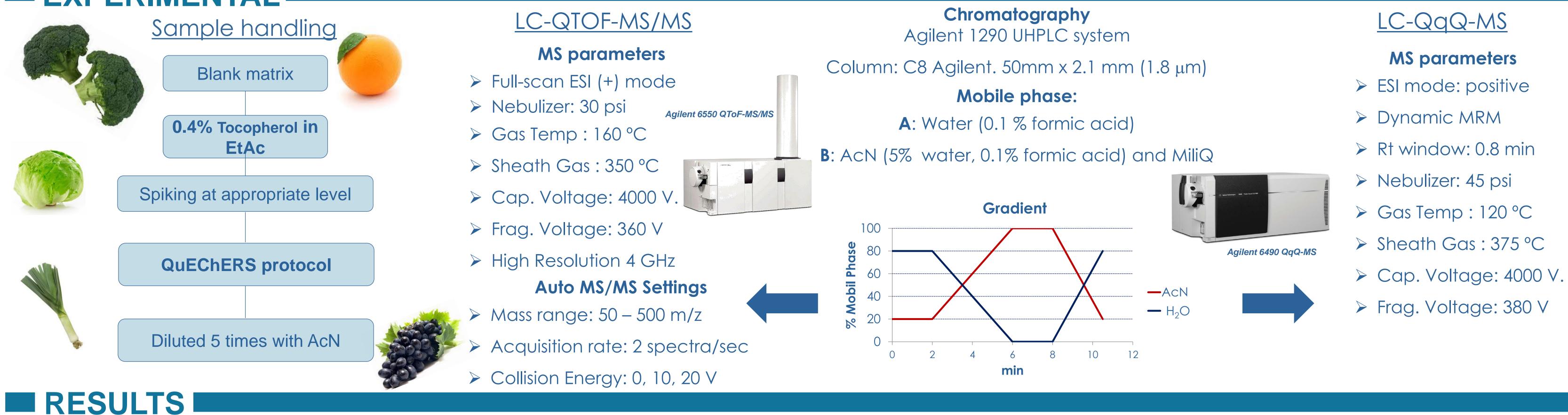
Influence of the antioxidant tocopherol on diafenthiuron recoveries using QuEChERS protocol

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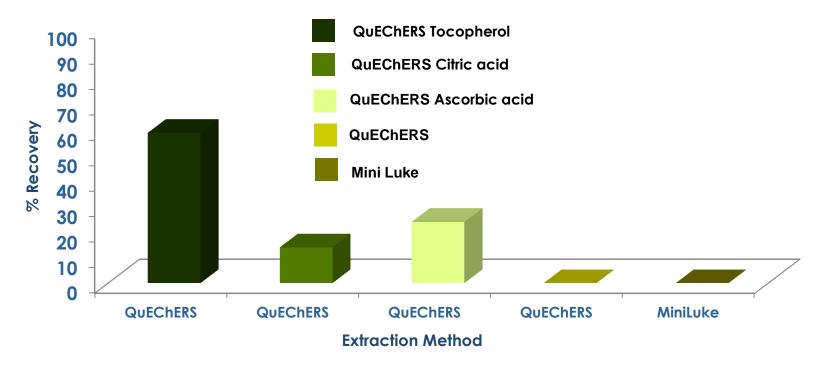
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In general, the implementation of a multiresidue method in the laboratory assumes that a group of pesticides can present some difficulties for the adequate extraction level as consequence of specific interaction with the matrix or degradation processes. In the EUPT-SM07, where the broccoli was selected as matrix, diafenthiuron was only reported by 9% of the laboratories due to the degradation of this compound through the analytical process. The current study seeks to explain and overcome the detection problems for diafenthiuron in the scope of QuEChERS multiresidue method. Three antioxidant agents such as ascorbic acid, citric acid and tocopherol were evaluated in QuEChERS protocol to intend to increase of diafenthiuron recovery in different matrices. In addition, the stabilization of the compound after recoveries was also evaluated. Furthermore, other studies at different pHs (3, 5 and 9) or different solvents were conducted in order to avoid the observed degradation in matrix.

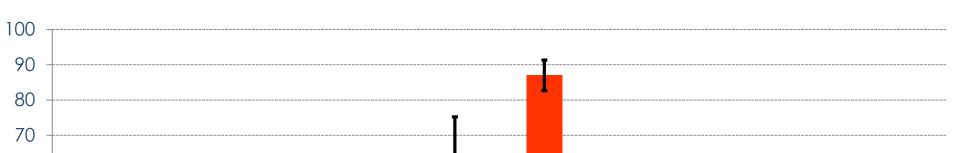
EXPERIMENTAL



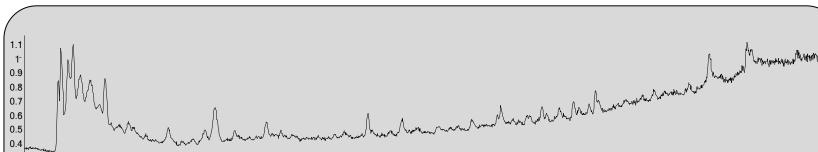
<u>Recovery of diafenthiuron in broccoli</u>



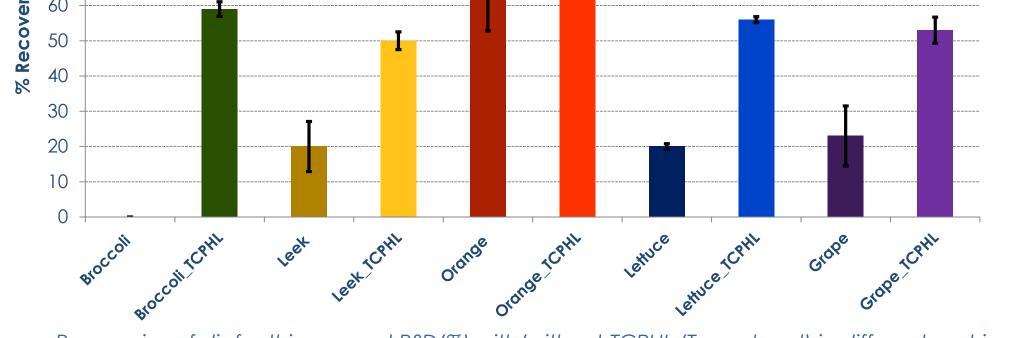
<u>Recovery of diafenthiuron in different matrices</u>



Identification of diafenthiuron and its main transformation products by Mass Structure Correlator (MSC)

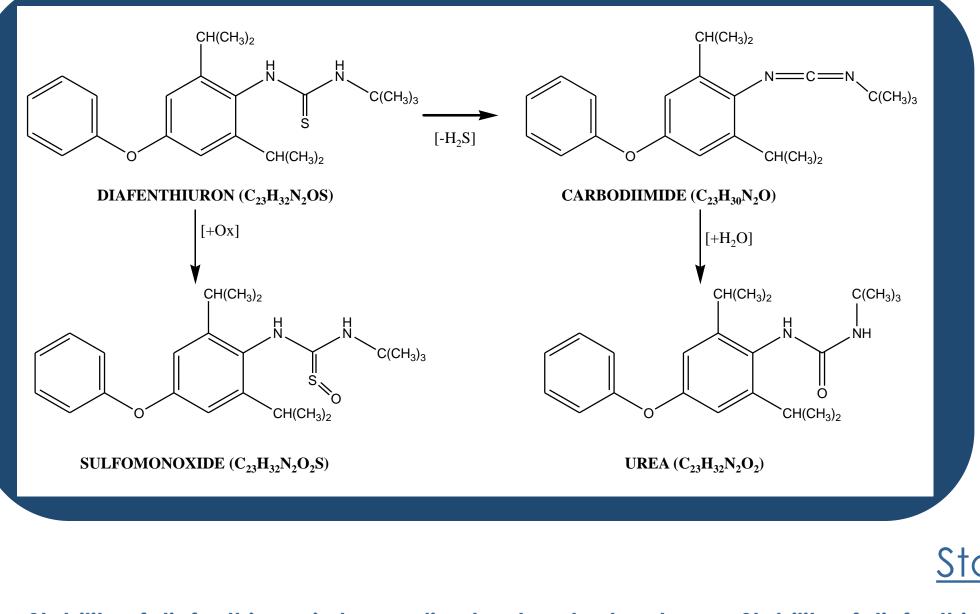


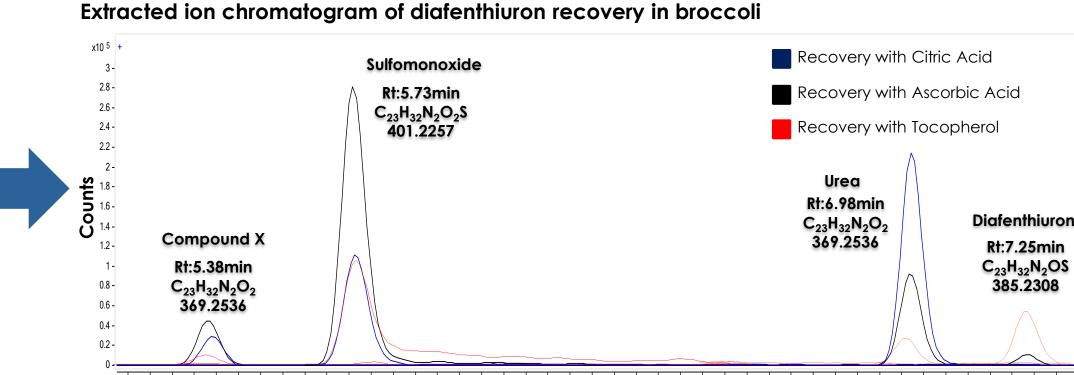
Recoveries of diafenthiuron by QuEChERS and Miniluke extraction methods using different antioxidants



Recoveries of diafenthiuron and RSD(%) with/without TCPHL (Tocopherol) in different matrices

Degradation of diafenthiuron in broccoli

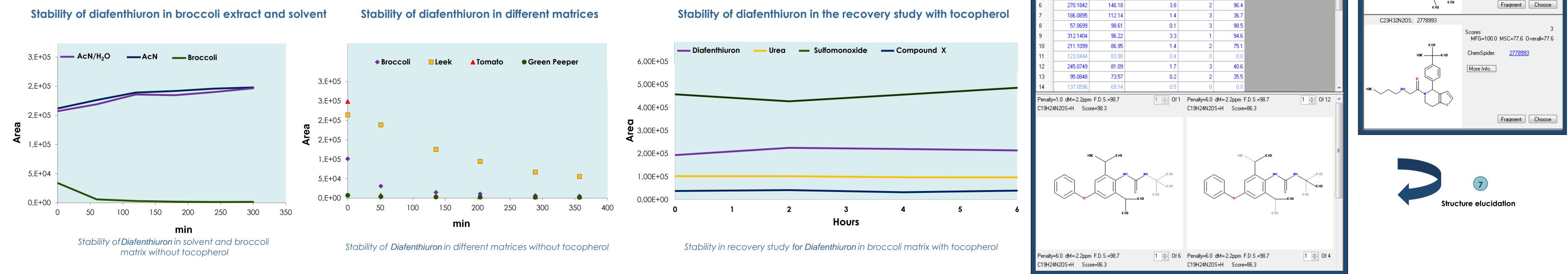


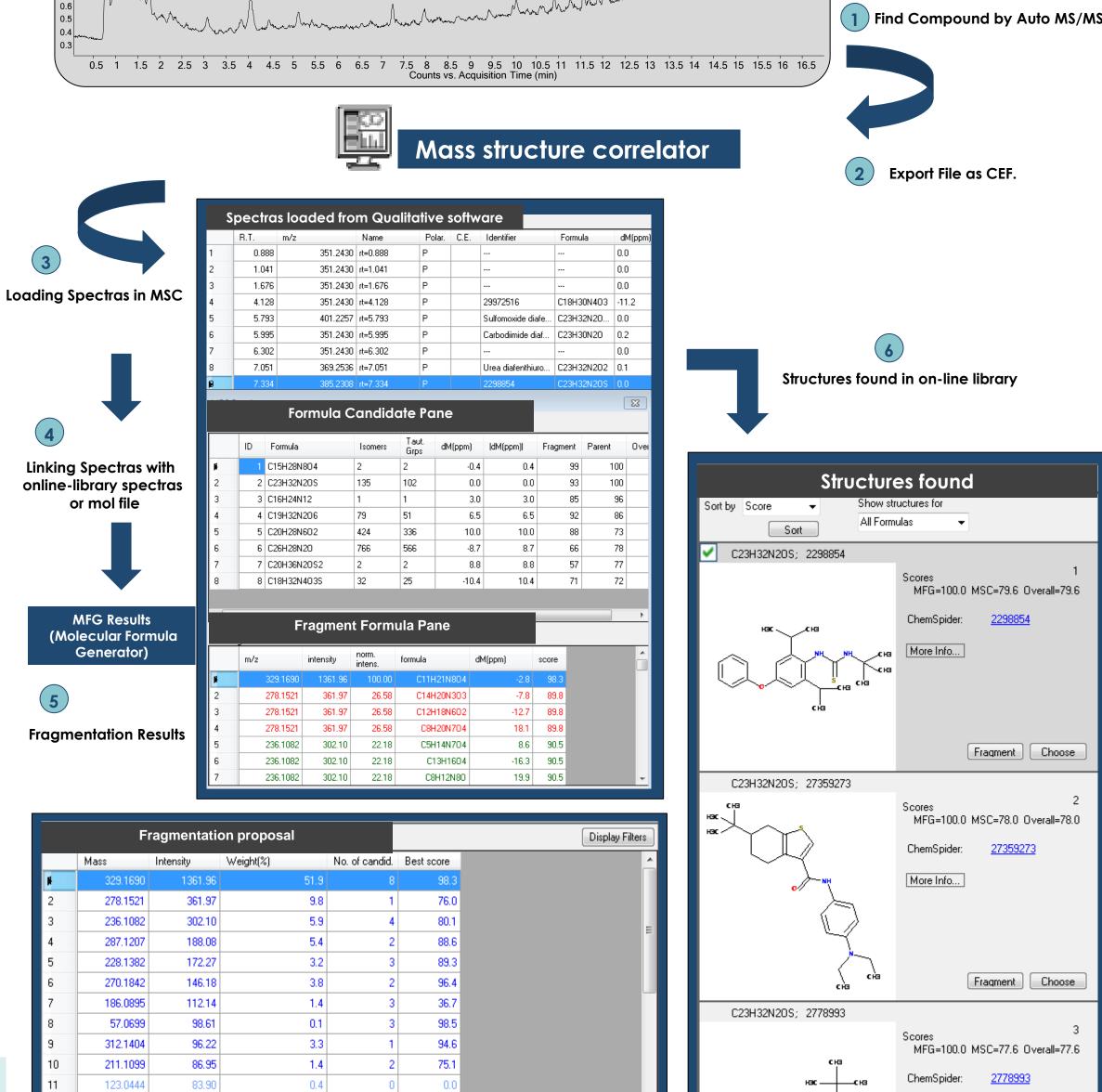


5.2 5.25 5.3 5.35 5.4 5.45 5.5 5.5 5.6 5.65 5.7 5.75 5.8 5.85 5.9 5.95 6 6.05 6.1 6.15 6.2 6.25 6.3 6.35 6.4 6.45 6.5 6.5 6.6 6.65 6.7 6.75 6.8 6.85 6.9 6.95 7 7.05 7.1 7.15 7.2 7.25 7.3 7.35 Acquisition Time(min)

Extracted ion chromatogram of diafenthiuron and its main transformation product in broccoli

Stability of diafenthiuron





1. The addition of 0.4% tocopherol allowed obtaining recoveries around 60% with good reproducibility in broccoli matrix.

2. The recoveries in the rest of matrices were also increased. RSDs (%) lower than the RSDs in recoveries without tocopherol were achieved.

3. The corresponding urea and sulfomonoxide of diafenthiuron were detected in the samples after recoveries. However its carbodiimide was not detected in any evaluated sample.

4. After several pHs studies, improvement in stability was not seen at the pHs evaluated therefore, the degradation of diafenthiuron is not related with pH.

Acknowledgements: The authors acknowledge funding support from the European Commission, DG SANTE, Specific Agreement No. 5 of Framework Partnership Agreement No