

Determination of Kresoxim-Methyl in Water and in Grapes by High-Performance Liquid Chromatography (HPLC) Using Photochemical Induced Fluorescence and Dispersive Liquid-Liquid Microextraction (DLLME)

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ABSTRACT

A high-performance chromatographic method was developed to determine the fungicide kresoxim-methyl. Off-line photochemical derivatization was used to induce the formation of a stable and fluorescent product since the fungicide does not present natural fluorescence. Intense fluorescence at 370/430nm was achieved by treating the analyte in solution at pH 6 to ultraviolet light for 45s. The chromatographic conditions included isocratic elution with 50/ 50% (v/v) acetonitrile/water and the photochemical product appeared at a retention time of 7.2min. The short and long term stabilities of the photoproduct were evaluated and variation of less than 5% was achieved. The limits of detection in water samples and in grapes samples were 0.019mg kg⁻¹ and 0.065mg kg⁻¹ of kresoxim-methyl residue, respectively. The linear response covered three orders of magnitude up to 10.6mg kg⁻¹ of kresoxim-methyl. The robustness was evaluated through a Box–Behnken experimental design showing the insignificance of all factors and their interactions. The potential interference of tebuconazole for the determination of kresoxim-methyl was studied. The use of the dispersive liquid-liquid microextraction (DLLME) allowed recoveries between 80% and 101% depending on concentration with the minimum generation of waste products.

KEYWORDS Dispersive liquid-liquid microextraction (DLLME); high-performance liquid chromatography (HPLC); kresoxim-methyl; photochemical-induced fluorescence; strobirulin