Validation of a gas chromatography method for analysis of bromopropylate in citrus essential oils

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Bromopropylate is a specific acaricide from the benzilate family that is generally used in citrus. This fruit represents the most important crop in the Concordia Department, Entre Ríos Province, Argentina. The objectives of this study were to optimize and validate a gas chromatography method for analysis of bromopropylate in citrus essential oils. Linearity was determined for standard solutions and spiked samples with the following concentrations: 0.01, 0.025, 0.05, 0.075, and 0.1mg/kg. Comparison of the regression curves indicated the occurrence of matrix effect; consequently calibration was performed on spiked samples. $R^2$ values for both samples ($R^2 >0.9999$) showed an excellent linearity for the range studied. Limits of detection and quantitation (LOD and LOQ) were obtained from the calibration curve at low concentration ($n=15$), being 2.2 and 3.5μg/kg, respectively. Precision was evaluated by spiking samples at 0.01, 0.05 and 0.1mg/kg ($n=10$) with relative standard deviations (RSD) of 0.12, 0.01 and 0.01% for essential oil. The main recovery was 97.9%. The method was applied to 5 lots of polished and dewaxed oils from fruits treated with bromopropylate. The amount of fungicide residues observed in the different lots were 46 and 42; 124 and 109; 40 and 35; 78 and 73; and 27 and 25mg/kg, for polished and dewaxed oils, respectively. The partitioning of bromopropylate in the wax phase may be the reason for the slight residue reduction observed in the dewaxed oils. The suggested method was found to be selective, precise, highly accurate and sensitive.