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ACCEPTED MANUSCRIPT

Multi-residue methods for the determination of over four hundred pesticides in solid and liquid high sucrose content matrices by tandem mass spectrometry coupled with gas and liquid chromatograph

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Abstract

For the first time three methods: matrix solid phase dispersion (MSPD), original and modified QuEChERS, with and without clean up step were studied in order to evaluate the extraction efficiency of various classes of pesticides from solid and liquid high sucrose content matrices. Determinations over four hundred pesticides were performed by gas and liquid chromatography with triple quadrupole mass spectrometry (GC/LC/MS/MS) using multiple reaction monitoring. The proposed methods were validated on sugar beets and their technological product beet molasses. In general, the recoveries obtained for the original QuEChERS and MSPD method were lower (<70%) than for the modified QuEChERS without clean up in sugar beet and with clean up in beet molasses. Among these methods, high extraction yields were achieved as recommended in SANCO/12571/2013, with repeatability of 4.4-19.2% and within-laboratory reproducibility of 7.1-18.4% for citrate QuEChERS, whereas greater ruggedness were observed for MSPD. The limit of quantification (LOQ) at (the lowest MRL=0.01 mg kg⁻¹ e.g. for oxamyl) or below (0.005 mg kg⁻¹) the regulatory maximum residue level for the pesticides were achieved. The expanded measurement uncertainty was not higher than 30% for all target analytes. Matrix effects were compared and observed for both matrices at both gas and liquid chromatography. The most compounds showed signal enhancement and it was compensated by using matrix-matched calibration and modified QuEChERS characterized lower matrix effects. The confirmation of

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