

HIGH PRESSURE LIQUID CHROMATOGRAPHIC METHOD FOR ESTIMATION OF DISLODGEABLE RESIDUES OF METHOMYL

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A NEW reverse phase high pressure liquid chromatographic method (HPLC) with Rp 18 column developed for the separation and determination of residues of methomyl (a carbamate insecticide) on vegetables is presented in this note.

The original method advocated¹ for the determination of methomyl residue is an indirect one where methomyl is converted into their oximes by hydrolyzation and then quantified by GLC using a flame photometric detector in the sulphur mode. Reports of large variations between 73 and 100% in recoveries and the mode of indirect determination necessitated an attempt for direct determination. For this a HPLC system with solvent methanol was tried and found useful.

Methanol was selected as the solubility of methomyl was greater in polar solvents. The solvent used for separation of methomyl was methanol/water 70:30 V/V. This mixture was degassed by sonication before use. Crystalline analytical grade standard methomyl at a concentration of 1 mg/ml of methanol was prepared afresh and used for standardization with a flow rate of 0.5 ml/min at the absorbance of 0.05 absorbance unit. Methomyl was found to have a retention time of 6.15 min at 254 nm. Control spiking experiments were made on bhendi and brinjal leaves and fruits with a recovery percentage of 90 and over 80 respectively.

In a field trial on bhendi and brinjal two concentrations of methomyl were sprayed at fruiting stage. Composite samples were collected and the easily removable surface (dislodgeable) residues were washed thoroughly with 100 ml methanol and the volume reduced to 5 ml in a vacuum flash evaporator. The residue was quantified by the integration of peak area using external standard calibration method and reported as mg/kg of fruit and $\mu\text{g}/\text{cm}^2$ for leaf (one side only). Here the clean-up stage was skipped as the extract presented no interfering peaks. The sensitivity range was 1 μg . The results are given in table 1.

Table 1 Dislodgeable residue of methomyl in bhendi and brinjal

Crop	Dosage used kg ai/ha	Interval between treatment and sampling (days)	Residue mg/kg (fruit) $\mu\text{g}/\text{cm}^2$ (leaf)
Bhendi fruit	0.45	0	21.23
		3	BDL
		5	BDL
	0.75	0	42.91
		3	BDL
		5	BDL
Brinjal fruit	0.45	0	5.04
		3	BDL
		7	BDL
	0.75	0	6.05
		4	BDL
		7	BDL
Bhendi leaf	0.75	0	6.48
		3	BDL
		5	BDL
Brinjal leaf	0.75	0	2.03
		4	BDL
		7	BDL

(BDL: Below detectable level)

The data indicate the usefulness of this method for residue analysis of methomyl besides showing that it dissipates very quickly (within two days of spray) and less persistent on vegetables. Thus this chemical falls within the new class of shortlived pesticides and has an edge over the persistent pesticides for use against vegetable insect pests.

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1. Pease, H. L. and Kirkland, J. J., *J. Agric. Food Chem.*, 1968, **16**, 554.