

INFRA-RED SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF CYPERMETHRIN IN TECHNICAL, EMULSIFIABLE CONCENTRATES AND ULV FORMULATIONS

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INTRODUCTION

Cypermethrin or NRDC 149, IUPAC name (RS) – Cyano-3-phenoxybenzyl (IRS) *Cis, trans*-3-(2, 2-dichlorovinyl)-2, 2-dimethyl cyclopropanecarboxylate is a synthetic insecticidal pyrethroid [1] and has been assessed to be the most promising broad-spectrum insecticide for agricultural use, owing to its extraordinarily high potency and sufficient field persistency.

Cypermethrin, under the trade name Ripcord, (Shell) and Cymbush (ICI), is being used extensively on cotton in Pakistan, while three more brand products, i.e. Arrivo (FMC), Sherpa (M&B) and Nurelle (Dow) are undergoing the process of registration.

Two standard GLC and HPLC procedures for the analysis of this product are available. The GLC/FID methods involves the determination of *total toxicant* (cypermethrin) by measuring (in technical and formulated materials, [2,3,4] the peak areas for the internal standard and the insecticide by a data handling system. In the HPLC method [5], use of a UV detector is involved. As the above facilities were not available in the laboratory, efforts were concentrated to employ the available facility of infrared spectrophotometry which resulted in the development of the new analytical procedure being reported.

EXPERIMENTAL

IR spectrophotometer, Beckman Model 4240 with sodium chloride cavity matched cells of 0.1mm path length were used. Cypermethrin 95.8% analytical standard was obtained from FMC Corporation, Middleport, NY 14105, USA. Standard solution of seven concentration of analytical grade cypermethrin in between 0.1g/10ml to 0.4g/10ml representing a concentration range of 1% to 4% with all the five aforementioned brand products were prepared. For obtaining accurate quantitative results on the samples of

cypermethrin, a concentration-vs-absorbance calibration curves were constructed from analytical standard solutions prepared with the concentration of 1, 1.5, 2.0, 2.5, 3.0, 3.5 and 4.0% cypermethrin in chloroform and dioxane respectively. In this way seven points were determined in respect of chloroform and dioxane solvent with cypermethrin for an accurate calibration curve (Figs. 1-3). After running an IR spectrum of a cypermethrin sample material, the absorbance of the peak at 1745 cm^{-1} is calculated according to Beer's Law. Thereafter the standard calibration curve is used for the determination of the actual cypermethrin

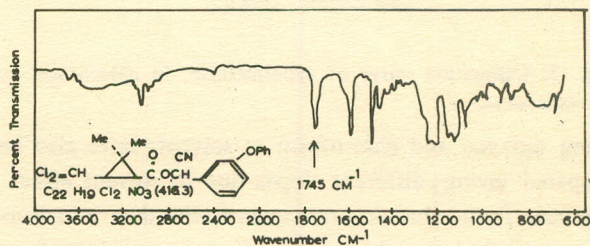


Fig. 1. IR spectrum of cypermethrin (Model: IR4240) (1). Cell NaCl windows (2). Path: 0.1 mm. (3). Gain: 1.47 (4). Solvent: Chloroform (5). Concentration: 3%. (6). Period/Speed: 2 sec/15 cm/min.

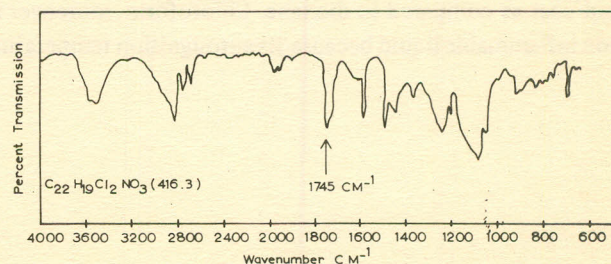


Fig. 2. IR spectrum of cypermethrin (Model: IR4240) (1). Cell NaCl windows (2). Path: 0.1 mm. (3). Gain: 1.47 (4). Solvent: Chloroform (5). Concentration: 3%. (6). Period/Speed: 2 sec/15 cm/min.

concentration (Fig. 3). Technical cypermethrin and its formulated forms were successfully analysed by the present IR method. This method is applicable to the gross determination of cypermethrin samples but will not give any information regarding individual isomer contents. Typical IR spectra of cypermethrin analytical standard in dioxane and chloroform (Figs. 1-2) are given. The calibration curves (Fig. 3) for a Beckman Model 4240 IR spectrophotometer

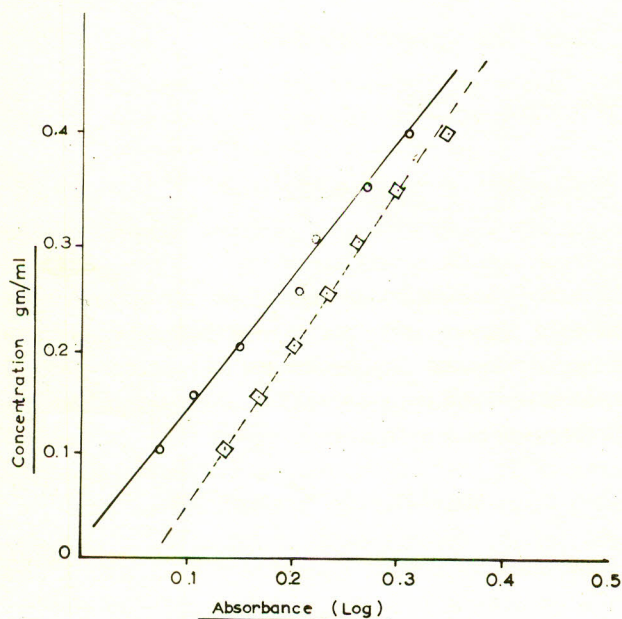


Fig. 3. Calibration curve of cypermethrin. In chloroform -○- in dioxane -□- - - -

using dioxane and chloroform as solvents have also been prepared giving different slopes due to solvent effect. It has been found that accurate quantitative determination of cypermethrin is possible both in dioxane and chloroform if the analytical conditions of the instruments as stated above are meticulously maintained. In our situation, however, the use of chloroform as solvent for absorption spectrum of cypermethrine would be preferable in view of its availability and cost as compared to dioxane. Chloroform, moreover is, non inflammable liquid because its auto-ignition temperature

is above 1000° [6]. The dioxane is highly inflammable as it forms explosive mixture, with air at 20° [6]. The method has been successfully used in the laboratory for the quality checks of various formulations of following brands of cypermethrin:—

| Brand product (Formulator's name) | Formulation (w/v) |
|--------------------------------------|-----------------------|
| Arrivo (FMC) | 10% EC |
| Nurelle (Dow Chemical) | 25% EC |
| Cymbush (ICI) | 10% EC |
| Ripcord (Shell Chem. Co.) | 10% EC |
| Sherpa (M&B) | a) 5% EC b) 5% ULV |

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