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A NOTE ON THE INFRARED SPECTROPHOTOMETRIC ASSAY OF PHOSALONE ALONE OR IN COMBINATION WITH DDT

SHAMIM FARHAT and M. M. H. BAIG

Federal Pesticides Laboratory, Agricultural Research Council, Malir Halt, Karachi

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Abstract. An infrared spectroscopic method for the estimation of phosalone alone, in formulation or in combination with DDT has been developed. In its emulsifiable formulation, phosalone is assayed directed by dissolving in chloroform and scanning in the region of 5.0–6.5 μ with its main peak at 5.6 μ, due to the carbonyl group present in its molecule. In case of an emulsifiable formulation of phosalone in combination with DDT, a column cleanup was necessary to separate DDT before IR estimation of phosalone could be made.

Phosalone (S-6-chloro-2-oxbenzoxazolin-3-yl methyl 0, 0-diethyl phosphorodithioate) is a non-systemic organophosphate insecticide and acaricide which is used on several agricultural crops. It is available as emulsifiable concentrates containing phosalone alone or in mixture with DDT.

UV spectroscopic, chemical and gas liquid chromatographic methods are available for the assay of phosalone, but so far no analytical procedure has been described for IR spectroscopic determination of this insecticide which is the subject in this contribution.

Material and Method

(i) IR spectrophotometer, DB model Beckman 5-A, with NaCl cells having path length of 0.3 mm, (ii) chromatographic column, 10 × 1.5 cm, glass stoppered, (iii) n-hexane, redistilled, (iv) chloroform, spectral grade 11 (v) aluminium oxide, activity grade No. 1 for column chromatography obtained from M. Woelm Eschwege, West Germany, (vi) phosalone 100% pure and p' p' DDT-70% obtained from May & Baker Limited, U. K.

Preparation of Standard Calibration Curve. Solutions of 10 concentrations of pure grade phosalone in between 1 mg/ml to 10 mg/ml in chloroform representing a concentration range of 0.1 to 1% were prepared. The spectrophotometer was adjusted to optimum setting for gain, slit width, response, speed, etc. Spectra of each concentration were recorded over the range from 5.0 to 6.5 μ in duplicate.

For each of the scans, a line was drawn between the base line points, 5.4μ to 5.9μ, a perpendicular was drawn from the zero radiation line through the absorption peak to the base line, and the distance was measured from the zero line to peak P and to base line PO (Fig. 1). The absorbance was calculated as log (PO/P) and a standard calibration curve of transmittance against percent composition (Fig. 2) was plotted according to Beer's Law. The curve is linear in the range of 0.1 to 0.7% solution.

Analysis of Phosalone Formulation. A sample was prepared in chloroform containing 2 to 6 mg

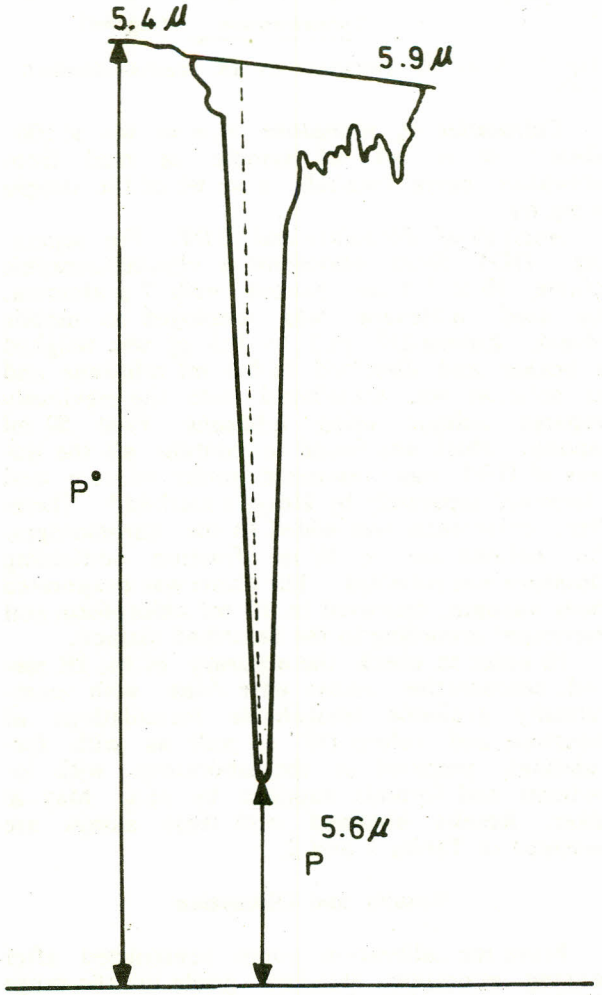

Fig. 1. IR spectra of phosalone—base line construction.

active ingredient/ml. The calibrated liquid absorbance cell used in plotting a standard curve of phosalone was filled with sample solution using same instrument settings as for calibration, and the sample solution was scanned in duplicate over 5.0—6.5 μ. The corresponding mg of sample/ml was read from calibration curve (Fig. 2).

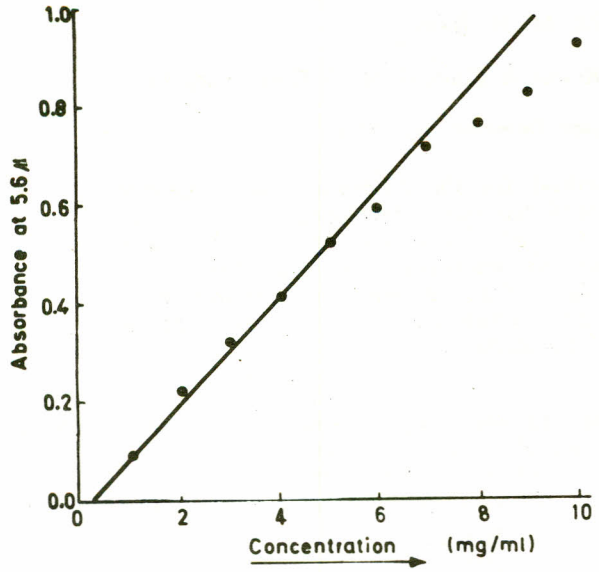

Fig. 2. Assay of phosalone by IR spectra—standard curve.

Calculation. % phosalone w/w = w/s × 100 where : w = wt. of sample as read from calibration curve (mg/ml), s = wt of the sample in mg/ml.

Analysis of Phosalone and DDT. For separating DDT from phosalone a chromatographic column 10 × 1.5 cm, packed with 7 g alumina, was used. n-Hexane was employed as mobile solvent. Zolone-DT (0.3 to 0.5 g) was weighed in beaker and dissolved in 0.5 ml n-hexane and the solution was transferred onto the previously prepared column using n-hexane. First 50 ml fraction, which was found to contain all the isomers of DDT, was evaporated under vacuum and determined separately by Heagy's method.[1] Thereafter, chloroform was added to the chromatographic column and a 50 ml fraction containing phosalone was collected. The eluate was evaporated under vacuum, dissolved in 10 ml chloroform and determined according to the described method.

In order to check the accuracy of the IR method, comparative assays were done with commercially available emulsifiable formulations of phosalone and zolone-DT as well as with formulations prepared in the laboratory with ingredients and formula supplied by M/s. May & Baker. Results obtained with these assays are presented in Tables 1 and 2.

Results and Discussion

From the calibration curve constructed after assaying phosalone standard grade in the range of 5.0 μ—6.5 μ (Fig. 2) it is evident that this insecticide may be estimated by using absorption in the IR region in the range of 0.1—0.7% phosalone due to presence of carbonyl group in its molecule.[2] The method is applicable to both commercial and laboratory prepared formulations of phosalone 33% E. C. for which the technique gave good reproducibility shown by standards deviation of ± 0.07 and 0.05 respectively. A method has also been described for separating DDT and phosalone from their mixed emulsifiable formulations available commercially as well as from their identical laboratory prepared formulations. The results of assay in this case provide standards deviation of ± 0.20 — 0.31 for phosalone while standards deviation of ± 0.50 and 0.20 were observed in the case of laboratory prepared and the commercial formulations of DDT respectively. By comparison, the standards deviation in respect of phosalone is a bit higher in the case of mixed formulation with DDT (± 0.20—0.31) than when phosalone alone was present in the formulation (± 0.05 — 0.07). It is possible that inspite of use of a cleanup procedure for separating both DDT and phosalone from a mixture, some interfering substances are still left behind

TABLE 1. REPETITIVE ANALYSIS OF TWO PHOSALONE FORMULATIONS

Commercial 33% E.C. (w/v) (%)	Laboratory prepared 33% E. C. (w/v) (%)
32.98	33.27
33.03	32.99
33.28	32.95
33.02	33.13
33.48	32.94
32.86	32.91
32.92	32.92
33.35	32.88
32.95	32.84
32.92	32.95
Average results % 33.08	32.98
Standard deviation ±0.07	±0.05

TABLE 2. REPETITIVE ANALYSIS OF TWO PHOSALONE + DDT FORMULATIONS.

Commercial formulation (phosalone, 15% w/v + DDT, 30% w/v)		Experimental formulation (phosalone, 15% w/v + DDT, 30% w/v)	
Phosalone (%)	DDT (%)	Phosalone (%)	DDT (%)
14.73	29.68	14.95	28.59
14.35	30.02	15.03	29.75
13.95	29.83	14.24	29.34
14.10	30.13	15.02	29.44
15.02	28.40	13.90	29.35
Average 14.43	29.61	14.63	29.30
Standard Deviation ±0.20	±0.50	±0.31	±0.20

and are responsible for this phenomenon. There is also a possibility that the small quantitative losses that occur due to the use of a cleanup procedure cause these deviations. The standards deviation in this case is, however, insignificant in view of the fact that the FAO allow[3] a range of tolerance of ± 6% on methods of assay used in testing formulations containing toxicants between 10—25%.

References

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