

A COMPARATIVE STUDY OF ANALYSIS OF TECHNICAL GRADE FENITROTHION BY THREE DIFFERENT TECHNIQUES

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Abstract. Gas liquid chromatography and IR spectrophotometry were compared with WHO recommended chemical method for the quality control of technical grade Fenitrothion insecticide. The chemical method involves a diazonium reaction which is time-consuming. It is evident from the comparative data that results of the three methods are in close agreement with one another. GLC and IR spectrophotometric methods which are far more convenient and less time-consuming, may be reliably employed for the analysis of Fenitrothion insecticide.

Fenitrothion, (*O,O*-dimethyl 3-methyl-4-nitrophenylphosphorothioate), is an effective contact insecticide and a selective acaricide, having a relatively low mammalian toxicity.¹ It is widely used in Pakistan for the control of a variety of pests on cereals, sugarcane and many other crops. Due to its increased usage, the quality control of the product has become important.

A chemical method employing a diazo-reaction² is the official recommended procedure for the determination of fenitrothion insecticide. It is tedious and time consuming. Studies were, therefore, undertaken to comparatively evaluate this method with two other methods, namely, gas liquid chromatography (GLC) and infra red spectrophotometry for the analysis of this insecticide so as to select a quick and a reliable method to enable determination of a large number of samples in a short time. Results obtained are presented in this communication.

Experimental and Results

Chemical Method. According to the chemical method employing a diazonium reaction,² 0.75 g of technical grade fenitrothion was dissolved in 100 ml ether and 4-nitro-*m*-cresol was extracted with 1% w/v sodium carbonate. The ether layer was treated with zinc and acetic acid—hydrochloric acid mixture and the amino group formed was titrated with sodium nitrate solution. Starch iodide paper was employed as an external indicator in the titration.

GLC Determination. Pye Panchromatograph equipped with an electron capture detector was used for GLC determination of fenitrothion.³

Operating Conditions. A glass column 30 cm long × 4 mm i.d. packed with 5% QF-1 on 80–100 mesh Phasesep-W (Phase Separations Ltd., Cheshire, England), Temperatures: column oven 150 ; detector oven 175° ; detector voltage 30 V; electrometer setting 10⁻⁹ amp full scale ; nitrogen (carrier gas) flow rate 50 ml/min, Honeywell recorder 1 mV, chart speed 125 mm/hr.

Five microlitre solutions containing 60 nm (technical grade) samples and standard (99.5% purity)

in hexane were injected on to the GLC column with a 10 μ l syringe. The limit of detection was in the range of 0.02–0.04 μ g. Percentage of toxicant was calculated by comparing the peak heights of samples with those of the standard insecticide.

IR Spectrophotometric Determination. Beckman IR-5A spectrophotometer with 0.4 mm matched cells was used for the determination of the insecticide.⁴ A standard calibration curve was prepared with fenitrothion (99.5% purity) by plotting absorbance against concentration in mg/ml (Fig. 1). The samples were prepared in 10 ml of carbon disulphide and each sample was scanned thrice in the range of 10 and 11 μ . The amount of the insecticide present therein was read from the calibration curve.

Five different samples of fenitrothion (technical grade) were analysed by the above described methods and comparative results, with standard error (SE), are presented in Table 1.

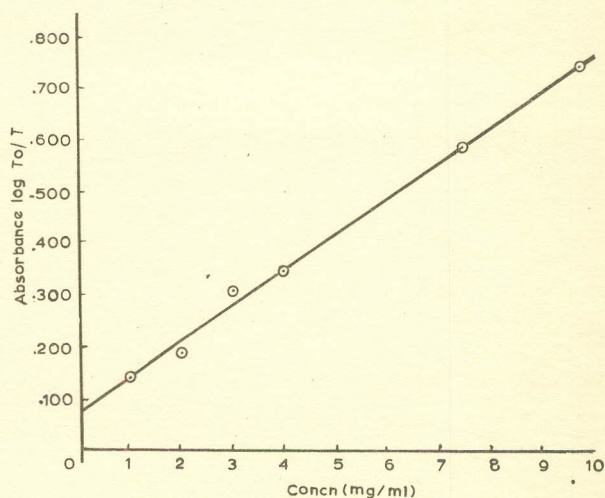


Fig. 1.

Conclusion

By comparing GLC and the IR spectrophotometric methods with the official diazonium reaction

TABLE 1. RESULTS OF COMPARATIVE ASSAYS OF TECHNICAL GRADE FENITROTHION WITH THREE DIFFERENT ANALYSIS METHODS.

Sample No.	Chemical assay	Gas liquid chromatography	IR
1	97.57 ± 0.33	98.1 ± 0.71	97.5 ± 1.50
2	96.98 ± 1.29	100.4 ± 0.64	101.15 ± 1.95
3	101.75 ± 0.65	96.72 ± 0.74	97.8 ± 1.29
4	98.63 ± 0.38	100.23 ± 0.78	97.65 ± 1.55
5	101.15 ± 0.65	97.4 ± 1.66	97.5 ± 0.50

Each figure is the average of three replicates.

method recommended by the WHO, it can be inferred that the results of all the three methods are in close agreement with each other. It may be noted from the above table that SE's in respect of the 3 methods tested are in the order of chemical assay < GLC < IR spectrophotometry. It is perhaps due to the higher sensitivities of the GLC and IR spectrophotometry than the chemical assay. No work has been done on the possibility of contaminants which might interfere with each of the three methods.

It is, therefore, evident that GLC and IR spectrophotometric methods which are far more convenient and less time consuming may as well be

employed for quality control purposes.

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