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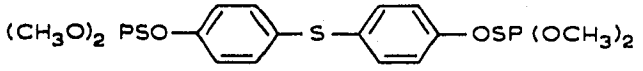
Alkali-flame gas chromatography of Abate*

The insecticide Abate® (O,O,O′,O′-tetramethyl-O,O′-thiodi-*p*-phenylene phosphorothioate) (I) is one of the newer chemicals proven to be effective in the control of mosquito larvae[1,2]. A number of analytical methods for residue determinations of this chemical have already been published, the majority of which use the gas chromatograph. Flame ionization[3], electron capture[4], and flame photometric detectors[4–6] have all been employed in these chromatographic procedures. Because of the limited sensitivity of flame ionization and the loss of specificity of electron capture, the flame photometric detector, operated in either the phosphorus or sulfur mode, appears to be the detector of choice. DALE AND MILES[4] and BOWMAN *et al.*[5] report levels of sensitivity at 2–5 ng when operating in the phosphorus mode and 30–50 ng when using the sulfur mode. However, both indicate the necessity of adapting a water cooling system to the detector to prevent damage while operating at the high temperatures required. A current model of the flame photometric detector is now available which is designed to operate at higher temperatures and does not require a water cooling system.



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In addition, BOWMAN *et al.*[5] found it difficult to maintain the conditioned state of the column, necessitating the injection of 50 ng or more of a standard solution alternately with the unknowns being analyzed. However, DALE AND MILES[4], using an XE-60 column of 2-in. length, had no problem in maintaining the conditioned state. SHAFIK[6] developed the technique of hydrolysis of Abate to 4,4′-thiodiphenol and treating this compound with trimethylchlorosilane and hexamethyldisilazane to form a silylated derivative. This technique allows the conditioned state of the column to be easily maintained with operation of the detector at a lower temperature (thus omitting the use of a cooling system) and produces a level of sensitivity of 5–10 ng when operating in the sulfur mode.

Since many laboratories may lack the photometric detector, the following investigation was made to develop a rapid, specific, sensitive method of analysis for Abate using the relatively inexpensive alkali-flame detector.

Materials and methods

A Varian Aerograph, Model 1700, equipped with an alkali-flame detector and an aluminum column, 1/8 in. O.D. × 10 in., packed with 2.5% E-301 + 0.25% EPON 1001 on 80/100 mesh Gas-Chrom W (AW-DMCS)HP was used.

Operating parameters were: injector, column and detector temperature 235°; flow rates, nitrogen 50 ml/min, air 120 ml/min, hydrogen 20 ml/min; electrometer

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attenuation, $2 \cdot 10^{-10}$ A/mV. Air and nitrogen were supplied by pressure-regulated cylinders, and hydrogen was produced from an Elhygen Hydrogen Generator. All Abate standards were prepared in diethyl ether.

The column was preconditioned for 24 h, after which five injections of 1 μg/μl were made at intervals of 10 min each. The column was considered ready for use when injections of 20 ng every 10 min produced identical responses of 10% scale. Retention time for the chemical was 3 min.

Results

We encountered the same problem of column conditioning as previously reported[5] and, in the manner suggested by these authors, alternating injections of the unknowns with a 20 ng standard eliminated the problem. The response of the detector was linear to 200 ng. Water samples, fortified with Abate to produce concentrations of 1.0, 0.1 and 0.01 p.p.m., were extracted by the method of DALE AND MILES[4] and were brought to a final volume of 1 ml in diethyl ether. Recoveries were 97% as determined by the above chromatographic procedure.

The increased sensitivity and specificity of the alkali-flame detector for phosphorus makes it more acceptable for analysis of such chemical containing compounds than either the flame ionization or electron capture detectors. The results of this investigation provide the analyst with a method which is rapid, sensitive, and specific, and closely approaches the results obtainable with the flame photometric detector.

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