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Rapid Liquid Chromatographic Analysis of Chlorothalonil in Fresh Produce Using Photoconductivity and UV Detectors in Tandem

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Liquid chromatography with a photoconductivity (PC) detector connected in tandem with a UV detector was evaluated for determining chlorothalonil residues in extracts of strawberries, tomatoes, and sweet and sour cherries. Samples were prepared by a rapid, multiresidue screening procedure consisting of acetone extraction, partitioning into petroleum ether plus methylene chloride, and concentrating. Sensitivity and selectivity of the PC detector were found adequate for reliable quantitation of residues at levels both above and well below those of current regulatory concern. UV detection was more prone to interferences at lower levels but served as a useful monitor of the chromatographic system and provided additional data. Recoveries were essentially complete from samples fortified at 0.05–5 ppm. Results for samples with field-incurred residues were in reasonable agreement with those previously determined by gas chromatography.

Chlorothalonil is a broad-spectrum fuungicide used on fruits, vegetables, and other agricultural products. Recovery of chlorothalonil by the rapid, multiresidue method of Luke et al. (1981), using gas chromatography (GC) for determination, has been reported. This widely used method, which eliminates adsorption chromatography cleanup (i.e., Florisil) for relatively polar compounds such as chlorothalonil, results in the injection of considerable coextracted sample material. Selective detectors are therefore required for reliable quantitation of residues in these crude extracts. Variable GC responses due to sample matrix effects on chromatography are commonly encountered, however.

In the present study, liquid chromatography (LC) with photoconductivity (PC) detection was investigated for the

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determination of chlorothalonil residues in selected fruits and vegetable extracts prepared by the method of Luke et al. The PC detector described by Popovich et al. (1979) offers selective detection for certain halogen, nitrogen, and sulfur compounds that will undergo postcolumn photolysis reaction to form stable ionic species then measured by conductivity. The PC detector has been applied to residues of several herbicides and fungicides in soil, water, and plant materials as reported by Büttler and Hörmann (1981), Slates (1983), Zahnow (1982, 1985–1987), Walters (1983), Walters and Gilvydis (1983), and Walters et al. (1984).

These applications included more extensive sample cleanup to eliminate interfering coextractives, however. In an early study of the PC detector by Walters (1983), standard chlorothalonil was found to give a good response on the PC detector. Hence, the suitability of this detector for chlorothalonil determinations in uncleaned Luke et al. method extracts appeared worthy of investigation. Findings of significant chlorothalonil residues in both imported strawberries and domestic sour cherries by GC analysis prompted further interest in the evaluation of this technique as an alternative or confirmatory quantitation method. Since UV response at 232 nm (λ_{max}) was also very good, a UV detector was connected in tandem ahead of the PC detector for the sake of comparison.

EXPERIMENTAL SECTION

Apparatus. LC equipment consisted of a Perkin-Elmer Series 3B reciprocating pump, a Waters WISP 710B autosampler, a Kratos Spectroflow 773 variable-wavelength absorbance detector set at 232 nm, and a Tracor Model 965 photoconductivity detector equipped with a 254-nm mercury lamp and with solvent flow splitter adjusted to a 1:1 split between the analytical and reference compartments. The PC detector was modified as follows: The photolysis reaction–conductivity cell assembly was detached from the interior of the instrument and placed in a Du Pont column oven located adjacent to the PC detector; wiring was strung through an orifice in the oven wall accessible by removal of a panel at the front of the oven; the orifice was then insulated with glass wool and sealed with duct tape; the oven, which also contained the HPLC column, was operated at 35 °C. The detectors were connected to recorders operated at 10-mV full-scale output. The columns used were Du Pont Zorbax TMS and Zorbax C-8, both 25 cm × 4.6 mm (i.d.), with attached cartridge guard columns containing similar material.

Reagents. Mobile phases used were prepared from methanol and water that had been previously circulated through a 1:1 mixture of cation- and anion-exchange resins (supplied with the PC detector). Each liter of solvent was circulated through the resins for 24 h at 4 mL/min to ensure complete and consistent deionization before use. Methanol used was HPLC grade, and water was obtained from a Millipore Milli-Q system.

Chlorothalonil standard was obtained from the U.S. Environmental Protection Agency repository (Research Triangle Park, NC) and was dissolved and diluted in 2-propanol (which was previously dried over anhydrous sodium sulfate) to a concentration of 1 μg/mL for HPLC.

Sample Preparation. Each sample was homogenized in a Waring blender, and a portion was then treated as described in the method of Luke et al. (1981): 100 g of homogenate was blended with 200 mL of acetone, centrifuged, and filtered. An 80-mL aliquot was extracted with a mixture of 100 mL of petroleum ether and 100 mL of methylene chloride, and the layers were separated. The aqueous phase was saturated with 7 g of NaCl and extracted with two 100-mL portions of methylene chloride.

Table I. Results for Strawberries Using LC-PC with TMS Column and Comparison with GC Result

sample	chlorothalonil aded,[a] ppm	recovery, % (LC-PC)
strawberry control	0.051	94, 102
	0.101	107, 109
	0.506	102, 106
	residue found, ppm	
	LC-PC	GC-HEC
strawberries with field-incurred residue	0.346	0.394

[a] Fortifications made in duplicate.

The organic solvent extracts were dried by passage through a column of anhydrous sodium sulfate and then concentrated to about 2 mL over steam in a Kuderna-Danish concentrator fitted with a Snyder column (Model K-570000; Kontes, Vineland, NJ). The solvent was evaporated completely by immersing the concentrator tube in warm water (30 °C) and applying a gentle stream of nitrogen. The residue was dissolved and diluted in 2-propanol (previously dried over anhydrous sodium sulfate) to a concentration suitable for LC determination.

RESULTS AND DISCUSSION

Early experiments were done on a Zorbax TMS column, which is recommended by the manufacturer for reversed-phase separations of polar compounds. A sample of imported strawberries, in which a significant chlorothalonil residue had been previously found by GC analysis of a Luke et al. method extract, was selected for initial evaluation. A strawberry control was also obtained for standard recovery studies. Results of the quantitation of the field-incurred residue and of standard recoveries from the control sample fortified at three levels in duplicate are shown in Table I. Standard recoveries were complete with little variation between duplicate determinations. LC–PC results for the field-incurred residue are in reasonable agreement with the GC result obtained by another laboratory using a Tracor Hall electrolytic conductivity (HEC) detector in the halogen mode.

Typical LC-PC and LC-UV chromatograms for strawberry extract are shown in Figure 1. Under the conditions listed, complete resolution of chlorothalonil from sample background was apparently achieved by the PC detector. The UV detector chromatogram shows some interference, however, which precluded reliable quantitation.

It became evident in a fairly short time that the TMS LC column was deteriorating, as retention capacity decreased significantly. Several other TMS columns available in our laboratory, which had been used for purposes other than pesticide residue analyses, were tested and found to be similarly deteriorated. We therefore assumed that the instability was inherent to the column packing and not a result of our work and informed the manufacturer of our experience.

Subsequently, we investigated Zorbax CN and C-8 columns. The C-8 column was more retentive and provided better resolution of chlorothalonil from sample background. However, higher methanol strength and column temperatures were required to achieve elution in a practical time. Increasing the flow rate was undesirable because PC response diminishes as a result of the decreased photolysis reaction time. Methanol strength greater than about 60% jeopardized resolution of chlorothalonil from earlier eluting sample background. At this point, the photolysis reaction–conductivity cell assembly was placed in the column oven along with the column as

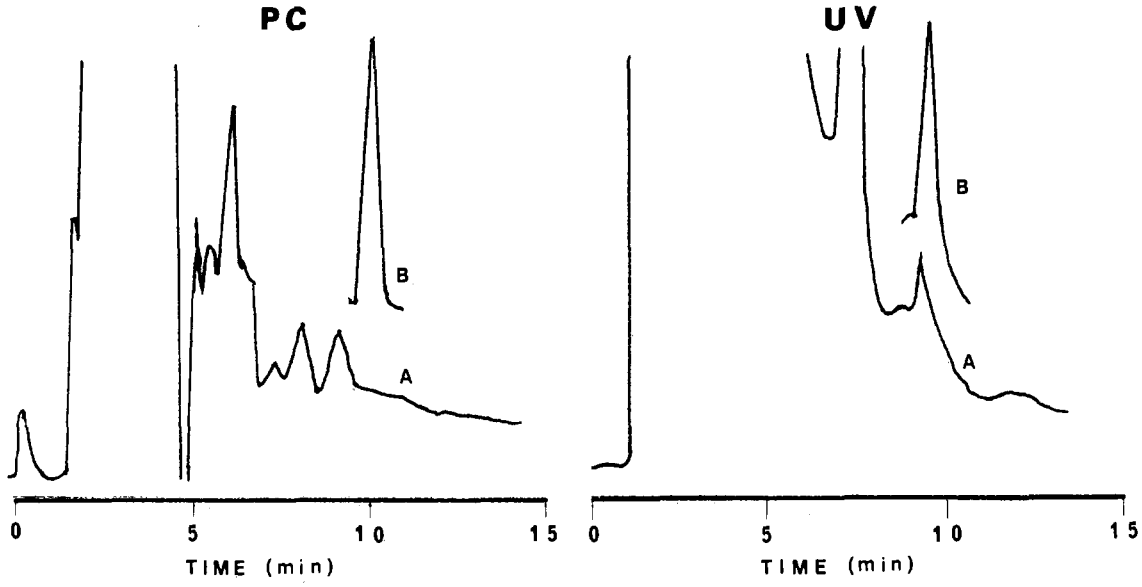

Figure 1. Chromatograms of strawberry extract from TMS column: (A) strawberry control; (B) chlorothalonil peak for fortification level of 0.1 ppm, or 3 ng in 30 mg of sample equivalent injected. Conditions: detector sensitivities, 10 × 5 (PC) and 0.01 AUFS (UV); mobile phase, 50% methanol in water at 1 mL/min flow rate; temperature, ambient (22 °C).

described under Apparatus. This eliminated base-line drift and response variability resulting from ambient temperature changes and provided some increase in conductivity response as well. However, the operating temperature was limited to <40 °C because of a tendency for bubble formation in the conductivity cells at higher temperatures.

The C-8 column was subsequently used for determining chlorothalonil in a sample of domestic sour cherries that had been found by GC-electron capture (GC-EC) detection to contain a significant level of field-incurred chlorothalonil. Control samples of both sweet and sour cherries and of tomatoes, none of which contained any detectable chlorothalonil residue, were also obtained for trial. Official U.S. tolerances of 5 ppm for tomatoes and 0.5 ppm for both sweet and sour cherries have been established. (No such tolerance currently exists for strawberries, however.) The control samples were fortified in duplicate at the respective tolerance levels and at a level substantially lower (i.e., 0.2 ppm) to test the quantitative capability of the method.

Analytical results for tomatoes and cherries are shown in Table II. Recoveries of standard chlorothalonil from fortified samples ranged from 86 to 103% with good agreement between duplicates and between HPLC-PC and HPLC-UV results. Field-incurred residue levels found in sour cherries by LC-PC and LC-UV were also in good agreement and were reasonably consistent with GC-EC results.

Typical LC chromatograms obtained from the C-8 column are shown in Figure 2. Tomato extract chromatograms contained considerable sample background that interfered in the LC-UV quantitation of chlorothalonil at the 0.2 ppm fortification level. Tomato extracts also produced several late-eluting peaks in the LC-UV chromatograms to which the PC detector did not respond because of its greater selectivity. The strawberry samples analyzed earlier on the TMS column were no longer available for evaluation on the C-8 column.

The C-8 column showed no deterioration. Chlorothalonil retention time and column efficiency remained virtually unchanged after extensive usage. Reproducibility of chlorothalonil peak responses was very good; e.g., the relative standard deviations of peak heights for seven consecutive standard injections bracketing six sample injections (sour cherries) were 1.8% and 1.3% for PC and

Table II. Results for Tomatoes and Cherries (Sweet and Sour) Using LC-PC and LC-UV with C-8 Column and Comparison with GC Result

control sample	chlorothalonil added,[a] ppm	recovery, %	
		LC-PC	LC-UV
tomato	0.205	101, 103	interference
	5.13	98, 102	95, 103
sweet cherry	0.205	96, 100	98, 98
	0.513	91, 97	91, 97
sour cherry	0.205	91, 90	91, 86
	0.513	95, 96	95, 97

	residue found, ppm		
	LC-PC	LC-UV	GC-EC
sour cherries with field-incurred residue	0.970	0.960	0.892

[a] Fortifications made in duplicate.

UV detection, respectively. The PC detector was previously found to be linear from 1 to 100 ng injected (Walters, 1983). All injections in the present study were made in the range of 1–10 ng of chlorothalonil in 5 μL aliquots, and the response was linear over this range for both detectors. The minimum levels of reliable quantitation by LC-PC and LC-UV are dependent on sample background, but both detection methods are clearly capable of determining residues at levels well below the tolerances established for tomatoes and cherries. UV detection typically shows more background interference from crop extracts than was encountered in this study. The relatively strong absorbance of chlorothalonil at 232 nm enhanced the utility of the UV detector for this application. Because the PC detector is more complex and sensitive to variables in system conditions (e.g., deionization and degassing of mobile phase, temperature, pumping fluctuations, etc.), the authors routinely operate it in tandem with a UV detector, which serves to monitor the chromatographic system and aid in the diagnosis of anomalies.

Standard solutions were initially prepared in methanol. Degradation of chlorothalonil in methanol was apparent within 24 h, however, and 2-propanol was therefore substituted for both standard and sample dilution. No degradation in 2-propanol was noted after several weeks at room temperature. The 2-propanol was dried over an-

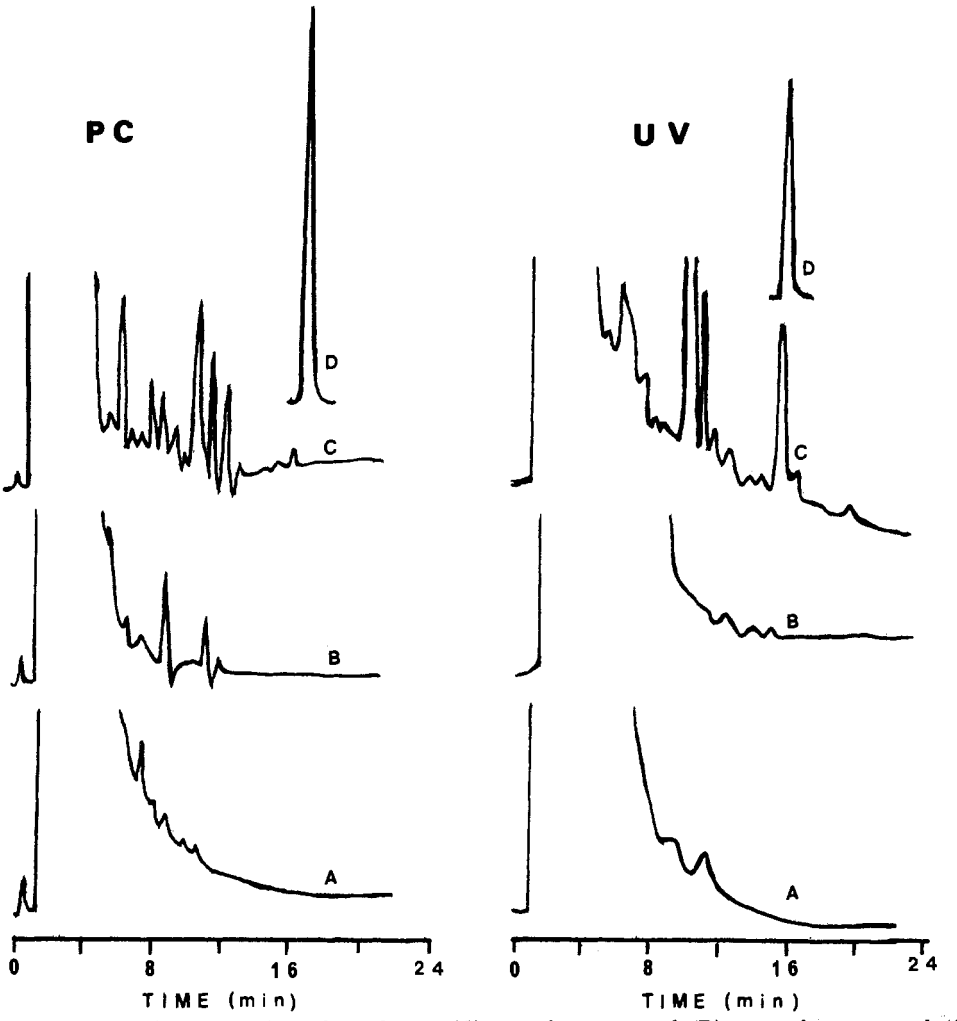

Figure 2. Chromatograms of sample extracts from C-8 column: (A) sour cherry control; (B) sweet cherry control; (C) tomato control; (D) chlorothalonil peak for fortification level of 0.2 ppm, or 3 ng in 15 mg of sample equivalent injected. Conditions: detector sensitivities, 10 × 5 (PC) and 0.01 AUFS (UV); mobile phase, 60% methanol in water at 1 mL/min flow rate; temperature, 35 °C.

hydrous sodium sulfate as a precautionary measure against hydrolysis.

A study of Szalkowski and Stallard (1977) on stability of chlorothalonil found it to be stable at pH 7 or lower; at pH >9, hydrolysis to 4-hydroxy-2,5,6-trichloroisophthalonitrile and 3-cyano-2,4,5,6-tetrachlorobenzamide occurred. Standard materials for these two hydrolysis products were obtained (the hydroxy compound from EPA and the cyano compound from Ricerca, Inc., Painesville, OH) and injected into the C-8 column with 60% methanol in water (v/v) as mobile phase. The polar hydroxy compound eluted in the solvent front (unretained), and the cyano compound eluted in about 8 mL (8 min) of retention volume. Both compounds were detectable by PC and UV, but to a lesser degree than chlorothalonil. Thus, their concurrent determination as trace metabolites in uncleaned sample extracts using the LC system for chlorothalonil did not appear readily feasible. Additional cleanup and different LC separation systems would be required, especially for the 4-hydroxy metabolite. Existing methodology developed specifically for chlorothalonil and the 4-hydroxy metabolite as crop residues involves acidified acetone extraction, partitioning into ether, Florisil cleanup and separation, and derivatization of the 4-hydroxy compound with diazomethane prior to determination by GC with microcoulometric or electron capture detection (*Pesticide Analytical Manual*, 1970). In consideration of the rather strongly alkaline condition reportedly required for hydrolytic formation of these metabolites, significant residues in fruits and vegetables would not seem probable. As reported by Vettorazzi (1977), only negligible residues of the 4-hydroxy metabolite have been found on most crops investigated. No evidence of the presence of these metabolites in samples or standard chlorothalonil solutions (including degraded solutions in methanol) was seen in the course of the present work.

This study has demonstrated the capability of LC with photoconductivity detection for accurately measuring chlorothalonil residues at levels well below those of official concern in uncleaned extracts of fresh produce prepared by a rapid, multiresidue screening method. With proper choice of column and mobile phase, the applicability of this technique to residues of other pesticides in other crop extracts similarly prepared seems probable.

Registry No. Chlorothalonil, 1897-45-6.

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Relationship between Liquid and Gas Chromatographic Retention Behavior and Calculated Molecular Surface Area of Selected Polyhalogenated Biphenyls

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The retention behavior of 57 mono- or polyhalogenated biphenyls was characterized by gas chromatography on a DB-210-CB capillary column and by high-pressure liquid chromatography on ODS phases. A possible relationship between the recorded retention data (Kovats index) and molecular surface area was explored. Generally, the addition of halogen atoms (Cl, Br, I) to biphenyl increased both the retention index and the calculated molecular surface area for the series of polyhalogenated biphenyls investigated. The addition of fluorine however resulted in a decreased retention index although the molecular surface area was slightly increased. The addition of halogen substituents resulted in more dramatic changes in retention as demonstrated by the Kovats index (I_K) for gas chromatography than for liquid chromatography. The homologues with chloro or bromo substituents in ortho positions displayed lower I_K values than those with halogens in meta and para positions. Generally, good correlation was observed between I_K and the calculated molecular surface area values.

Polyhalogenated biphenyls are particularly well-suited for a number of industrial uses, and these chemicals have been widely manufactured (e.g., Aroclor and fireMaster products are commercial mixtures used as fire retardants). Unfortunately their chemical stability and fat solubility contribute to their environmental persistence and biomagnification. Today polyhalogenated biphenyls, particularly polychlorinated biphenyls (PCBs), are routinely detected in environmental samples, in foodstuffs, and in human tissues. These products are complex in nature [209 isomers each of PCBs, polybrominated biphenyls (PBBs), and polyfluorinated biphenyls (PFBs) are possible]. Such a degree of complexity complicates the analysis of these mixtures in environmental samples. Even the use of capillary gas chromatography and high-resolution mass spectroscopy does not always give satisfactory results.

Often reversed-phase high-pressure liquid chromatography (HPLC) separations are used in sample cleanup procedures. In order to get more information about the influence of structure on the HPLC retention behavior of polyhalogenated biphenyls, 57 selected compounds have been synthesized and their retention characteristics are presented in this paper. The corresponding retention measurements are also presented for capillary (DB-210-CB column) gas chromatography.

The structures of several PCBs and PBBs have been shown to strongly influence retention behavior in both gas and liquid chromatography (Robertson et al., 1984; de Kok et al., 1977). Likewise, for many classes of compounds an excellent correlation between HPLC and GC retention times and the corresponding calculated molecular surface area (SA) has been demonstrated (Dunn et al., 1986; Möckel et al., 1987a–d). In order to explore such a correlation for polyhalogenated biphenyls and to eventually predict the structure of unknown compounds, we present

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