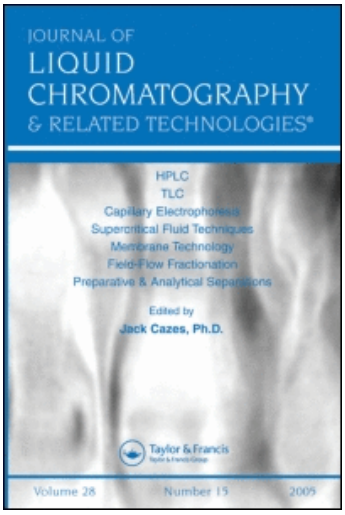

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Determination of Pentachlorophenol Laurate by High Pressure Liquid Chromatography

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DETERMINATION OF PENTACHLOROPHENOL LAURATE BY HIGH PRESSURE LIQUID CHROMATOGRAPHY

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ABSTRACT

Pentachlorophenol Laurate (PCPL) in canvas is determined by extraction and chromotography on silica gel. Conditions are chosen to eliminate peak splitting due to the presence of different "laurate" fatty acids. The determination is faster and more specific than previously reported methods.

INTRODUCTION

Pentachlorophenol (PCP) and its laurate ester are widely used as fungicides to inhibit the decomposition of fabrics and wool in contact with soil. PCP has been determined colorimetrically (1), by gas chromatography (2), by U.V. spectrophotometry (3) and by HPLC (4). PCPL has been determined by oxygen flask combustion and chloride titration (5), by hydrolysis to the free phenol, steam distillation and colorimetry (6), and U.V. spectrophotometry (3). Chromatographic determination of PCPL is complicated by the fact that the "laurate" is a mixture of C_{10}-C_{16} fatty acids, usually derived from cocunut fatty acids. Conse-

quently PCPL is a mixture of the esters of these acids, and will tend to give multiple peaks in chromatography, which may make both identification and quantitation uncertain. An HPLC system which yields only one peak for PCPL has been devised, which allows rapid and accurate determinations of the PCPL present in treated fabrics.

EXPERIMENTAL

Materials

PCPL was extracted from a commercial emulsion (Mystox). The emulsion was heated until the gum present as a protective colloid coagulated. It was then extracted with ether and washed repeatedly with 10% sodium carbonate and distilled water. The ether was removed under vacuum.

Internal standard: Benzyl-diphenyl (BDH, for gas chromatography) was recrystallized from hexane and methanol to yield chromatographically pure 4-benzyl-diphenyl, M.P. 85°C.

HPLC apparatus

A Varian Model 5030 liquid chromatograph equipped with a Waters R-401 differential refractometer was used. The stainless steel column (Varian Micro-Pak Si-5, 5 micron silica) was 30 cm. long and had an i.d. of 4 mm. Chromatograms were recorded and integrated with a Hewlett-Packard Model 3380A integrator. A Valco sample injector with a 10 microliter loop was used to inject samples into the chromatograph.

Method

A. Sample Prepararion

A 1 gm. sample of canvas is extracted with 20-30 cc of 1% acetonitrile in hexane for 5 minutes at the boil. After cooling, an aliquot of a solution containing 4-benzyl-diphenyl (10-15 mg/ml) is added as an internal standard. The combined solution is filtered and injected into the chromatograph.

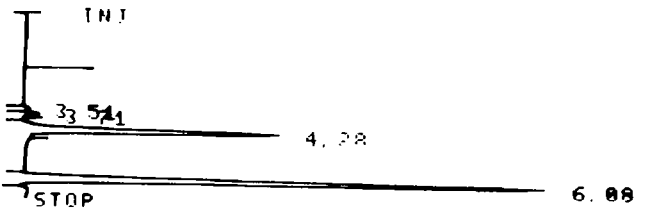
B. Chromatographic Conditions

The mobile phase consisted of 1% acetonitrile in hexane, with a constant flow rate of 0.7 ml/min. PCPL had a retention time of 4.3 minutes and 4-benzyl-diphenyl had a retention time of 6.2 minutes. The temperature was held constant at 25°C.

RESULTS AND DISCUSSION

Chromatography of PCPL on an octadecyl reversed phase column yielded multiple peaks corresponding to the different "laurate" esters. Elution of PCPL from an unmodified silica column yielded a compound peak which showed splitting i.e., partial separation was observed. Attempts to decrease selectivity by addition of as little as 0.1% isopropanol to the hexane resulted in elution of the PCPL with the solvent front. Elution with 1% acetonitrile in hexane yielded a single, somewhat assymetric peak which was readily integrated.

The assay was calibrated daily (Fig. 1) by injection of a known mixture of PCPL and internal standard (4-benzyl-di-

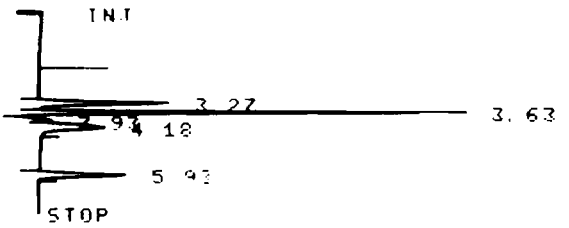


LIST
%RTW 5 ISTD

RT	ID#	AMT/AREA
6.08	1	.001 033
4.28	2	002 169

FIGURE I - Chromatogram of calibration mixture of PCPL and 4-benzyl-diphenyl, with integrator print-out of calibration factors.

phenyl) and allowing the integrator to compute a relative response factor which was stored in the integrator and used for the subsequent analyses (see Fig. 2). The integration was linear over a range of at least 10-100 micrograms PCPL, the amounts injected in the course of this study.



RT	TYPE	AREA	ISTD ID#	AMT
3.27		11350		.
3.63	M	23701		.
3.93	M	2248		.
4.18	M	9975	2	2.739
5.93		7508	1	.

FIGURE II - Chromatogram illustrating analysis of a fabric containing 2.6% PCPL (by U.V.).

TABLE I

A Comparison of the Two Methods of Analysis

Sample No.	% PCPL in Air Dried Sample	
	U.V. Method	HPLC Method
1	1.6	1.6
2	1.7	1.8
3	2.5	2.5
4	2.6	2.7
5	3.2	3.2
6	5.7	5.6
7	7.7	7.1

The results obtained by this method were compared with those obtained by the U.V. assay (3) on a series of canvas samples which had been treated with PCPL. This comparison (see Table I) indicates that this HPLC procedure effectively separates PCPL from other substances eluted from the fabric and yields results consistant with those obtained by the U.V. method. Reproducibility of the HPLC assay, as determined by replicate determinations on separate specimens cut from the same fabric sample (see Table II) is comparable to that achieved by other methods and is probably limited by the evenness of PCPL distribution in the fabric.

The HPLC method is more rapid than the previously reported methods and may be more specific and less subject to interferences than methods based upon chloride analysis, colorimetry or

TABLE II

Reproducibility of the HPLC Determination
% PCPL in Air Dried Sample

Replicate	Sample A	Sample B
1	2.5	5.5
2	2.4	5.7
3	2.4	6.0
4	2.4	5.7

spectrophotometry. Although sensitivity would be increased by the use of a U.V. detector instead of a refractive index monitor, the proposed method is adequately sensitive for fabrics containing 1-7% PCPL and avoids the necessity of using U.V. grade solvents.

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