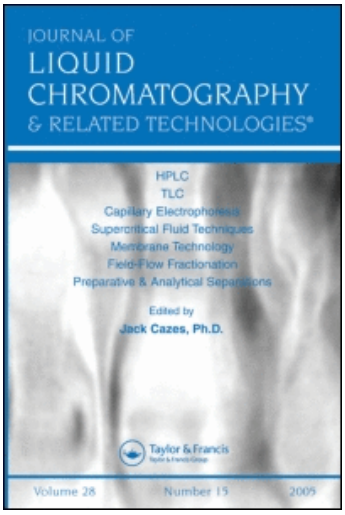

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A Rapid Gpc Method for the Estimation of Hatcol-200 in Polymers

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A RAPID GPC METHOD FOR THE ESTIMATION OF HATCOL-200 IN POLYMERS

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ABSTRACT

A simple, rapid Gel permeation Chromatographic method has been disuussed for the isolation and estimation of Hatcol 200, the newly introduced plasticizer for Poly(Vinyl Chloride). The non-interference of the ubiquitous phthalate esters is the added advantage of the method.

INTRODUCTION

The widespread use of phthalate esters and phthalates containing products for varied applications, including medical uses, has resulted in the extensive environmental pollution by this class of compounds. The occurrence of the esters particularly of Di-2(ethyl hexyl) phthalate (DEHP) in blood, fluids for intravenous administration, tissues etc

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and their toxic hazards have been discussed and documented widely (1,2,3,4,5,6). The need for alternative chemicals, having relatively less toxicity, simultaneously having the plasticizing efficiency of phthalates has emerged. Hatcol 200 [Tris (2-ethyl hexyl) trimellitate] is one such chemical available today which could be used in Poly (Vinyl Chloride) (PVC), the most commonly used polymer for fabricating the disposable medical devices.

It seems that a simple, fast and sensitive analytical method for the analysis of this plasticizer is lacking. This communication discusses a GPC method to estimate Hatcol 200 in PVC as well as in the presence of commonly found phthalate esters.

EXPERIMENTAL

Materials

Phthalate esters, Di-2-(ethyl hexyl) phthalate (DEHP), Di-butyl phthalate (DBP), Di-ethyl phthalate (DEP) and Di-methyl phthalate DMP) were from Indo-Nippon Co. Hatcol 200 (Hatco Chemical Co., Fords, NJ) is a generous gift from Mr. K. Rathinam. Analytical grade THF (Sisco, Bombay) was distilled prior to use.

Apparatus

The Chromatographic systems employed consisted of a Waters Assoc. Inc. Model 6000A solvent delivery pump, 440 absorbance detector, R-400 series RI detector and U6K injector. A strip chart recorder (Houston Instruments, Tx) was used for getting the Chromatograms. A 100A pore size μ-styragel column was used for the chromatographic separations.

Samples

A stock solution of phthalate esters and Hatcol 200 was prepared by dissolving appropriate concentration of the components (1-2 mg/ml) in THF. Indigenously prepared PVC films containing about 2% Hatcol 200 were dissolved in THF, filtered through millipore filters and used for further analysis.

Chromatography

Freshly distilled THF was used as eluent at a flow rate of 1 ml/min. 100 μl volume of the solutions was injected onto the column. The column effluents were monitored by UV detector (254 nm) or by the RI detector.

Calibration plot

A calibration plot for Hatcol 200 was constructed between the peak height (at 254 nm) and amount of

Hatcol 200 in the injected volume for varied concentrations.

RESULTS AND DISCUSSION

In fig.1 is shown the chromatogram of the five plasticizers. It is apparent from the figure that, phthalates do not interfere the analysis. Fig.2 illustrates a typical chromatogram of the PVC solution. PVC is excluded from the column and elutes earlier than Hatcol 200. The retention time under the experimental conditions and the molecular weights of the five components are shown in Table 1. Quantity of Hatcol 200 present in each of the PVC films was determined from the calibration plot. The recovery rate (10 analysis) was 96.3 $\pm$ 2.4% and the detection limit in the present case was 2 μg/ml.

Although we have not been attempted it, it appears the method can be extended for the separation and estimation of Hatcol 200 in presence of other common additives found in PVC.

Traditionally GPC is the method of choice for the analysis of macromolecules and its capability for providing the molecular weight distribution and molecular weight averages of polymers has been well established. In recent years, particularly with

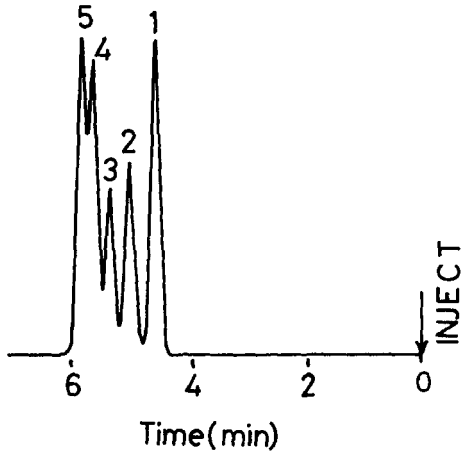

FIGURE 1 : Chromatogram of a mixture of five Plasticizers, Peaks : 1 = Hatcol 200 2 = DEHP; 3 = DBP; 4 = DEP; 5 = DMP

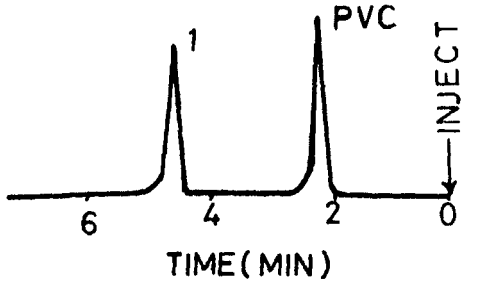

FIGURE 2 : A typical chromatogram of a THF solution of PVC containing Hatcol 200. Peak 1 : Hatcol 200.

TABLE - 1

RETENTION TIMES (t_R) AND MOLECULAR WEIGHTS OF PLASTICIZERS STUDIED

Sample	t_R (in min)	Molecular weight
Hatcol 200	4.6	547
DEHP	5.1	390
DBP	5.4	278
DEP	5.7	222
DMP	5.9	194

the advent of high resolution micro packing columns, GPC has routinely been employed to qualitative and quantitative analysis of polymeric additives (7,8,9). Both normal phase and reverse phase chromatographic methods can certainly separate Hatcol 200 from other additives. However, these methods require extensive chromatographic preparedness. Often the need for tedious, lengthy trial and error procedures arises in order to obtain a suitable mobile phase composition to provide acceptable resolution of multicomponent mixture. Moreover, the isolation of additives

from the polymer matrix is essential prior to chromatographic investigations. GPC, on the other hand, requires the least sample preparation, and the polymer solution can be directly injected for the analysis.

Certainly GPC has its own demerits. The limited peak capacity and the inability to differentiate structural differences of molecules having the same molecular weights are the major drawbacks. However, the molecular size dependence in elution behaviour, unlike other chromatographic methods, provides the molecular weights of the eluates which could be helpful in the identification of unknown components. This unique ability is particularly advantageous in the analysis of newly introduced chemicals like Hatcol 200.

An understanding of the toxic properties of the component as well as its metabolic products is of cardinal importance for incorporating the chemical in polymers intended for medical uses. The present method could be extended for the separation of the metabolic products of the chemical and the elution - molecular size correlation would provide the molecular weight data of the degraded products which could lead to a better understanding of the conver-

sion of the chemical in living systems. This method can also be utilized for routine quality control uses.

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