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Note

Liquid chromatographic determination of the concentration of a vinyl chloride-vinyl acetate copolymer in biological samples

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Certain high-molecular-weight polymers are generally accepted as safe for food contact, although there is a lack of published evidence to support this view. In particular, little information¹ is available on the toxicity of polyvinyl resins, which are widely used in food packaging and clothing materials, towards human and animals because of the difficulty of their determination.

This paper describes a high-performance liquid chromatographic (HPLC) method for the separation and determination of the concentration of a high-molecular-weight vinyl chloride-vinyl acetate copolymer (PVC-PVAc) in biological samples.

EXPERIMENTAL

Apparatus

The system consists of a 6000A liquid chromatograph (Japan Waters, Tokyo, Japan), a TSK-GEL-GMH column (Toyo Soda, Tokyo, Japan), an RID-2A refractive index detector and a strip-chart recorder (Shimadzu, Kyoto, Japan).

Materials

The PVC-PVAc was a copolymer of 70% vinyl chloride and 30% vinyl acetate, with a mean degree of polymerization of 400. It was prepared in the form of particles and was added to the diet of test rats at various concentrations.

Animal experiment

Male Wistar rats weighing approximately 80 g were separated into three groups. The first group was fed a basal diet (no copolymer), the second group a diet containing 1% and the third group a diet containing 2% of PVC-PVAc. During the experiment, excreta were collected every 7 days. After 3 months, the animals were killed and various tissues were removed. To the tissues (1 g) or the faeces (1 g), 5 ml

of tetrahydrofuran (THF) were added and the mixture was homogenized, and to the urine (1 ml) 5 ml of THF were added and mixed. The samples were then centrifuged at 35,000 *g* for 1 h and the supernatant solutions were used as samples to determine the PVC-PVAc concentration.

Determination procedure for PVC-PVAc

The mobile phase (THF) was pumped at a flow-rate of 1.0 ml/min into the HPLC column. A 100- μ l sample of a standard solution containing PVC-PVAc (0.025–0.5 g per 100 ml of THF) or a 1000- μ l sample of the animal samples was injected on to the column. The PVC-PVAc concentration was measured with the refractive index detector. The peak-height method was used for quantitative analysis.

RESULTS AND DISCUSSION

A typical chromatogram obtained with a THF solution containing 0.5 g of PVC-PVAc per 100 ml) is shown in Fig. 1. Fig. 2 illustrates the relationship between peak height and concentration of PVC-PVAc. A linear relationship was observed in the concentration range 0.025–0.5 g per 100 ml.

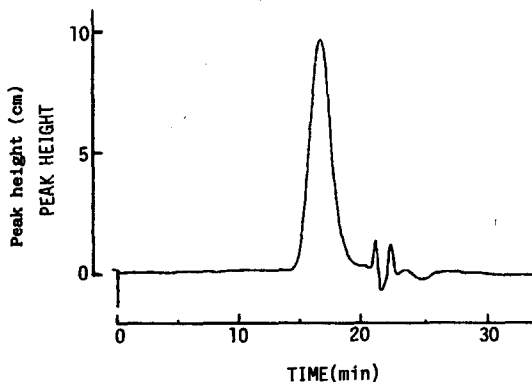


Fig. 1. Chromatogram of PVC-PVAc.

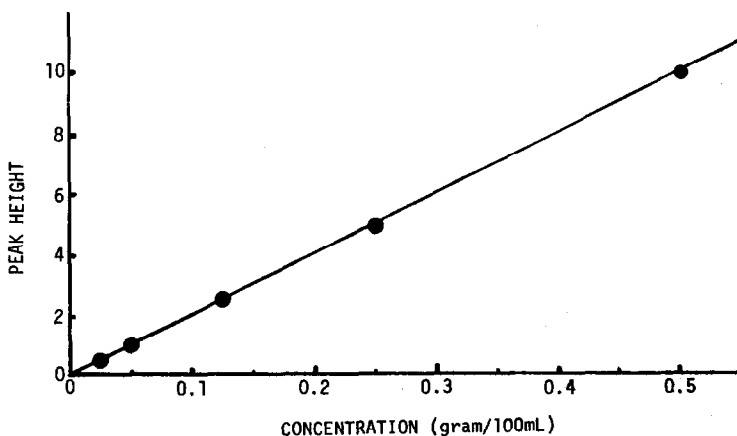


Fig. 2. Calibration graph obtained for PVC-PVAc.

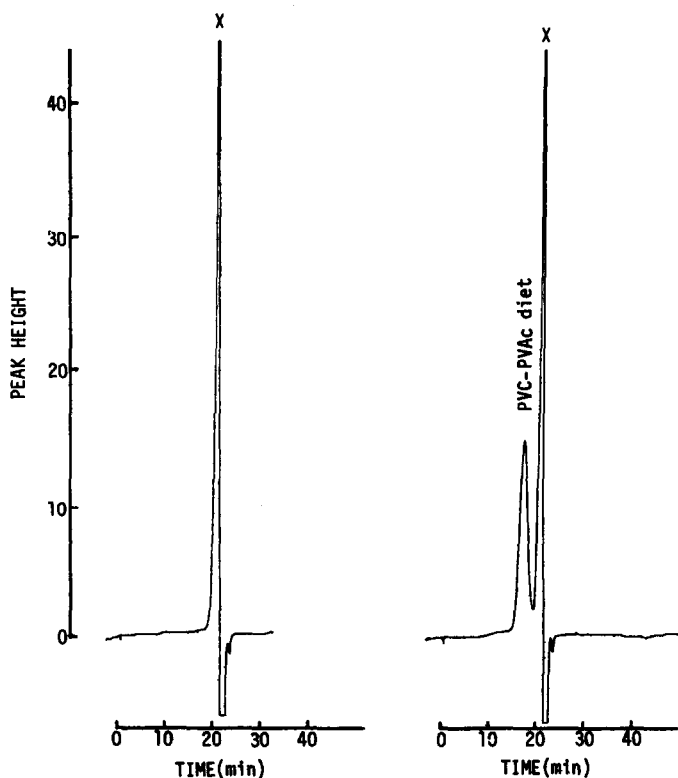


Fig. 3. Chromatogram of PVC-PVAc in the faeces of a rat fed the basal diet (left) and a diet with 1% of PVC-PVAc added (right). X = Unknown substance.

The animal experiment revealed that almost all of PVC-PVAc administered was detected later in the faeces and not in the urine, blood or tissues. This suggests that the PVC-PVAc was not absorbed from the intestine even when very large amounts were administered orally. Fig. 3 shows a typical chromatogram of the faeces of a rat that had been fed a diet containing PVC-PVAc.

REFERENCE

1 H. F. Smith, *Toxicol. Appl. Pharmacol.*, 9 (1966) 501.