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Preparative Layer Chromatography

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I. INTRODUCTION

Preparative layer (planar) chromatography (PLC) is a liquid chromatographic technique in which the solvent-solvent composition migrates through the stationary phase either by capillary action or under the influence of forced flow with the aim of separating compounds in amounts of 10–1000 mg (1). The compounds can be isolated for structure elucidation (IR, UV, MS, ¹H-NMR, ¹³C-NMR, CD, etc.), for various analytical purposes or for determination of biological activity (2).

Depending on the mode of solvent composition migration, PLC can be classified as classical PLC (CPLC) or forced-flow planar chromatography (FFPC). In the first type of PLC, the solvent migrates by capillary action (3). The category FFPC includes all methods in which the mobile phase migrates not only by capillary action but also by forced flow. For preparative purposes, two basic FFPC methods have so far become available: forced flow can be achieved either by application of external pressure [overpressured layer chromatography (OPLC)] (4–8) or by use of centrifugal force [the various types of rotation planar chromatography (RPC)] (9–12). The enhanced efficiency of FFPC techniques as evidenced by a comparison of their analytical properties with those of classical thin-layer chromatography (TLC) and high-performance TLC (HPTLC) is well known (e.g., 2), because by use of FFPC techniques the advantage of the optimum mobile-phase velocity can be practically exploited over the entire separation distance without loss of resolution. This effect is independent of layer thickness and the type of forced flow applied.

Both types of FFPC may be used as on-line preparative techniques (13,14), i.e., techniques in which the separated compounds are eluted from the stationary phase and isolated from the instrumentation. This enables connection of a flow detector, recording of chromatograms, and collection of separated compounds with a fraction collector (Fig. 1). FFPC methods enable not only micropreparative (OPLC) and preparative (RPC) separations but also, by using appropriate split systems, the coupling of these methods with various spectroscopic techniques, as is apparent from Fig. 1. In this way, not only isolation but also structure elucidation can be carried out in a single process.

The chromatographic processes operative in CPLC, preparative OPLC, and RPC basically resemble those of analytical TLC, OPLC, and RPC, respectively. The most important factors that may influence a PLC separation are shown in Fig. 2.

Working with PLC requires consideration of some special characteristics, such as the average particle size, the thickness of the stationary phase layer, the chamber type, the application of large amounts of sample, the location and detection of the separated compounds, and the removal of the desired compounds by elution or extraction. The type of stationary phase, the composition of the mobile phase, the separation distance, the mode of development, and the working temperature may be identical with those in analytical TLC. The procedures have been described extensively for analytical TLC (15–18) and summarized for PLC (19–21).