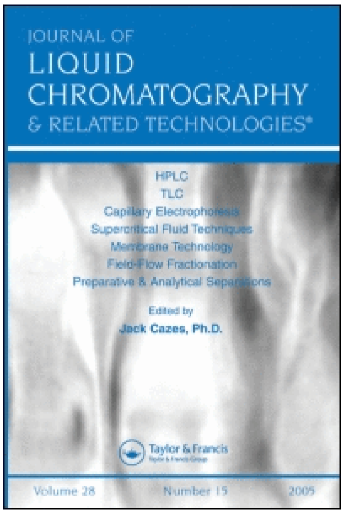

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A NEW DETECTOR SYSTEM FOR CONTINUOUS-FLOW TLC

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SUMMARY

A new detector system for continuous-flow TLC is described that provides possibilities both for optical and for electrochemical measurements with low detection limits.

1. Introduction

The advantages of the continuous-flow thin-layer chromatography /CFTLC/ over the conventional TLC methods are higher speed and separation power. The benefits of CFTLC first came to the attention of a wide audience in 1955 when M. Mottier and M. Potterat /1, 2/ published their well known work. The onset of the modern CFTLC equipment goes back to the mid-1960s when E. Stahl /3/ introduced a convenient TLC chamber. This was followed by important works of M. Brenner and A. Niederwieser /4,5/, S. Hara /6/, F. Geiss and F. Schlitt /7/, E.V. Truter /8/, L.M. Libbey and E.A. Day /9/ and J.H. VanDijk /10/. The possibilities of CFTLC are now routinely available with

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* Presented at the First Symposium on Advances of TLC and HPLC, May 14-15, 1982, Szeged, Hungary.

commercial equipment of the firms Camag /11/, Desaga /12/ and The Dime's Group, Inc /13/.

Since the selection of a mobile phase of adequate strength and selectivity is a major task is designing LSC separations, anything that simplifies this task will prove helpful. The right solvent can be chosen using, directly, the LC unit, but this is often a tedious, time consuming procedure. A much easier and faster one is to use TLC in the initial scouting for an appropriate mobile phase /14/. Under similar conditions separation by LSC and TLC proceed by the same retention mechanism, and R_f values in TLC can be used to predict k' values in LSC /same adsorbent and mobile phase/ by relationship $k' = /1-R_f//R_f$. However, there are several reports of the poor correlation between the TLC and LSC data. This can be accounted for by solvent demixing, variations in the adsorbent and adsorbent water content and by solvent concentration gradient in TLC. These difficulties are minimized in CFTLC as it is shown by H. Schmid, J. Cramer and H. Arm /15/. Their experimental installation is like a column chromatograph equipped with a thermostat , solvent system, sample injector for "wet dosing", chamber for evaporation of eluate, thin layer plate with glass support subdivided into two symmetrical parts of adsorbent - sample and reference. The detection takes place directly on the thin layer foil during the separation of sample components. The optical detector has a maximum of sensitivity at 550 nm /transmission/. The detection limit is about 10^{-9} g.

2. Experimental

Merck TLC Aluminium Sheets, Plastic Sheets, HPTLC pre-coated glass plates and home made plates /Silica gel 60, Merk/ without any binder. The experimental installation is similar to /15/ and includes solvent pumping system with controlled composition of the mobile phase during the development process. The electrochemical detection system is analogous to that described in ref. /16/. On plates with the glass support a small region of adsorbent layer is removed preliminarily and the light from fibre optic detection system passes through such optical tranparent region /17/ / Fig. 1/.

In the support of Aluminium and plastic TLC sheets apertures were made. If the support has a smooth surface, it is possible to utilize the reflectance detection system, such as described in ref. /17/. The diameter of the region without adsorbent and the aperture does not exceed 1-3 mm.

3. Results and Discussion

Excelent systems for CFTLC with the transparent optical region are HPTLC pre-coated plates from Merk, which compare favourably with well filled HPLC columns fith respect to the quality of packing /N-3450 for migration distance Z_f =60 mm/. With these plates the transparent optical region of any shape and size can be easily obtained. The efficiency of the plates prepared in the laboratory /layer thickness 0.4mm/ reaches N=700 for Sudan /Z_f=100 mm, Fig.2/.

Direct comparison of plate performance may present difficulties because of the variations in the quality of

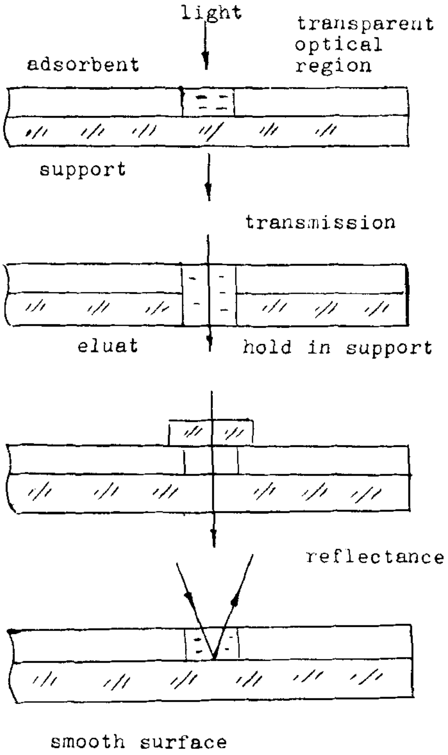

FIGURE 1

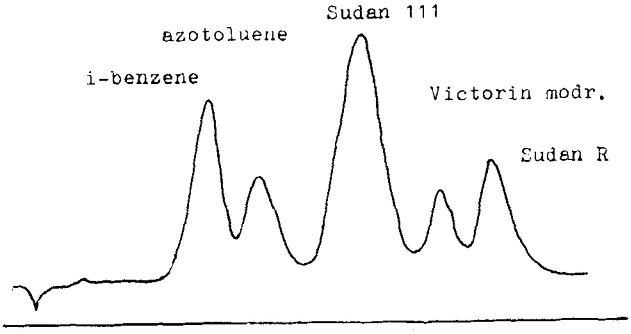

FIGURE 2

TLC plates and extra plate effects /e.g. sample injection, evaporation of the eluate, etc./.

In the same experimental conditions/transmission light/ the detection limit becomes /10 times/ lower for TLC plates in the laboratory than for precoated HPTLC plates /1,5--3 times/.

There are some problems in the reflectance detection due to specific properties of support, larger area of the transparent optical region, etc.

All the experiments were made on a linear chromatographic system, but the circular thin-layer and overpressured thin-layer chromatography may be used as well /19-22/.

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