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# Circular thin-layer chromatography on the standard plate with the closed sorption layer under the small pressing pressure

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## 1. Introduction

The velocity of eluent migration is one of the most significant characteristics of the chromatographic method [1,2]. Not high and steadily decreasing velocity of migration of the mobile phase is the significant drawback of all the traditional variants of TLC. To overcome this drawback the Hungarian chromatographists proposed to use a forced flow of a mobile phase through a TLC plate temporarily turned into the flat column (TLC under pressure) [3,4]. The Hungarian variant of TLC has been successfully used for many decades, however, its practical using is slightly restricted what is conditioned by the high cost of the used equipment. That is why the working out of simpler systems, in which only very small overpressure was used [5,6], seemed expedient to us. The arrangement of TLC under pressure is substantially simplified if circular chromatography is used [7]. Using the idea of R.Kaiser [7] the variant of the simple chamber with using of very small initial pressure of the mobile phase (Fig.1) has been designed by us. In the proposed variant of TLC the small pressing pressure of the polymeric film to the sorption layer which is evenly distributed on the whole TLC plate area is used.

## 2. Experiment and results

The research was conducted on the plates HPTLC-P-UV "Sorbfil" (company IMID, Russia) of size of 10×10 cm. As the mobile phase toluene was used. As the object for separation dye mixture - Test dye mixture III (CAMAG) – was used. The schematic of the experimental arrangement is given at the Fig.1. The chamber consists of the TLC plate, above which the quartz capillary (3) is placed. The mobile phase under the small pressure was supplied from the reservoir by the capillary to the TLC plate. For arranging of the closed layer the plate sorption layer was covered by the teflon film and the rubber gasket, and then it was pressed by means of the protective glass (6) and the loads of the different mass. As the pressing loads the steel disks of the total mass of 3.2-13.0 kg were used. It should be noted that the observation over the process of separation was implemented from below of the chamber by means of the mirror (it is not shown at the figure). In order to register the separation the videodensitometer and the software "Sorbfil" (IMID, the error of characteristics determination is 3-7%) were used. The distance for separation made up 4 cm. To estimate the method of circular TLC under pressure the experiments were also conducted with traditional circular TLC with the open sorption layer, when the distance between the plate sorption layer and the protective glass made up 2 mm.

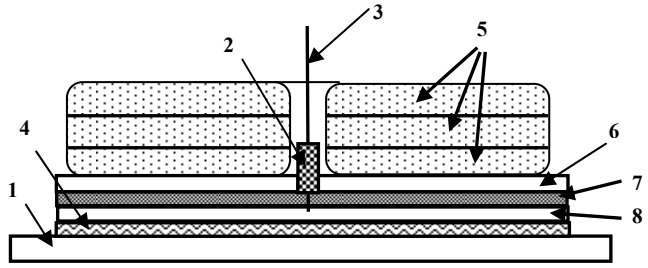


***Fig.1. The schematic of the chamber for circular TLC under the small pressure.***
1 - base, 2 - fastening of capillary, 3 – capillary for eluent supply, 4 – plate support, 5 – load, 6- protective glass, 7 – sealing, 8 – sorption layer

The small pressure of the mobile phase was provided at the expense of the difference of the levels (Δh), that is the difference between the level of the mobile phase in the tank and the level of TLC plate. The experimental results are given at the Fig.2 and Fig.3. The least time of the analysis characterizes the circular TLC variant under the pressure at the levels difference Δh=18 cm, what is 52% faster than in case of the traditional circular TLC. The variants of circular TLC with the levels difference Δh=14 cm and Δh=16 cm have the analysis time less by 33% and 46%, respectively. The levels difference increases, the small increase in the efficiency of separation is observed, what is connected with the less broadening of the zones of the compounds. At Δh=18 cm the sharp decrease in the efficiency of separation is observed, what is connected with the excess, in the given conditions, eluent supply to the TLC plate.

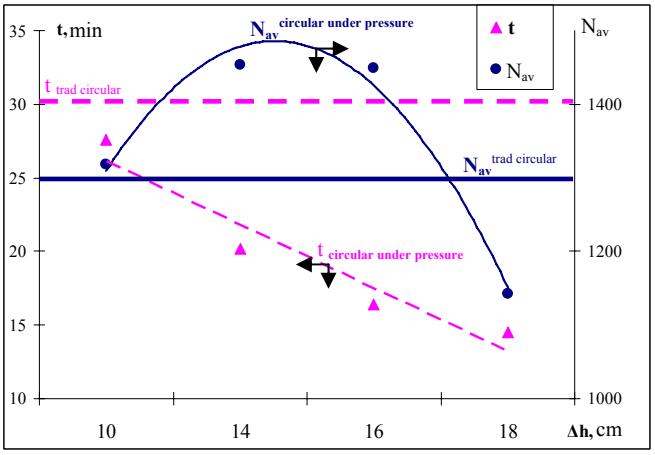


***Fig. 2. The chromatographic characteristics of separation in circular TLC under pressure at the various level of the supply (m=9.4 kg).***

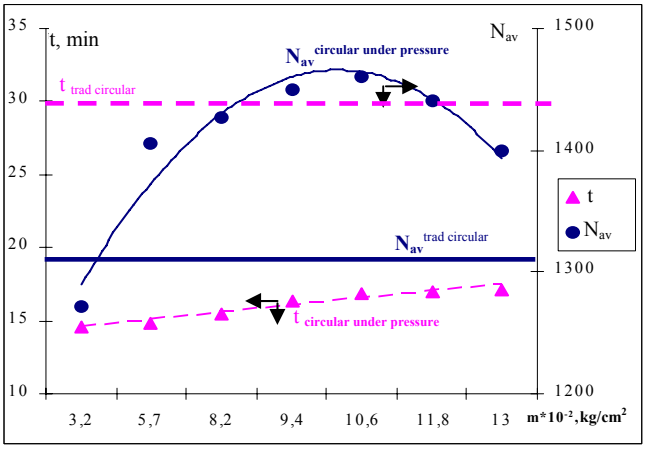


***Fig. 3. The chromatographic characteristic of separation and the analysis time in circular TLC under pressure at the various pressing pressure (Δh=16 cm).***

where $t_{trad\ circular}$ – duration of separation in the traditional circular TLC, $t_{circular\ under\ pressure}$ - duration of separation in the circular TLC under pressure, $N_{av}^{trad\ circular}$ – the average number of theoretical plates in the traditional circular TLC, $N_{av}^{circular\ under\ pressure}$ - the average number of theoretical plates in the circular TLC under pressure.

As it follows from the Fig.3, the variants with the intermediate mass m=8.2-10.6 kg are characterized by the less analysis time to compare to traditional circular TLC by 44-49%. The pressing pressure increases, the separation duration increases. The change in the pressing pressure does not substantially influence on the efficiency of separation (when increasing of the pressing pressure the small increase in the efficiency of separation by 7-11% is observed).

The similar results have been obtained when using Silica gel 60 $F_{254}$ ("Merck") as the plates and ethanol as the eluent.

## 3. Conclusion

The using of circular TLC with the closed layer and the small external pressure of the mobile phase permits to decrease the separation duration (~ by 40-50%), as well, it permits to carry out the separation of the researched compounds with the slightly higher efficiency (~ by 10%).

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