1h

The application of frontal and displacement chromatography in TLC for the concentration of impurities

V.G. Berezkin, A.V. Chausov

A.V. Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences, 29, Leninsky pr., Moscow, 119991, GSP-1, Russia

e-mail: berezkin@ips.ac.ru

1. The new approach to concentration in TLC

The working out of the new methods for impurities identification is the significant area of development of analytical chemistry. The most universal approach of decreasing the threshold of impurities identification is concentration [1]. For concentration of impurities it is expedient to use frontal and displacement TLC. It is interesting to note that frontal-elution variant of circular TLC, that is the variant, in which a sample and an eluent moves to one point on the TLC plate was described by N.A. Izmailov and M.S. Shraiber in the first work on TLC [2]. The application of frontal-elution variant in TLC (Fig.1) is expedient since when carrying out separation of compounds by the method of frontal chromatography not only the initial separation takes place, but also concentration of the sample components does, what allows more reliably identifying many impurities. When using the given variant of circular TLC the chromatogram looks like narrow concentric circumferences. Using of displacement variant in TLC leads to concentration of diluted samples. Thereby, the combined consecutive using of frontal, displacement and elution methods when identifying the impurities is expedient to be used in TLC. In the given work the estimate of possibility of application of frontal-displacement-elution method in circular TLC has been carried out on the example of impurities identification. Although these tasks are solved in frames of using of circular TLC, the given approach might be applied in linear TLC, as well.

2. Experimental research and results

The experiment conditions: the plates Silica gel 60 F_{254} ("Merck") of size of 5×5 cm, the mobile phase is toluene, the displacer is ethanol, the model object of the analysis is the solution of phenol derivatives (2,6-dimethylphenol, 4-butylphenol) in toluene with the total concentration from 10 g/l to 0.001 mg/l, the samples of sewage waters of by-product coke industry before the purification (the concentration of phenols is not known) and after the purification (the total concentration of phenols is 0.275 mg/l). The volume of the sample being applied is 70 and 130 µl. The chamber for circular TLC was used. It should be noted that the supply of the sample and the solvents was implemented at the same point on the plate. To work out the chromatograms videodensitometer "Sorbfil" ("IMID") was used. The experiment was conducted in three stages: 1) the sample application; 2) the concentration using the development by the solvent, in which the researched compounds have R_f=1; 3) the elution (development by the solvent). After every stage the plate was dried, and the obtained chromatogram was detected by means of the densitometer (UV=254 nm).

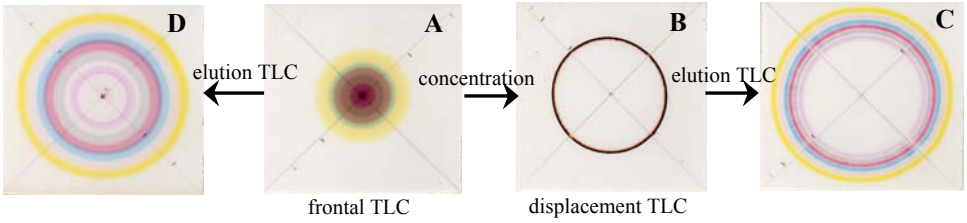

Fig. 1 The application of frontal-elution and frontal-displacement-elution TLC
(the mobile phase is toluene, the displacer is ethanol, the object of separation is Test dye mixture III, CAMAG). A – the chromatogram within the frontal sample application; B – the chromatogram after the concentration with using the displacer; C – the chromatogram after the separation of the concentrated sample B; D - the chromatogram after the separation of the frontal chromatogram A.

The total concentration of phenols was determined when analyzing the solutions of the sewage waters before the purification and of the sewage waters after the purification. The total concentration of phenols was determined by the graduated diagram of changing of the analytical signal (the zone area, the peak height) (Table 1).

When using samples big in volume for the analysis (both for using the peak height as an analytical signal and of the zone area) the obtained concentration is closer to the concentration, which was independently determined by the colorimetric method. The error of determination makes up 6%, what is the sufficiently satisfactory characteristic for impurities identification.

Table 1. The determination of the total concentration of phenols in sewage waters of by-product coke industry.

Sample volume, µl	The total concentration of phenols, mg/l			Concentration determined colorimetrically for sewage waters after purification, mg/ml
	Sewage waters before purification	Sewage waters after purification	Determination error, %	
70	4.44±0.44	0.31±0.02	15	0.275
130	4.41±0.27	0.29±0.01	6	
70	5.03±0.50	0.32±0.02	17	
130	4.83±0.25	0.30±0.01	9	

3. Conclusion

When identifying the impurities in TLC it is expedient to use combined consecutive frontal, displacement and elution methods. In the given work the estimate of possibility of application of frontal-displacement method in circular TLC has been carried out on the example of impurities identification. Frontal-displacement-elution method of TLC allows separating and analyzing the diluted samples solutions, what permits to increase the sensitivity of determination, for example, of phenols impurities in water. It should be noted in addition that the approaches described by us are successfully applied in linear TLC, as well.

References

1. Zolotov Yu.A. O khimicheskom analize i o tom, chto vokrug nego (About chemical analysis and about that what surrounds it). Moscow: Nauka, 2004, 477 p. (In Russia)
2. Berezkin V.G. N.A. Izmailov i M.S. Shraiber: Otkrytiye tonkosloynoy chromatographii (N.A. Izmailov and M.S. Shraiber: The discovery of thin-layer chromatography). Moscow: GEOS, 2007, 128 p. (In Russia)
3. Berezkin V.G., Chausov A.V., Bannikova A.S. Zavodskaya laboratoriya, 2011, vol. 77, № 8 (In Russia) (in press).
4. Berezkin V.G., Chausov A.V. Russian Journal of Phisical Chemistry A, 2011, vol. 85, №10, p. 1814-1819.