



RUSSIAN ACADEMY OF SCIENCES

A.V. Topchiev Institute of
Petrochemical Synthesis



*The new variant of multidimensional
planar chromatography*

Prof., Dr. Victor Berezkin,

PhD student Svetlana Khrebtova

The main advantages of TLC

1. Simplicity;

2. Cost-effectiveness;

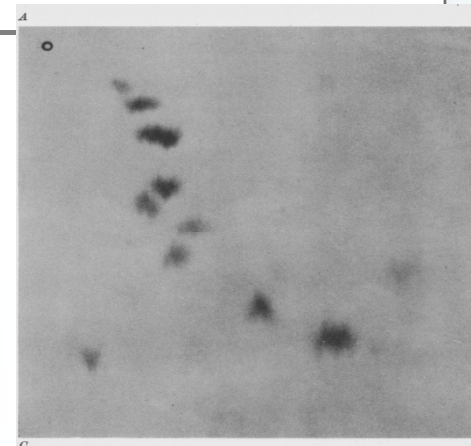
3. High selectivity

(the use of multiple mobile phases differing in their selectivity).

Development of two-dimensional planar chromatography

1944 – the first report on two-dimensional paper chromatography

R. CONSDEN, A. H. GORDON and A. J. P. MARTIN, *Biochem. J.*, **38** (1944) 224.

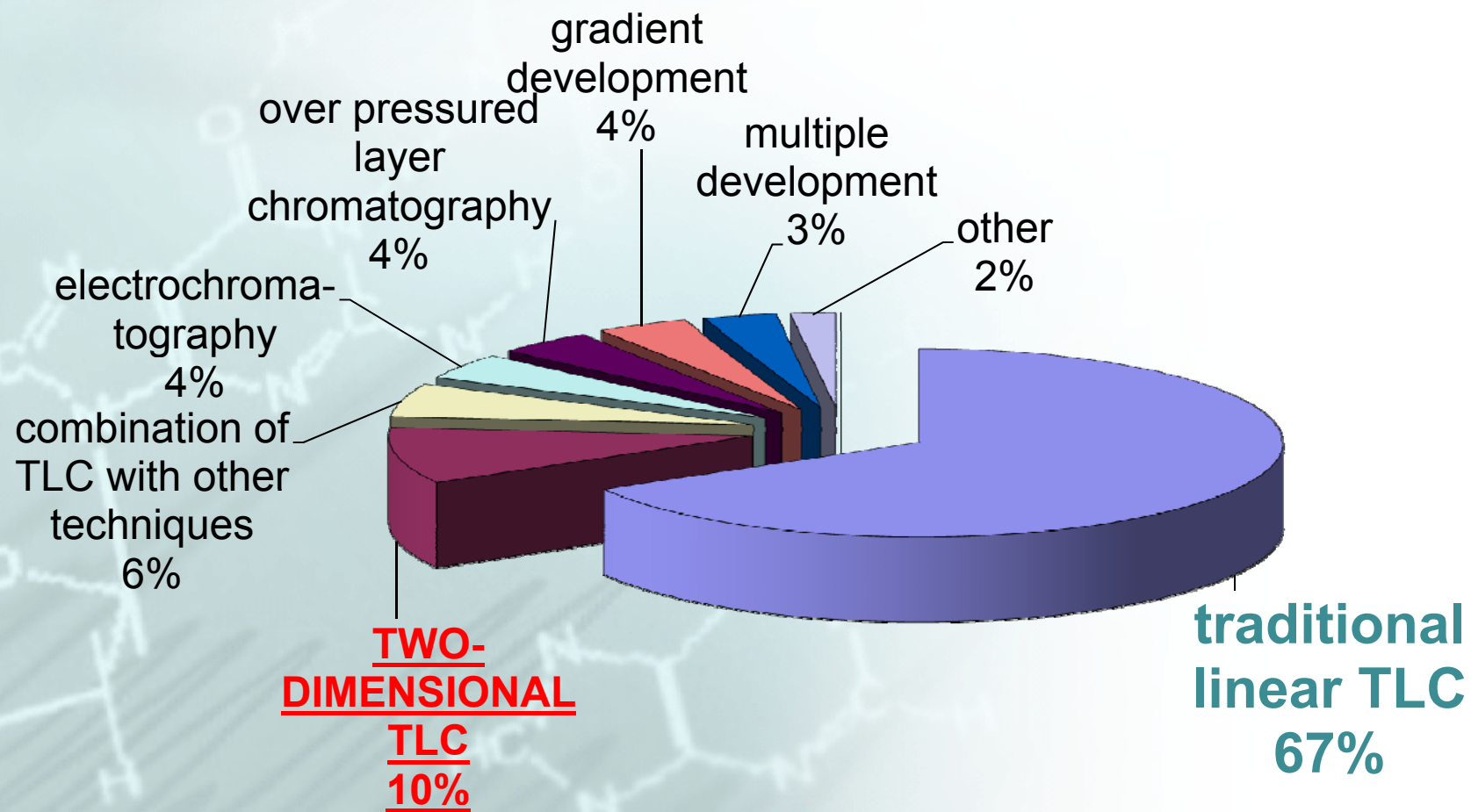


1951 – the first report on two-dimensional thin-layer chromatography

J.G.KIRCHER, J.M.MILLER, and G.L.KELLER,
Anal. Chem., **23** (1951) 420.

More than **700** published articles devoted to the realization of 2D-TLC by now

The structure of modern planar chromatography



V.G. Berezkin, S.S. Khrebtova. Evolution of Applied Planar Chromatography over the 1980-2010. *Journal of Planar Chromatography*, 2011, v. 24, №6 (in press).

Multidimensional (n-dimensional) chromatography

is one in which the components of a mixture, firstly, are subjected to two or more separation steps and, secondly, each separation step is implemented in various separation systems in which displacements depend on different factors [1,2].

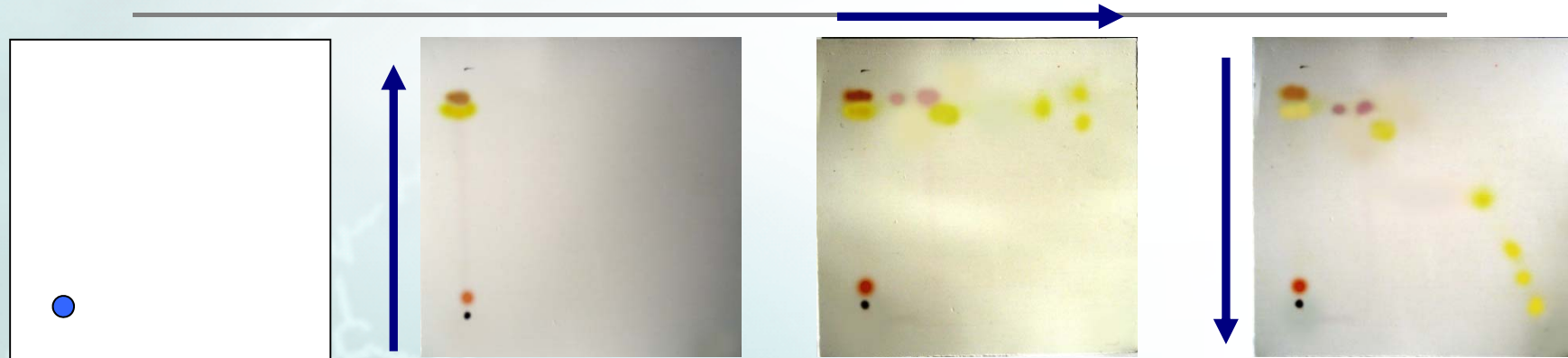
1. *J. C. Giddings, Use of multiple dimensions in analytical separations in multidimensional chromatography. In H.J. Cortes (Ed.) Multidimensional Chromatography Techniques and Application. Marcel Dekker. New York, 1990. pp. 1-27.*
2. *Sz. Nyiredy. Multidimensional Planar Chromatography. In L. Mondello, A.C. Lewis, K.D. Bartle, Multidimensional Chromatography. Chichester. John Wiley and Sons, 2002. pp. 171-197.*

The purpose of the work:

**to develop the new variants of n -dimensional
TLC**

**allowing to increase the velocity of multi-
dimensional TLC**

Implementation of three-dimensional TLC, based on known principles of two-dimensional planar chromatography



→ the direction of the mobile phase movement

“+”

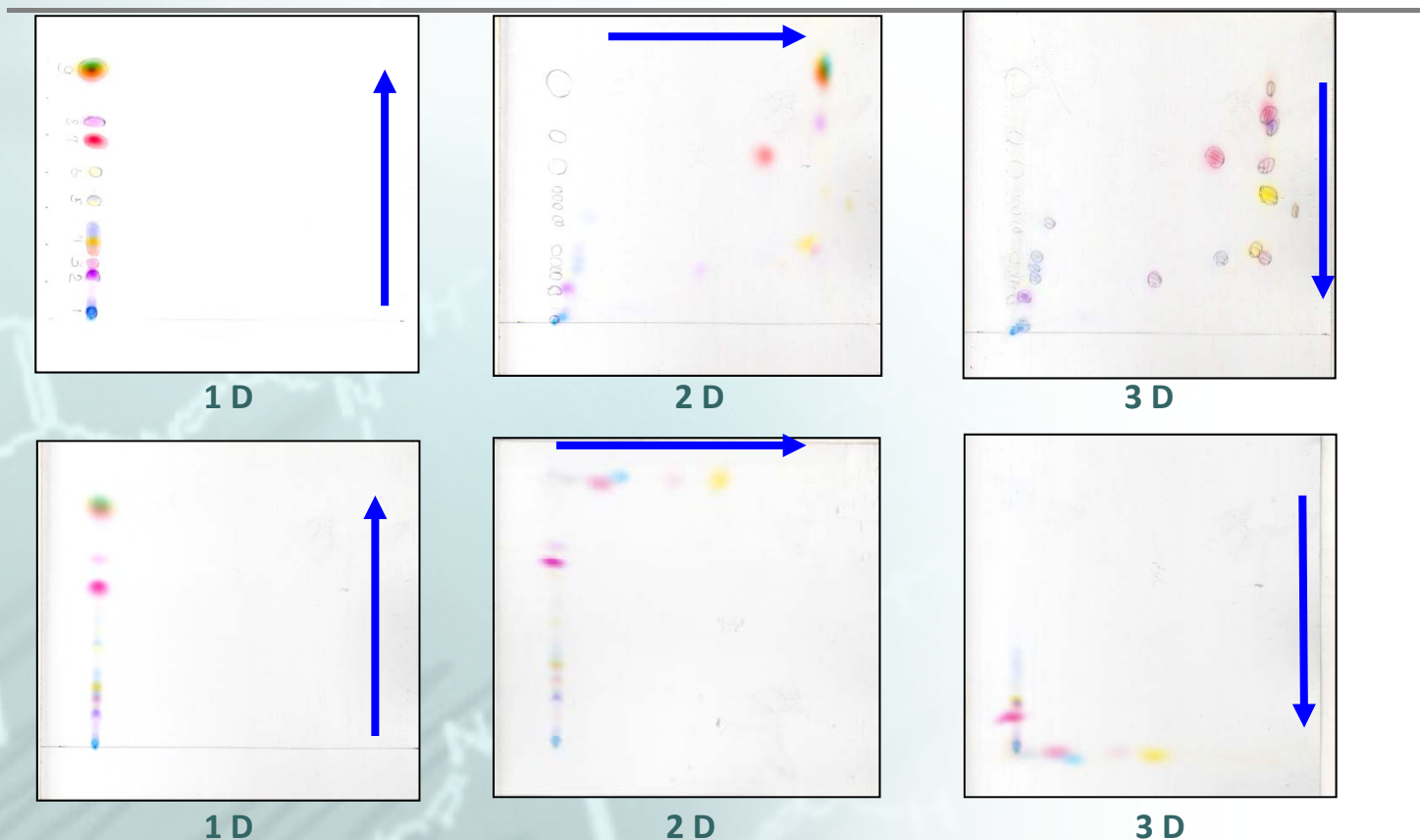
- ✓ high resolution;
- ✓ opportunity of the simultaneous determination of a large number of compounds

“-”

- ✓ the long duration of the analysis;
- ✓ the absence of a differentiated approach to the separation of a sample, and as a result, the overlapping zones of the test compounds

V.G. Berezkin, N.Y. Kulakova, S.S. Khrebtova. Three-Dimensional Thin-Layer Chromatography with open and closed adsorption layer. **Journal of Planar Chromatography**, 2009, v.22, №5, p.313–319.

Application of the standard variant of 3D-TLC to separation of a mixture



Some results:

The use of three-dimensional TLC allowed us to obtain only **17** of the possible **21** compounds

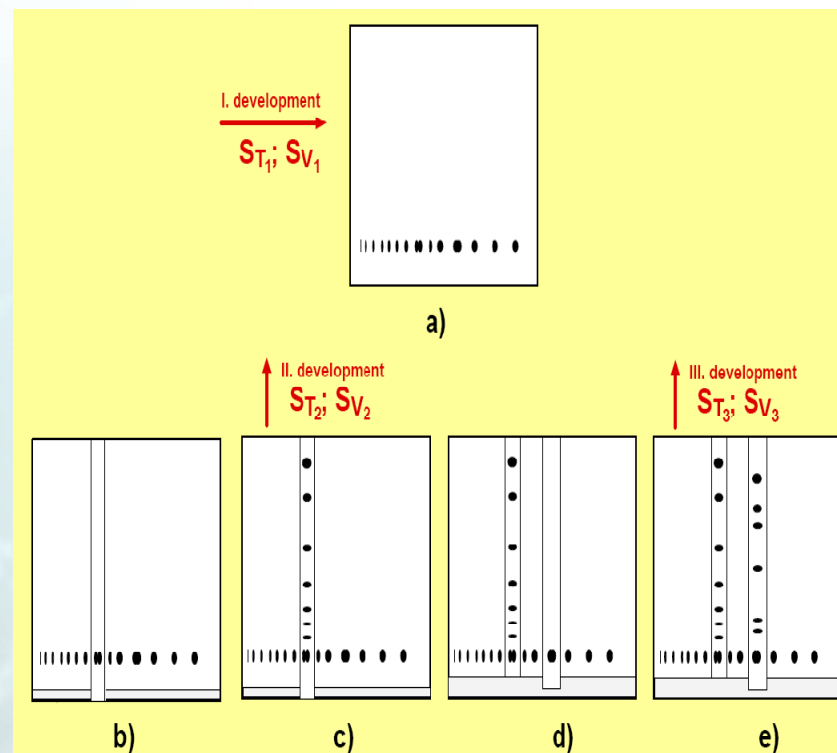
Targeted (selective) multidimensional TLC proposed by Sz.Nyiredy

The main idea:

The use of different mobile phases for the selective separation of each group of a test mixture

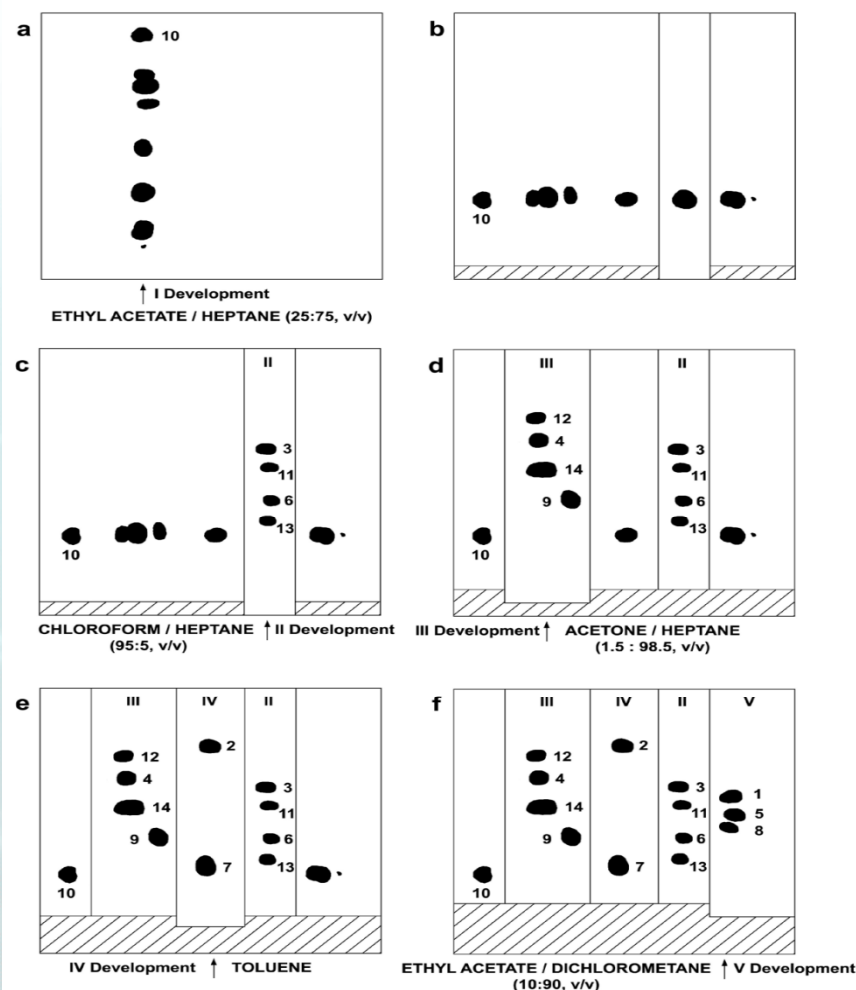
**For the separation of each group
the following operations should be
done:**

scraping part of the stationary phase to separate
one group from the others and then
carrying out the subsequent separation, and,
after every such operation, the length of the sorbent
decreases_

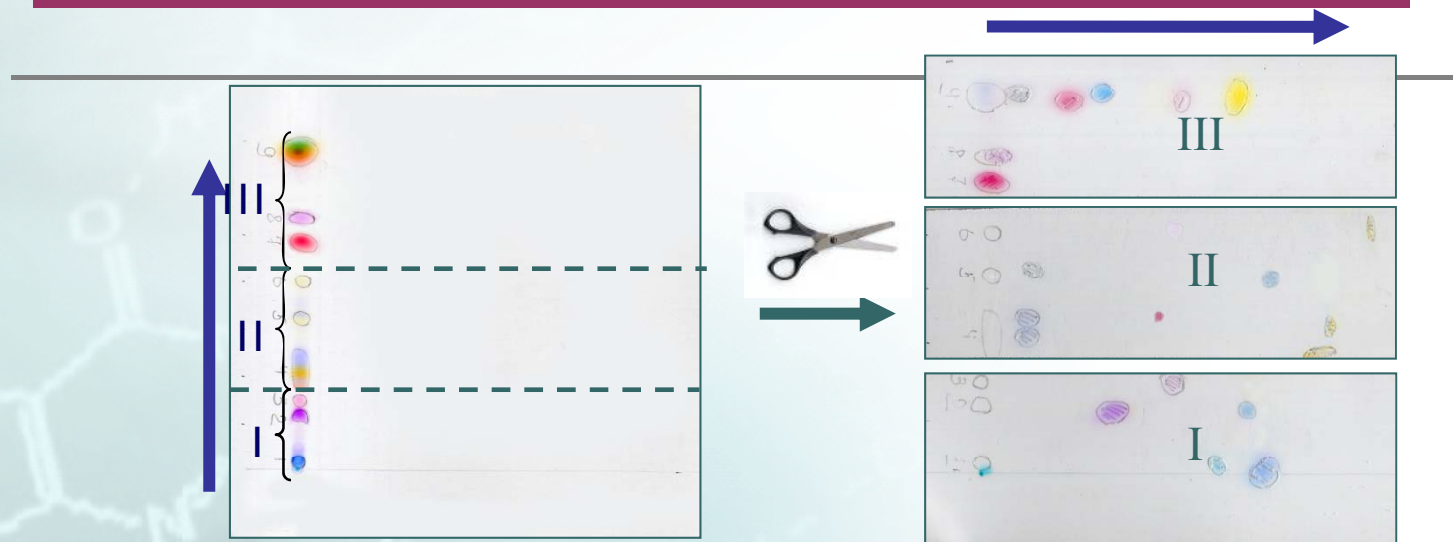


Multidimensional TLC proposed by Sz.Nyiredy was realized by T. Tuzimski

Disadvantages of the Nyiredy method :
Time-consuming and usage manual labor associated with the release of fractions



An approach to implementing rapid variants of n-dimensional TLC

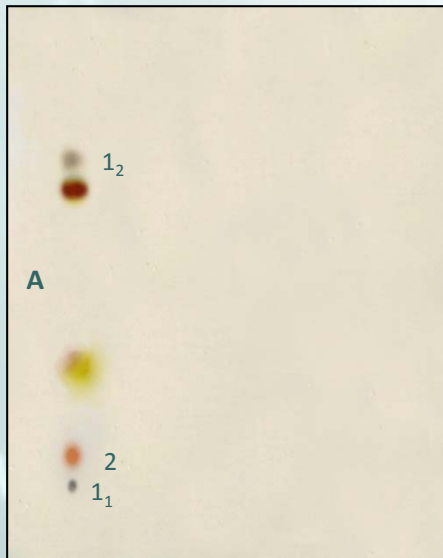


Stage 1: Initial separation of a multicomponent mixture;

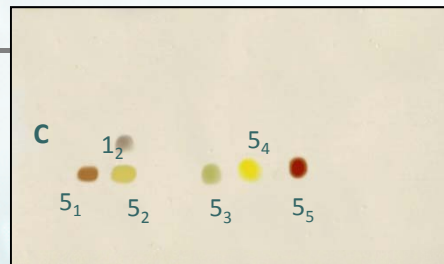
Stage 2: (**a**) Selection of groups of bad or unseparated compounds, and (**b**) cutting a plate into narrow strips containing selected groups in the direction perpendicular to the mobile phase movement in the first stage;

Stage 3: Simultaneous and independent separation of the selected fractions performed using various selective mobile phases.

Examples of the implementation of multidimensional separation

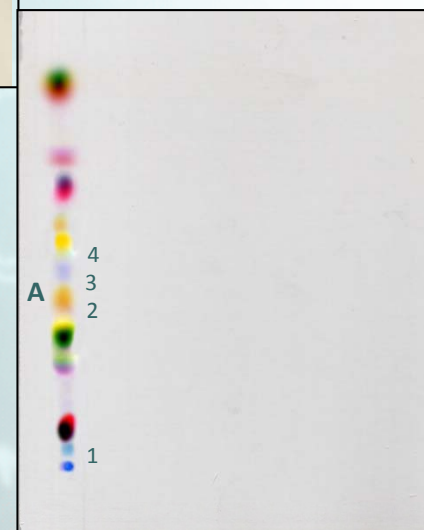


Separation of a pigments mixture of dyes for polymers by three-dimensional TLC

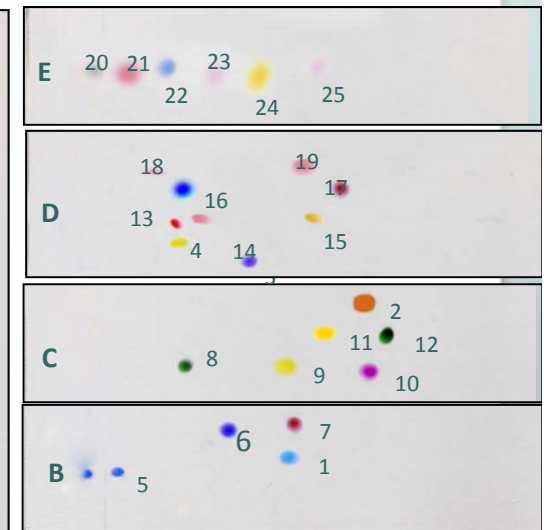


The total time of five-dimensional TLC - **74 min.**

The total time of 3D-TLC - **51 min.**
The time of 3D-TLC in the traditional way - **105 min**



Separation of a mixture of dyes by five-dimensional TLC



Advantages and disadvantages of the proposed method of multidimensional TLC

“ ”

—

“ + ”

- Increased speed;
- High resolution.

➤ The method is not universal:
it is necessary to use plates on a flexible support (polymer or aluminum).

V. G. Berezkin, S. S. Khrebtova, N. Yu. Kulakova. Four Dimensional Thin Layer Chromatography. **Doklady Physical Chemistry**, 2009, Vol. 429, Part 1, pp. 229–232.

V. Berezkin, S. Khrebtova, N. Kulakova. Four-Dimensional TLC on Plates with Open and Closed Adsorbent Layers. **Chromatographia**, 2010, v. 71, № 9-10, p. 907-911.

V.G. Berezkin, S.S. Khrebtova. The use of an Smin-chamber for implementation of two- and multidimensional TLC. **Mendeleev Communications**. 2011, v. 21, №.2, p.101-102.

Berezkin, V.G.; Khrebtova, S.S. The method of multidimensional planar chromatography **Patent RU** No. 2010118257/28, 07.05.2010.

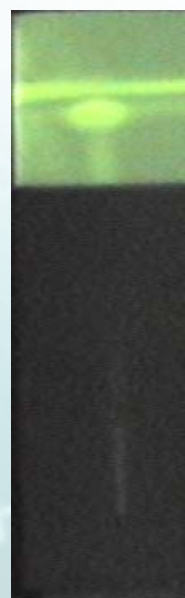
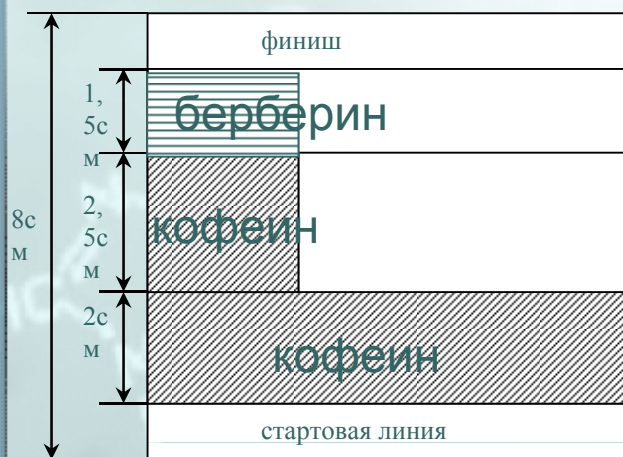
Conclusion

A new method of n-dimensional TLC was proposed and developed, which allows to implement accelerated multidimensional TLC.

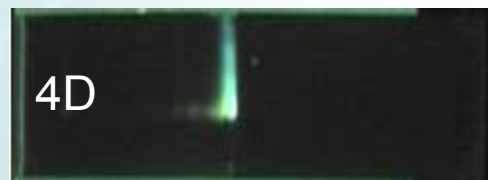
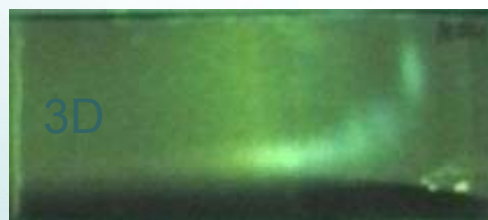


**Thank you
for your
attention!**

Разделение смолы пиролиза в результате 4-х мерного разделения в S_{min} -камере



1D



Ди-,дизамещенные и триароматические УВ

Алканы

Полициклические ароматические УВ, содержащие не меньше 4-х ароматических колец

Хроматограммы разделения n-алканов

