PRACTICAL APPROACH FOR TEMPERATURE-CONTROLLED TLC

Paweł K. Zarzycki

Section of Toxicology and Bioanalytics, Department of Civil and Environmental Engineering, Koszalin University of Technology, Śniadeckich 2,75-453 Koszalin, Poland

www.wilsig.tu.koszalin.pl/labtox/

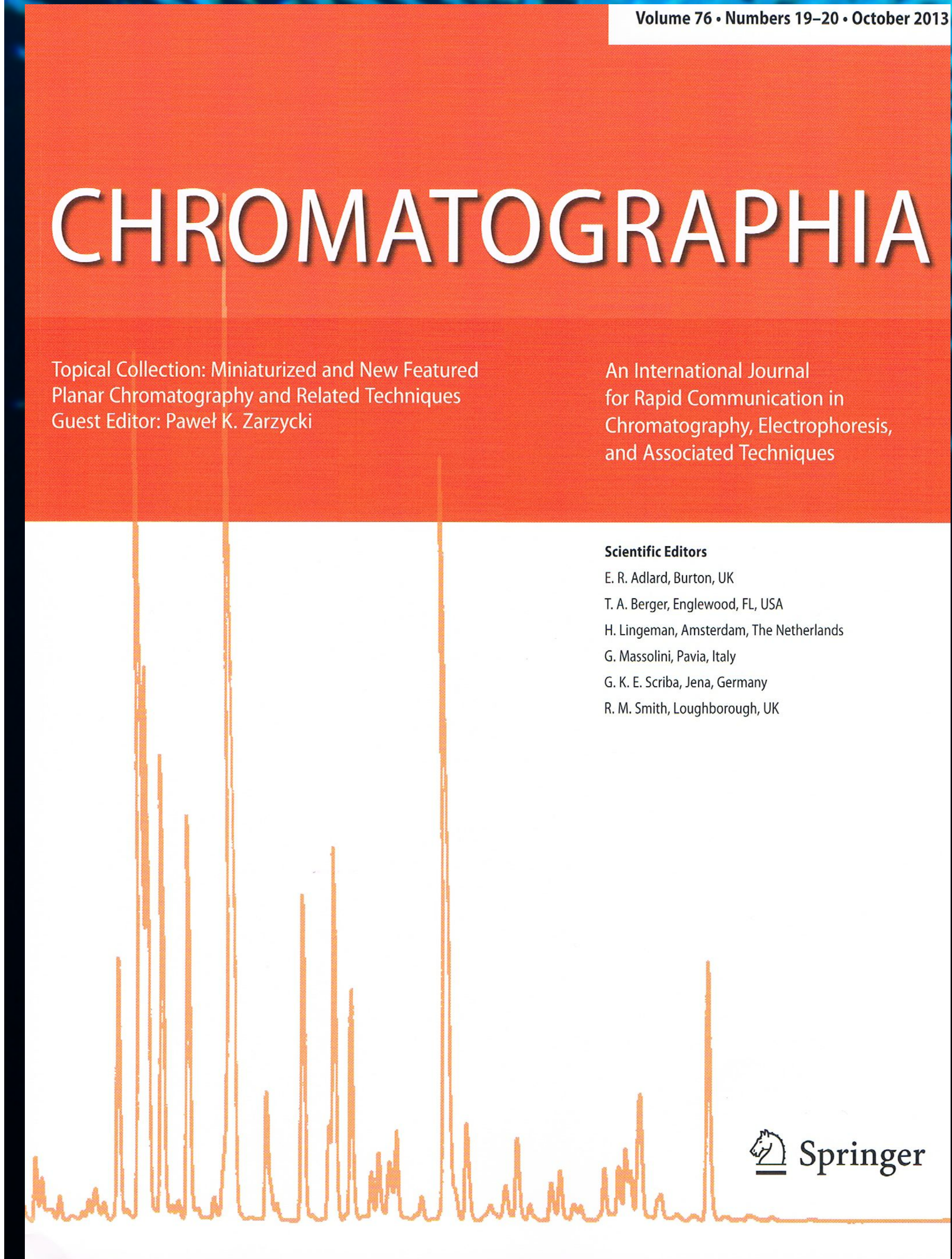
HPTLC 2014, Lyon, France

SECTION OF TOXICOLOGY AND BIOANALYTICS

KOSZALIN UNIVERSITY OF TECHNOLOGY POLAND

Professor Henryk Lamparczyk (1947-2012) successfully proposed an electrostatic retention index system common to GC, HPLC and TLC based on the experimental data involving silica, octadecyl-silica, arenyl-silica as well as liquid crystalline stationary phases. His work entitled "***The role of electric interactions in the retention index concept. Universal interaction indices for GLC, HPLC and TLC***" (Chromatographia 20, 1985, 283-288) is commonly recognized as the separation science milestone.

He was enormously generous with sharing, exchanging and discussing new research ideas and really appreciated any involvement of his co-workers. He never forced to be popular but always was clear in his opinions, statements and principles. Great passions of Professor Henryk Lamparczyk were history, architecture and music.



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Miniaturized and New Featured Planar Chromatography and Related Techniques

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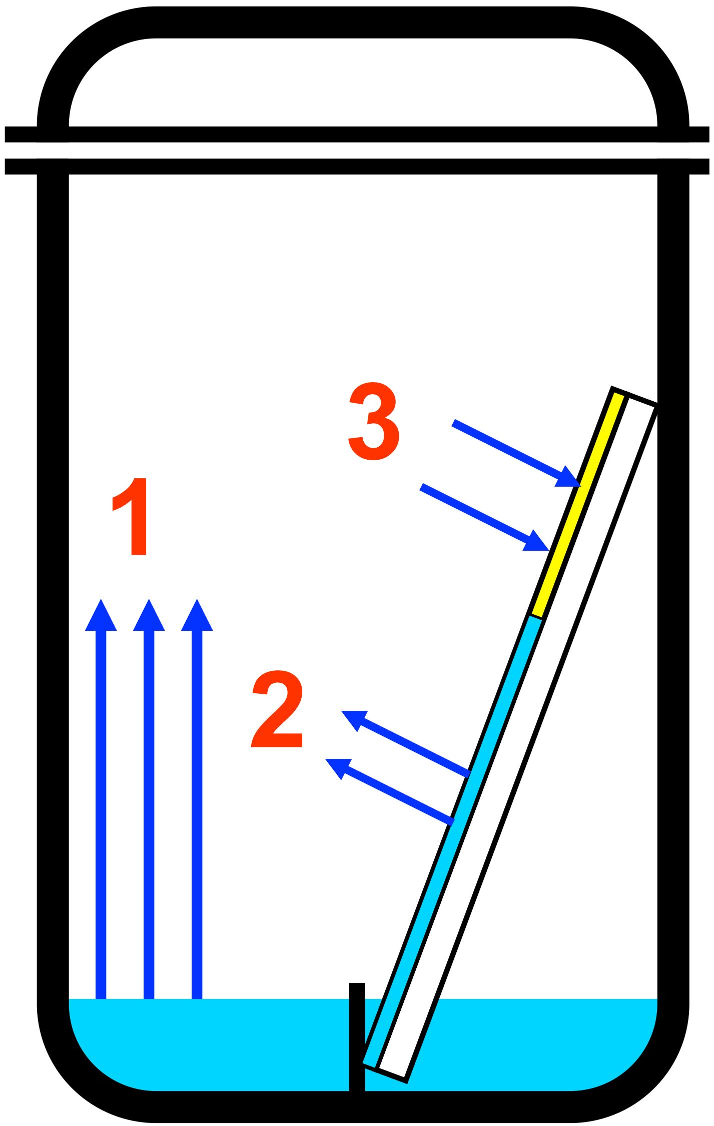
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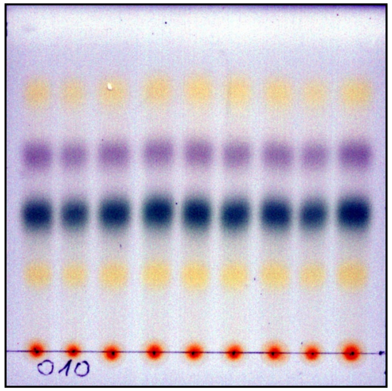
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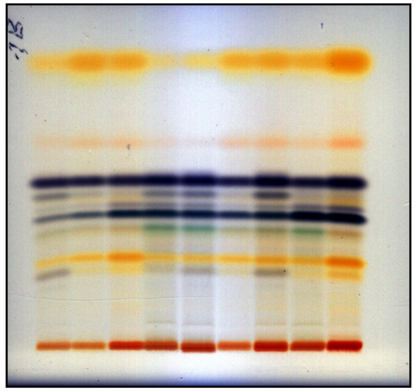
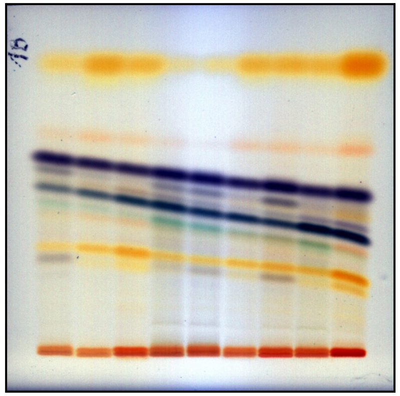
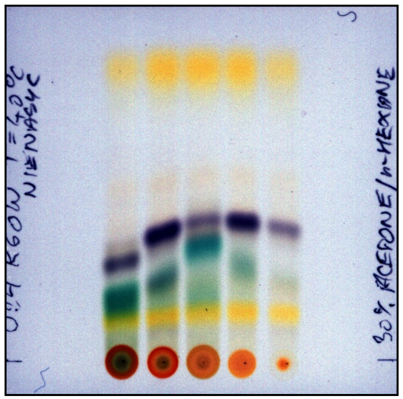
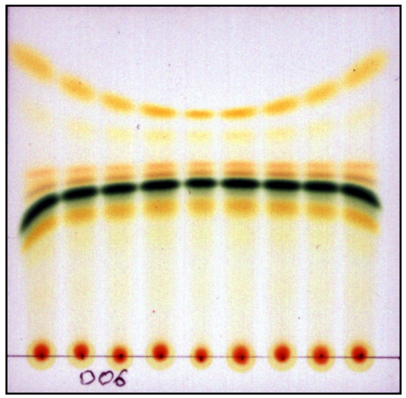





30% ACETONE/n-HEXANE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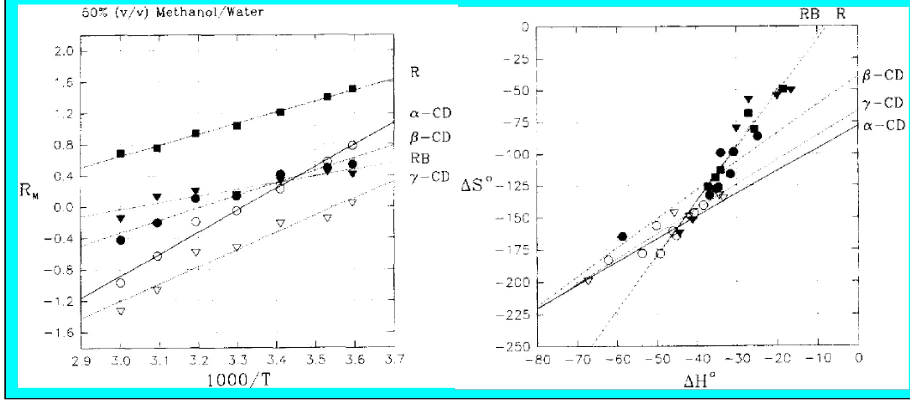
TEMPERATURE EFFECTS:

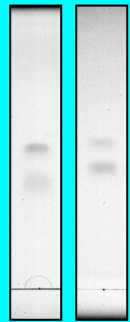
• SEPARATION

MACROCYCLES



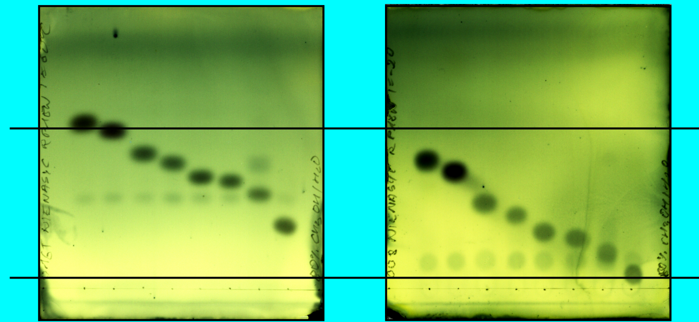
FULLERENES

-20°C +20°C



STEROIDS

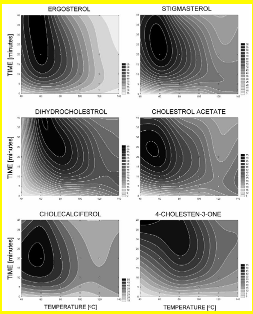
-20°C +60°C

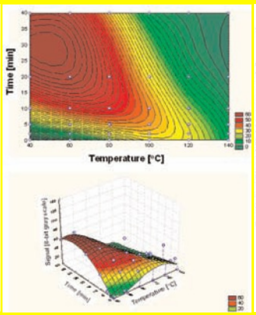


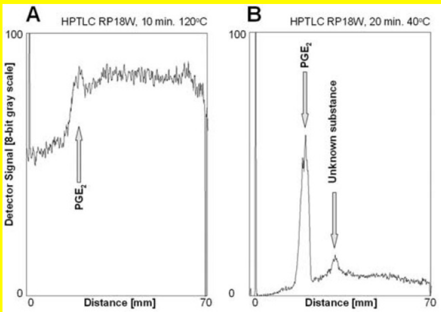
• DETECTION

CHEMICAL DERIVATIZATION

(*e.g.* optimization of PMA staining)







SPECTROSCOPIC MEASUREMENTS

(visit our poster presentations concerning active thermography method

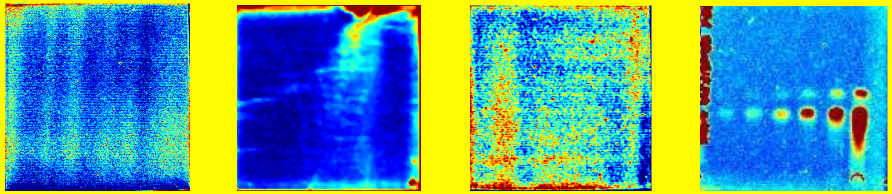
Z. Suszyński, M. Bednarek, P.K. Zarzycki;

THERMAL-WAVE INVESTIGATION OF PHYSICAL NON-UNIFORMITIES WITHIN TLC STATIONARY PHASES

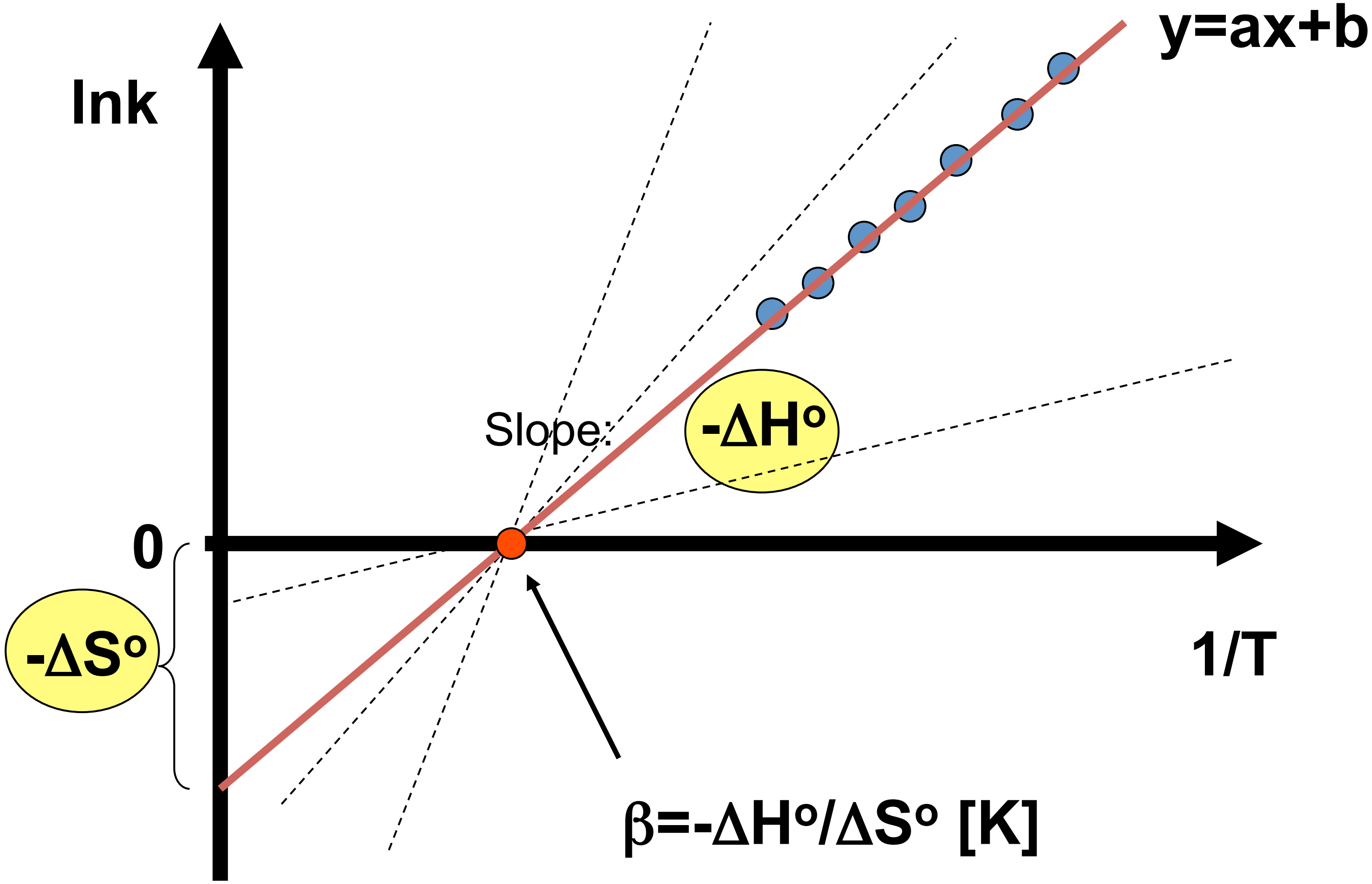
and

P.K. Zarzycki, R. Świta, Z. Suszyński

NEW ACTIVE THERMOGRAPHY METHOD FOR SENSITIVE DETECTION OF FULLERENES SEPARATED ON MICRO-TLC PLATES



$$\ln k = -\frac{\Delta H^o}{RT} + \frac{\Delta S^o}{R}$$



SYSTEM I ($t_{R\ CD} \approx t_0$)

Macrocyclic modifier:

Cyclodextrin

Mobile Phase: 30% CH_3CN/H_2O

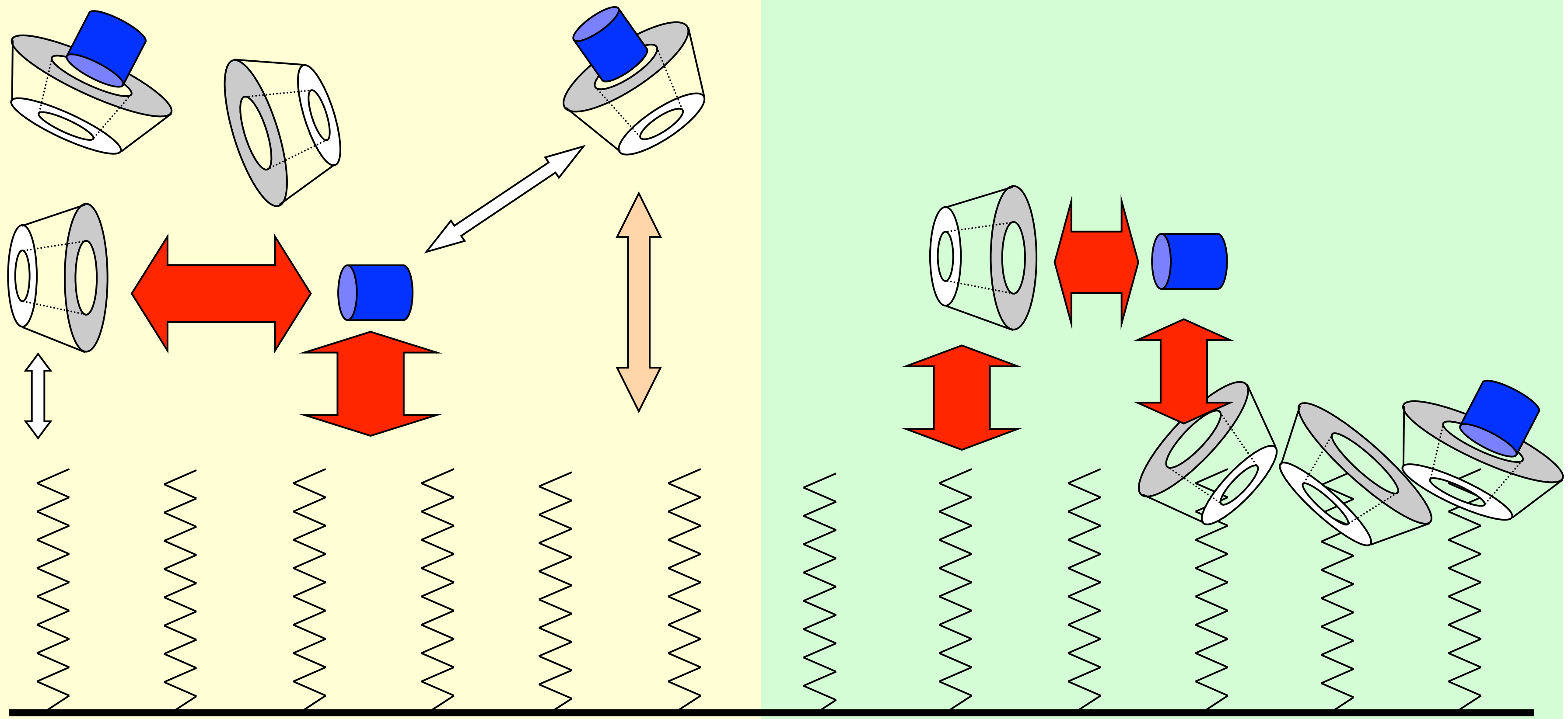
SYSTEM II ($t_{R\ CD} > t_0$)

Macrocyclic Modifier:

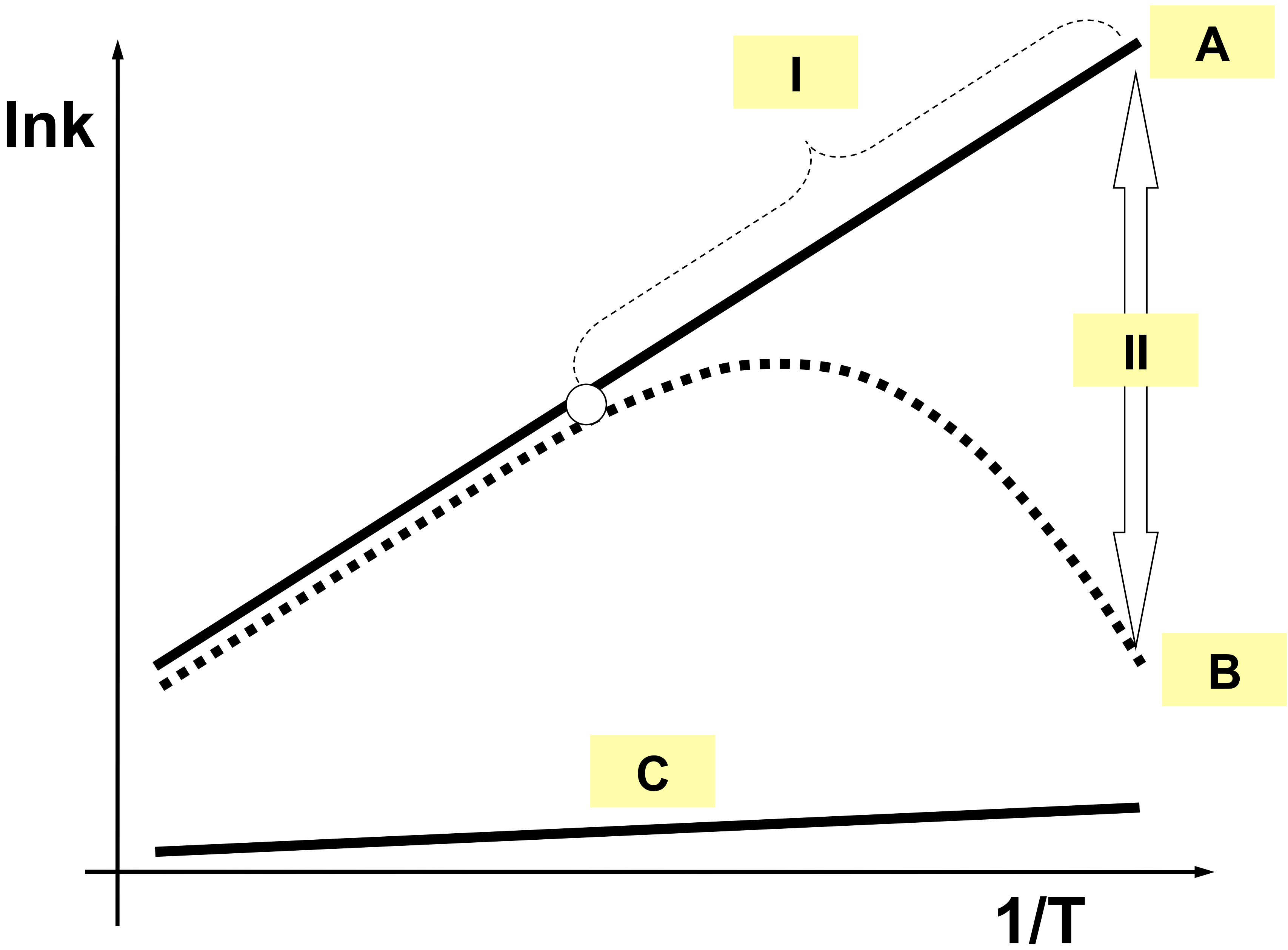
Cyclodextrin

Mobile Phase: < 5% CH_3CN/H_2O

$C_{CD,\ System\ I} >>> C_{CD,\ System\ II}$



Stationary Phase: C-18



lnk
I
A
II
B
C
1/T

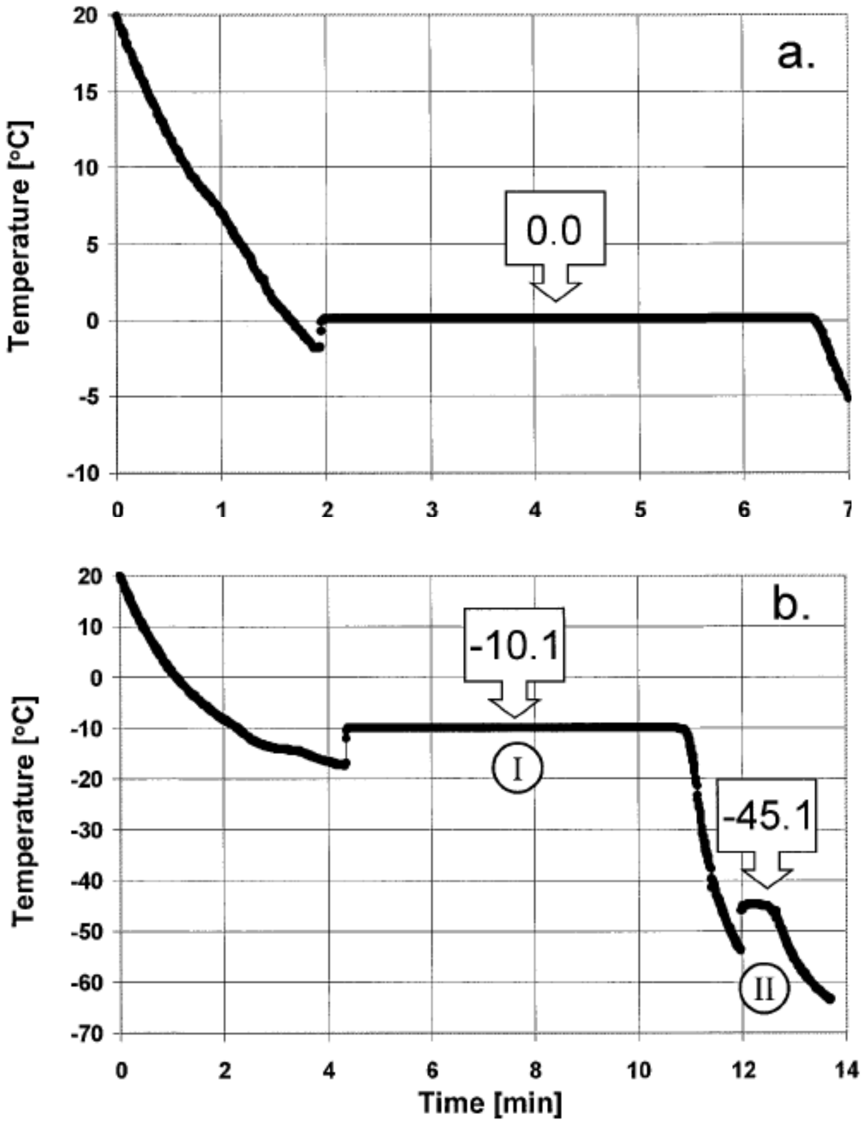


Fig. 1 Freezing temperature profiles of pure water (a) and an acetonitrile–water mixture at a concentration 35.6% v/v (b). The X-axis measurement error does not exceed ±0.3°C.

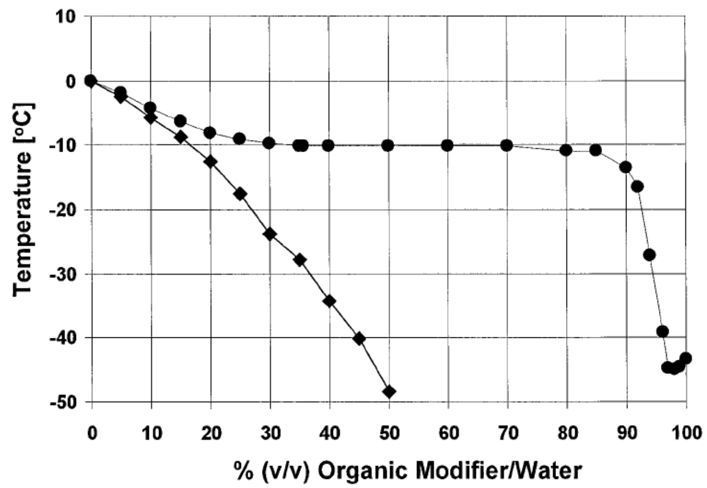
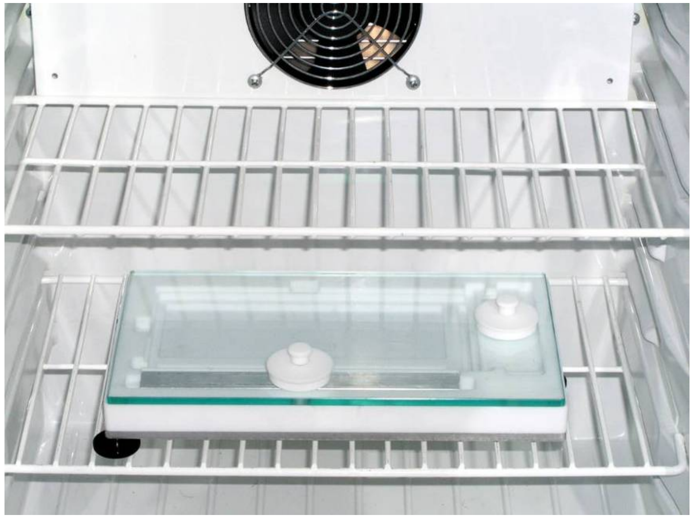
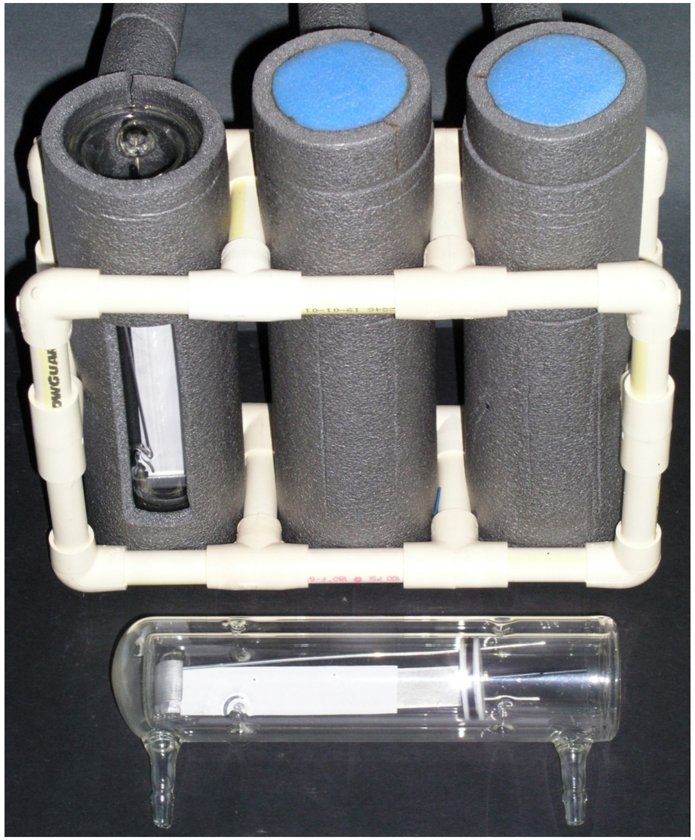


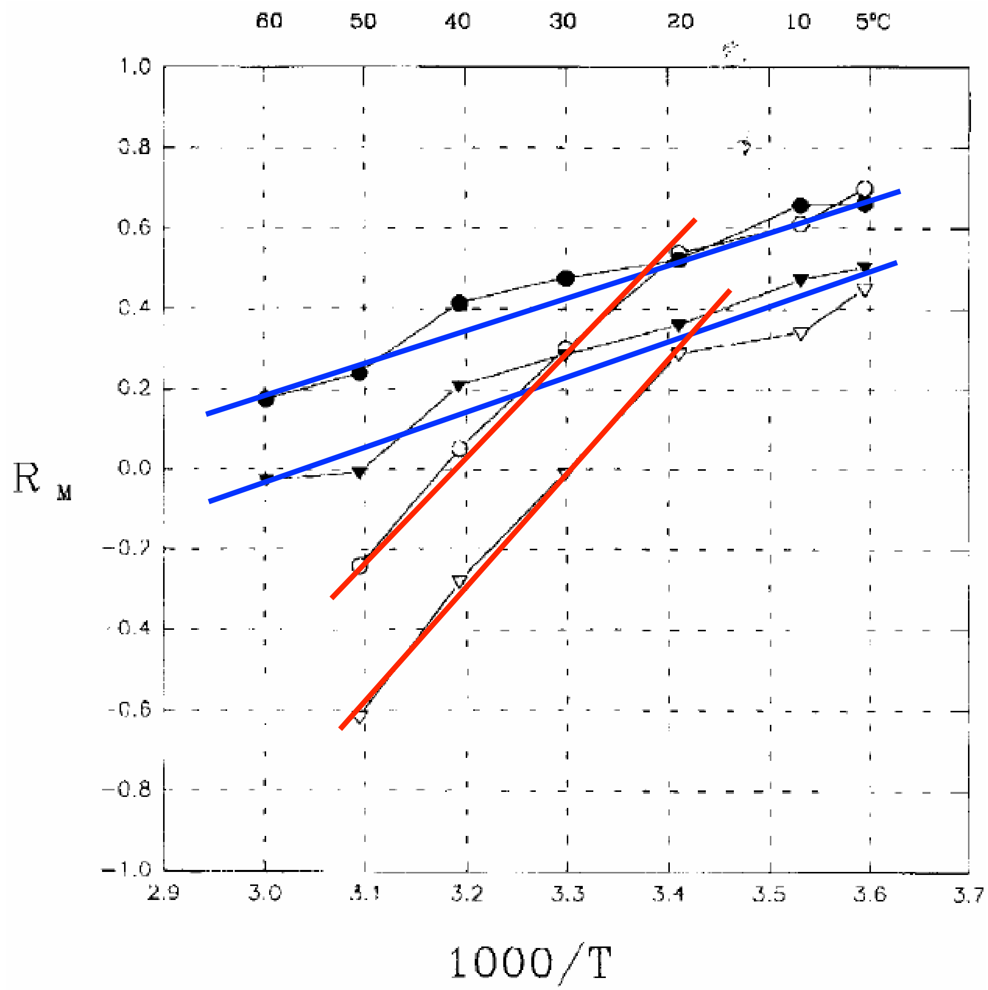
Fig. 2 Freezing temperature trajectories for binary mobile phases composed of methanol–water (diamonds) and acetonitrile–water (dots). The X-axis measurement error does not exceed ±0.3°C.

Adapted from:

P. K. Zarzycki, E. Włodarczyk, Da-Wei Lou, K. Jinno "Ewaluation of methanol-water and acetonitrile-water binary mixtures eluents for temperature-dependent inclusion chromatography" Analytical Sciences, 22 (2006) 453-456







Adapted from: P. K. Zarzycki. "Some Technical Problems Associated with Temperature-Controlled Thin-Layer Chromatography", J. Planar Chromatogr., 14 (2001) 63-65.

Adapted from: P.K. Zarzycki; „Simple horizontal chamber for thermostated micro-thin-layer chromatography"; Journal of Chromatography A, 1187 (2008) 250–259

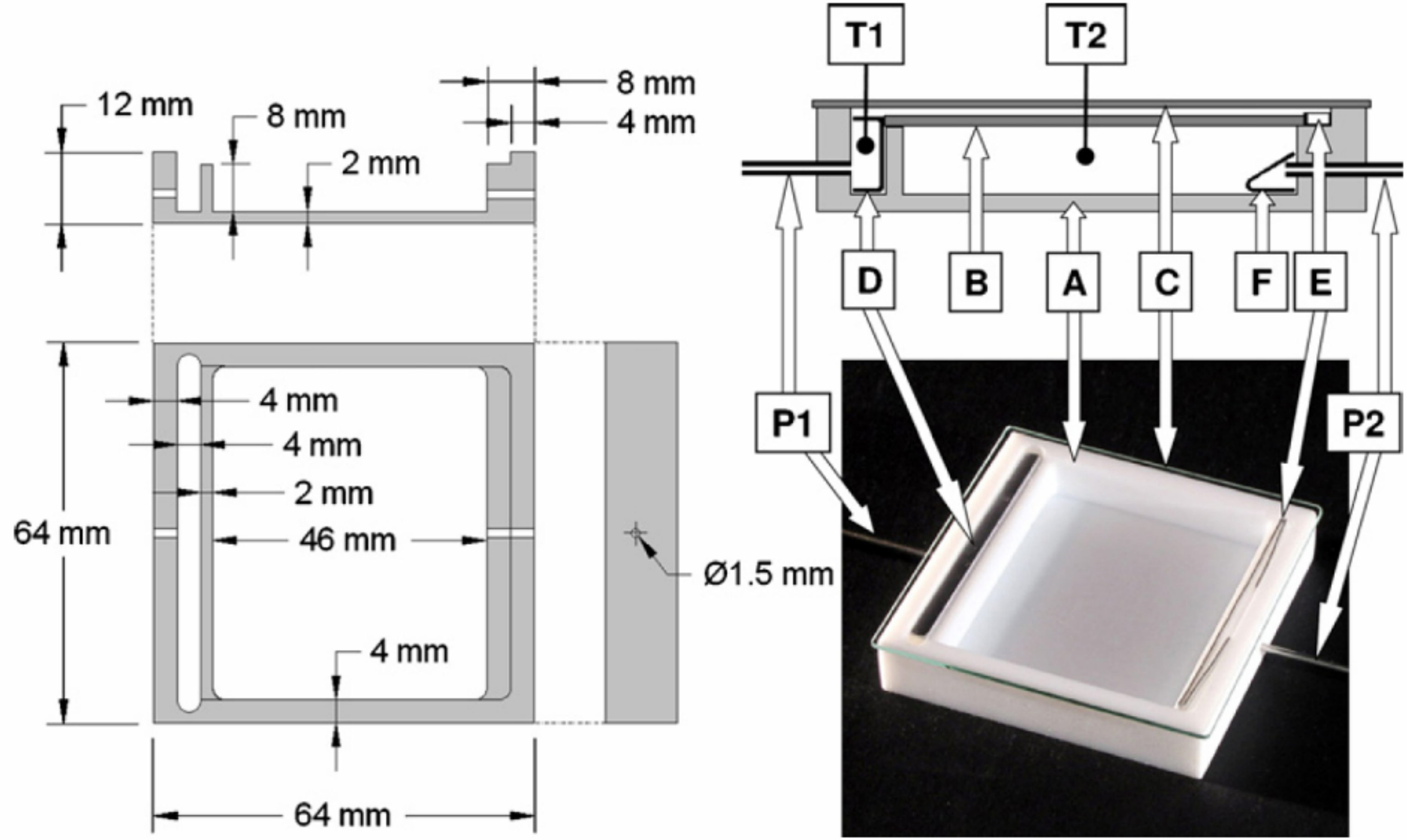
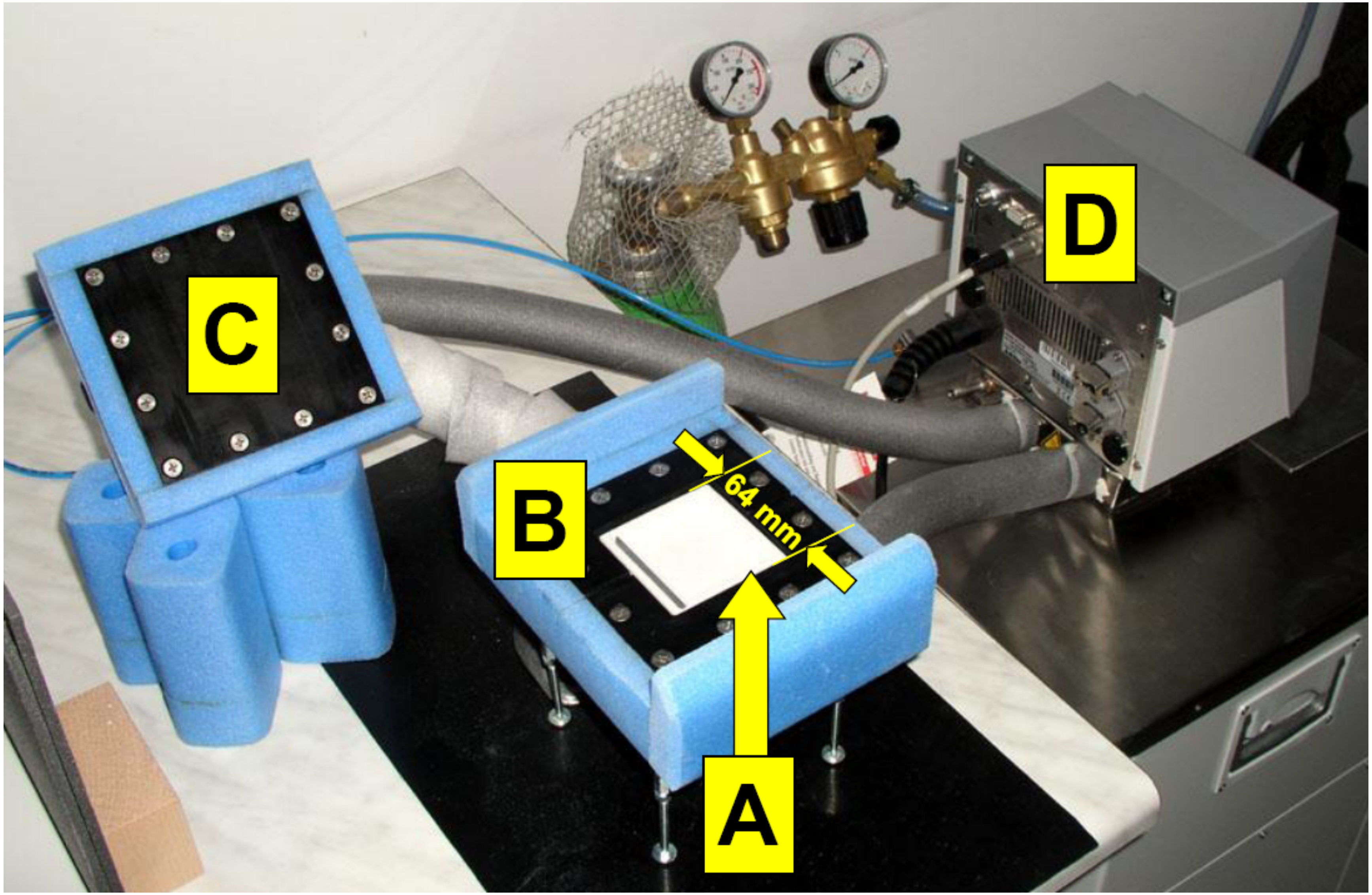


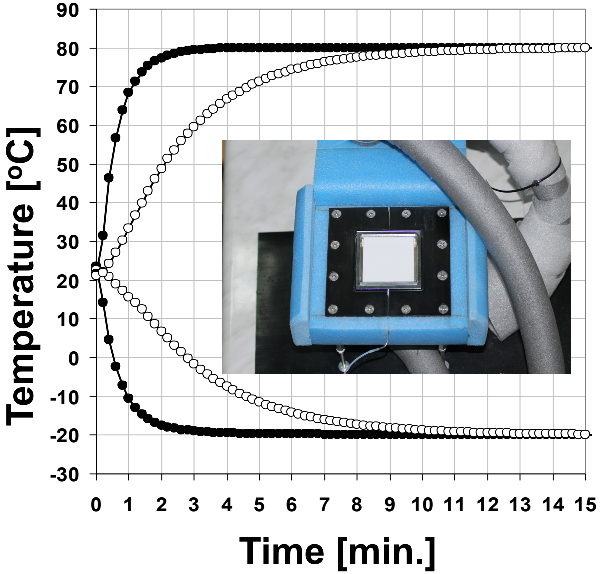
Fig. 1. Section drawing and perspective view of removable horizontal micro-TLC chamber unit. Chamber body (**A**), TLC plate (**B**), glass cover (**C**), eluent application bar (**D**), metal spring for the plate positioning (**E**), eluent splash protection bar (**F**), eluent application pipes (**P1**, **P2**), eluent and saturation trays (**T1**, **T2**).

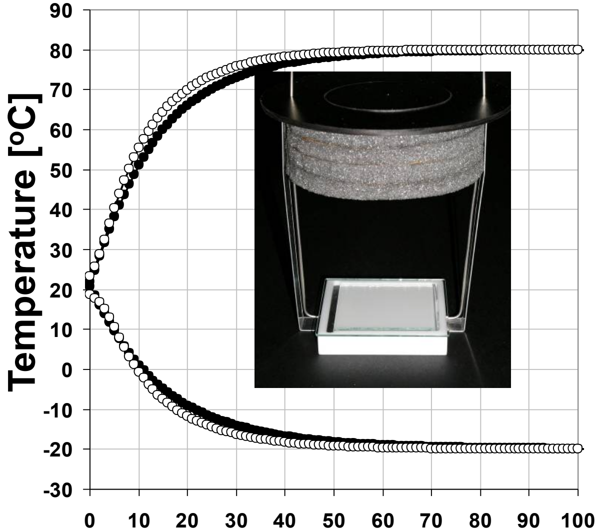


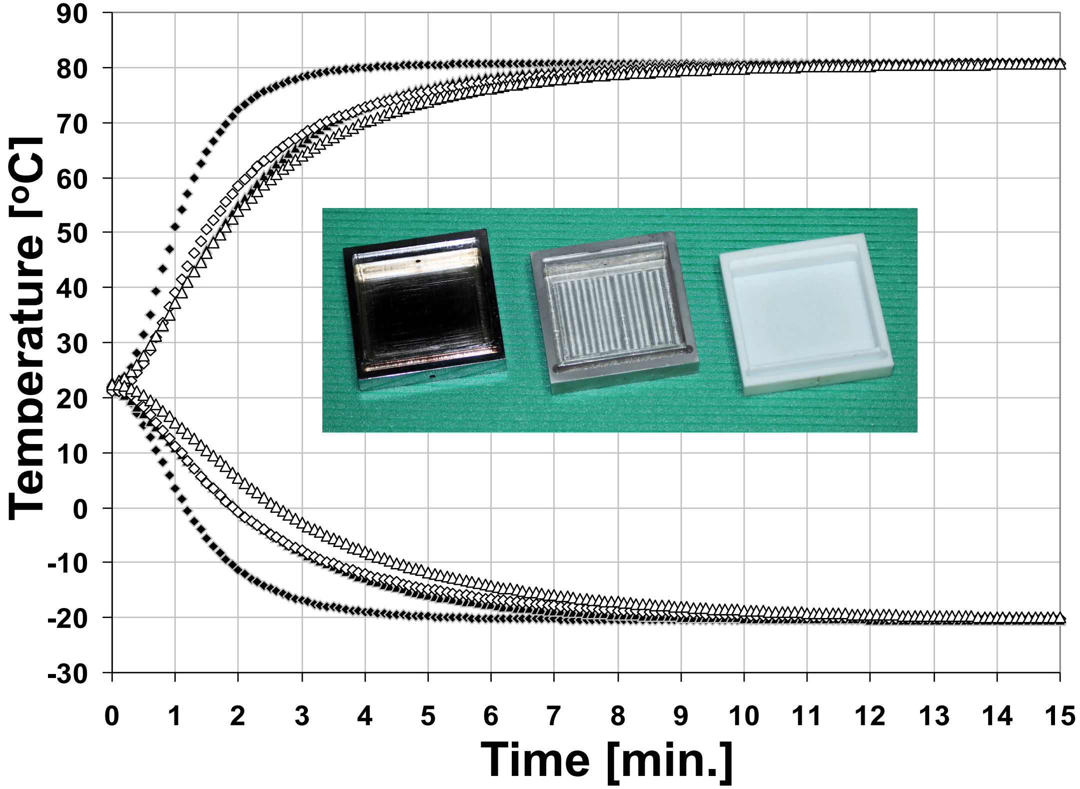
Perspective view of temperature controlled micro-planar chromatography device: horizontal chamber unit (A) working inside temperature controlled metal oven (B) equipped with movable cover (C) and connected to external liquid circulating thermostat (D).

Source: P.K. Zarzycki, M.M. Ślączka, M.B. Zarzycka, E. Włodarczyk, M.J. Baran; "Application of micro-thin-layer chromatography as a simple fractionation tool for fast screening of raw extracts derived from complex biological, pharmaceutical and environmental samples" Anal. Chim. Acta, 688 (2011) 168–174.

Adapted from: P.K. Zarzycki; „Simple horizontal chamber for thermostated micro-thin-layer chromatography”; Journal of Chromatography A, 1187 (2008) 250–259.







Heating and cooling plots for aluminium (diamonds) and glass (triangles) backed TLC plates measured inside PTFE (white symbols) and brass (black symbols) made units placed in the metal oven working with the external liquid circulating thermostat.

Heating and cooling plots for aluminium (diamonds) and glass (triangles) backed TLC plates measured inside PTFE (white symbols) and brass (black symbols) made units placed in the metal oven working with the external liquid circulating thermostat. Temperature equilibration rate of the PTFE and brass made micro-chamber units (circles and black dots, respectively) from room temperature to the oven temperatures of minus 20 and plus 80◦C. Micro-chamber units were thermostated inside the metal oven working with the external liquid circulating thermostat (A)and submersible gravity convection container placed in thermostated water bath (B).

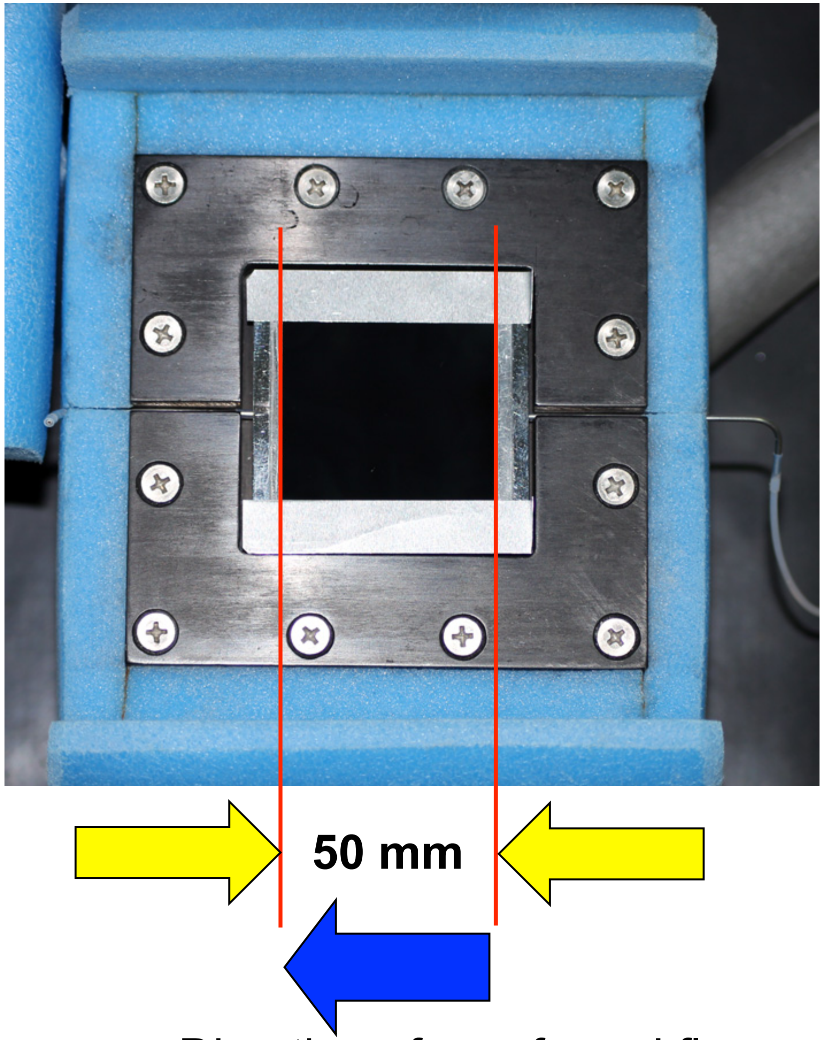
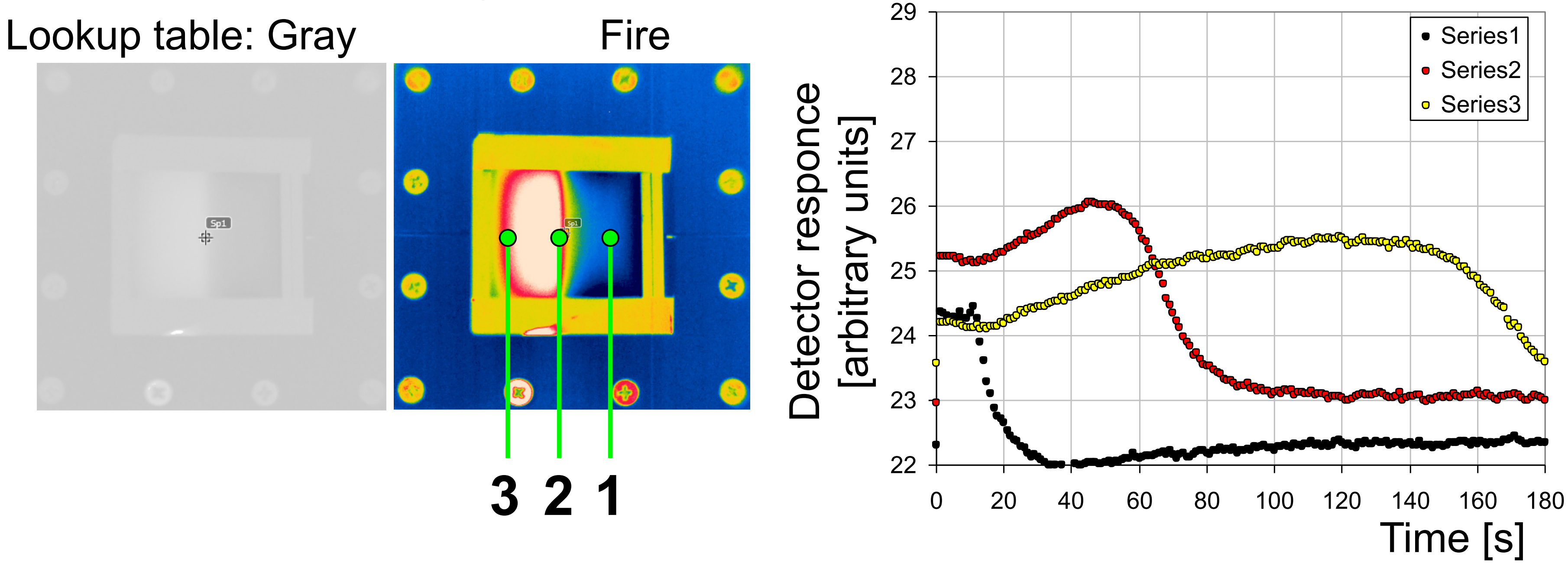
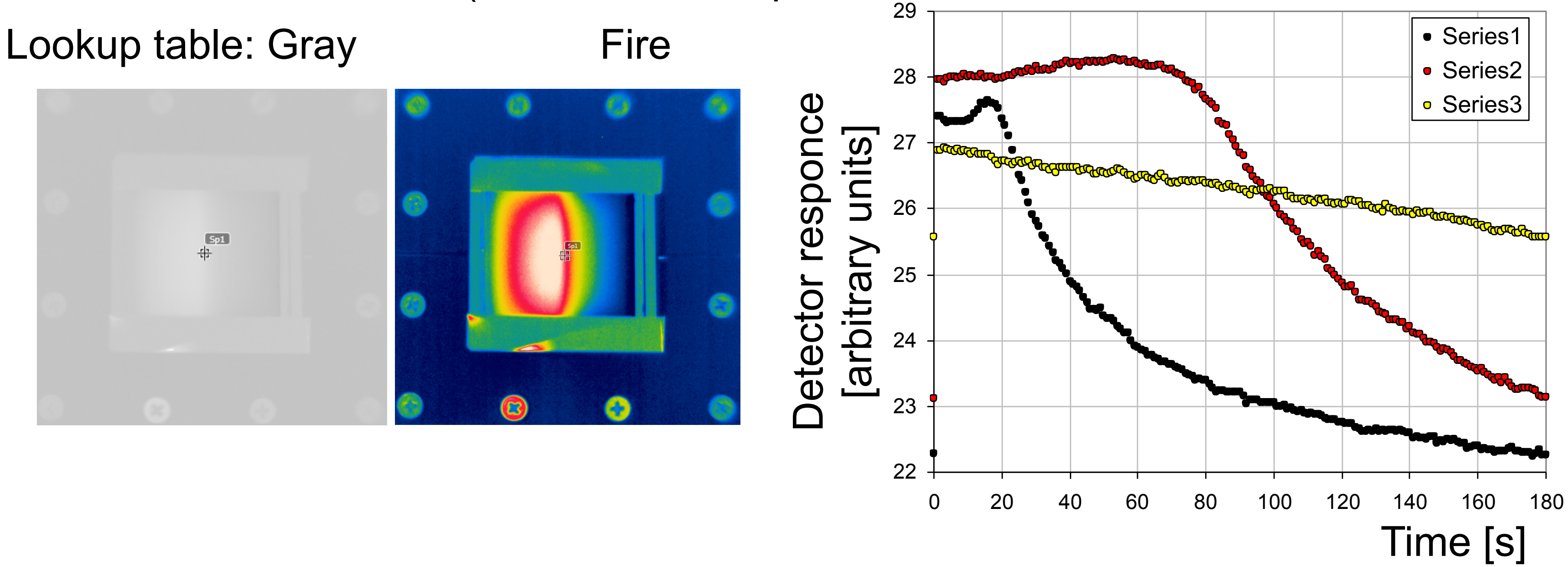


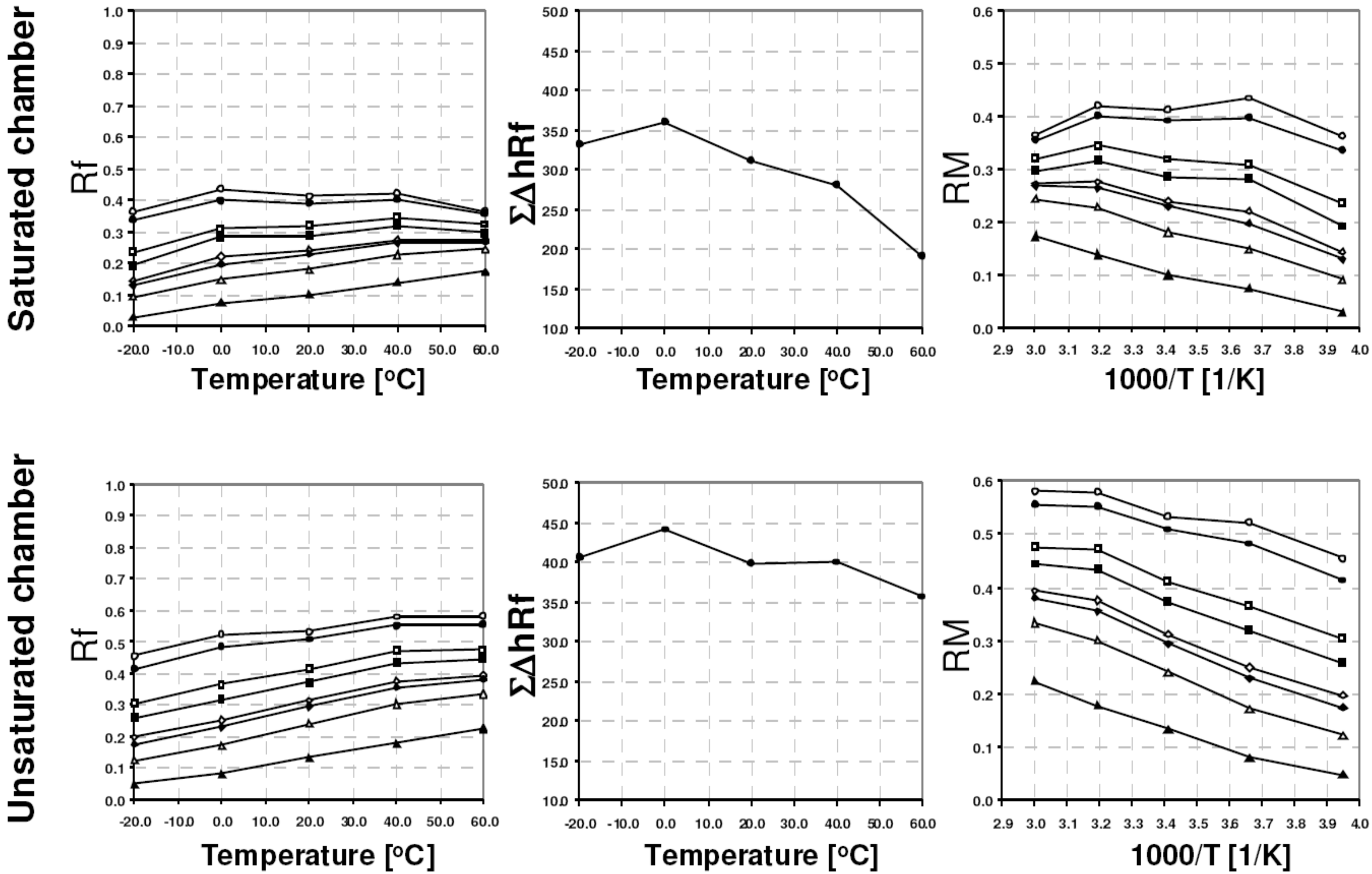
Plate: TLC glass based silica gel 60

Mobile Phase: *n*-Hexane (T=20^{o}C; Development time = ~3 min.)



Mobile Phase: Methanol (T=20^{o}C; Development time = ~6 min.)





Adapted from: P. K. Zarzycki, M. B. Zarzycka; „Application of temperature-controlled micro planar chromatography for separation and quantification of testosterone and its derivatives"; Anal. Bioanal. Chem.; Published online 13 March 2008; DOI 10.1007/s00216-008-1919-x

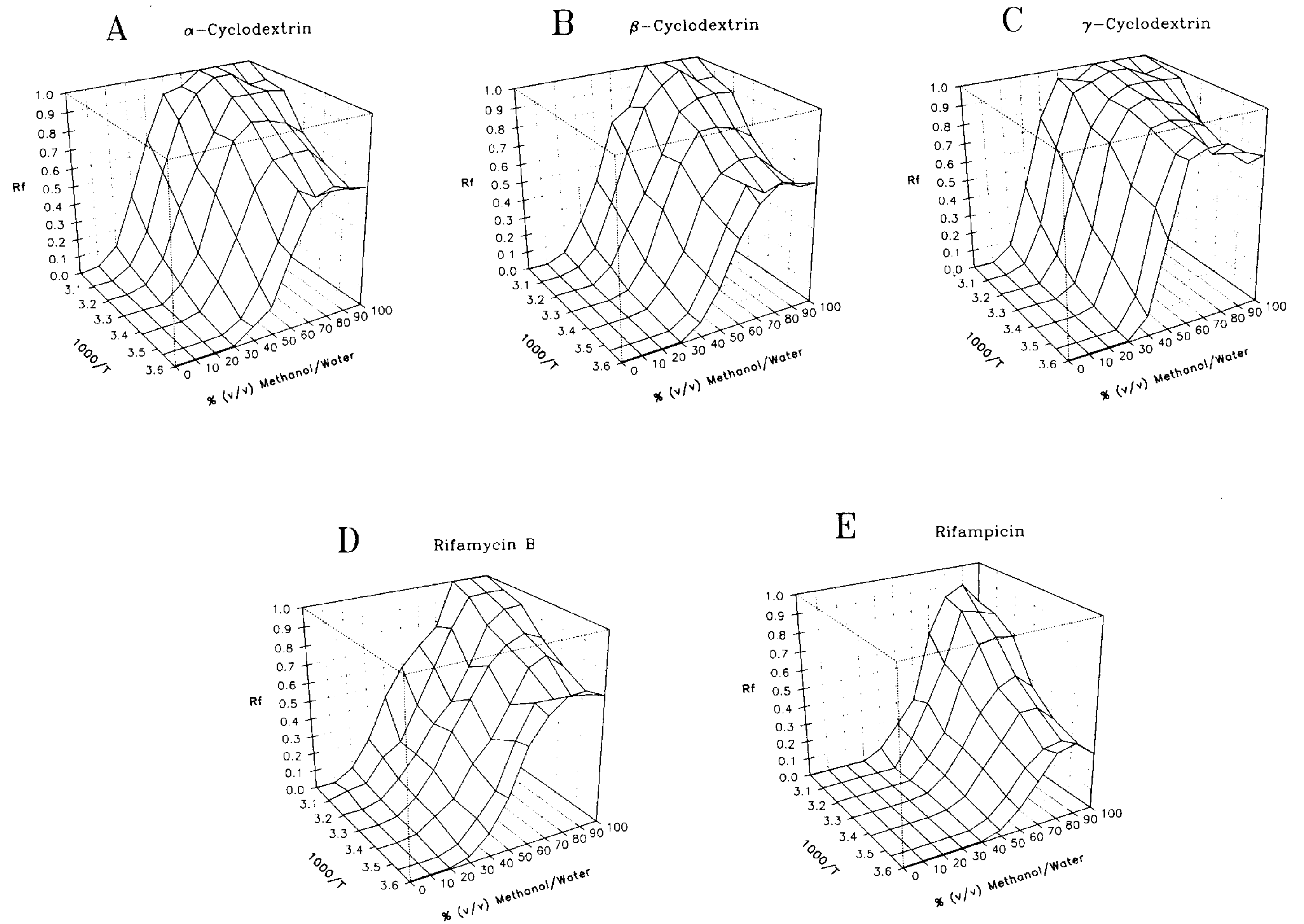
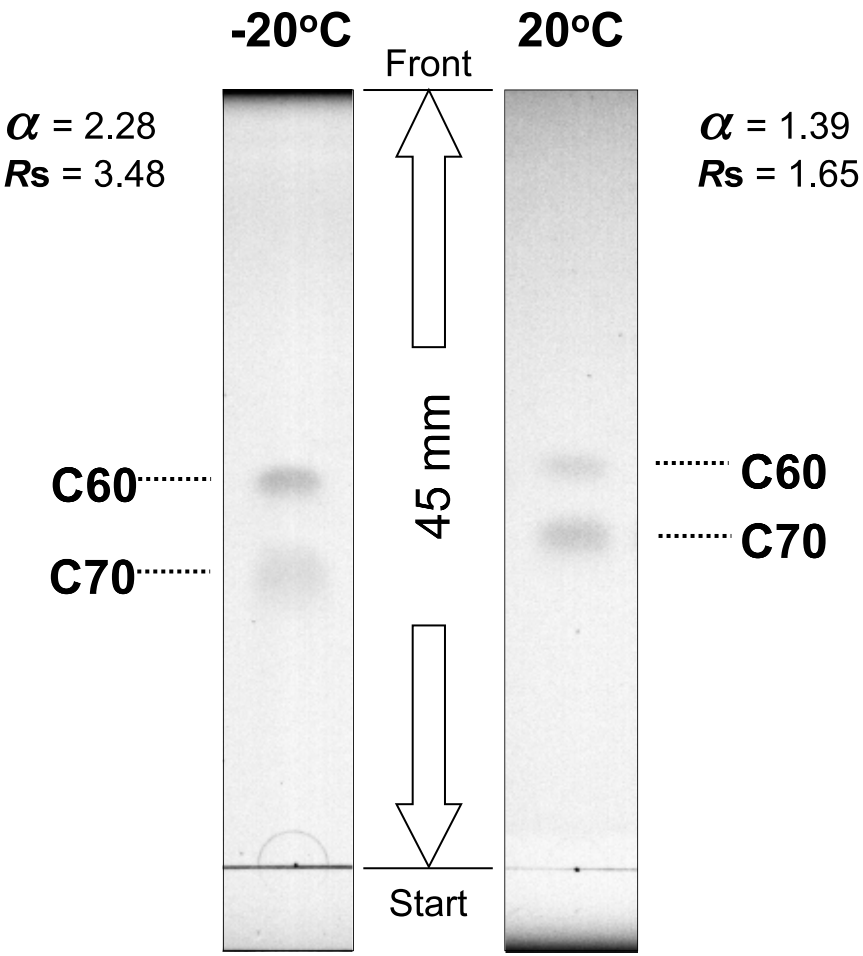


Fig. 1. Relationships between R_F values of α- (A), β- (B), and γ-cyclodextrin (C), rifamycin B (D) and rifampicin (E) versus different mobile-phase compositions and reciprocal of absolute temperature.

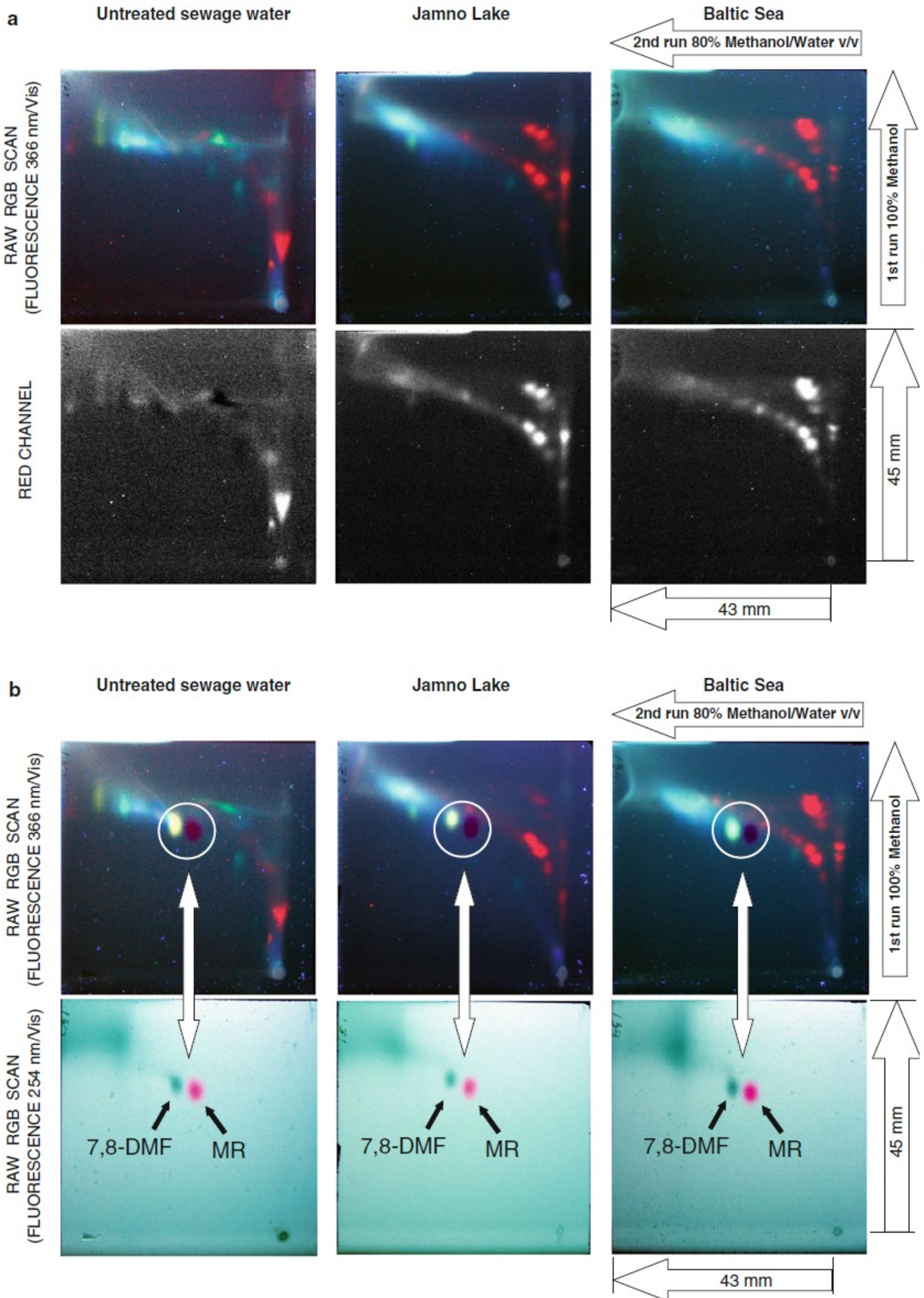
Adapted from:
P. K. Zarzycki, J. Nowakowska, A. Chmielewska, M. Wierzbowska, H. Lamparczyk, "Thermodynamic study of retention of selected macrocycles using RP-HPTLC plates and methanol/water mobile phases", *J. Chromatogr A.*, 787 (1997) 227-233.

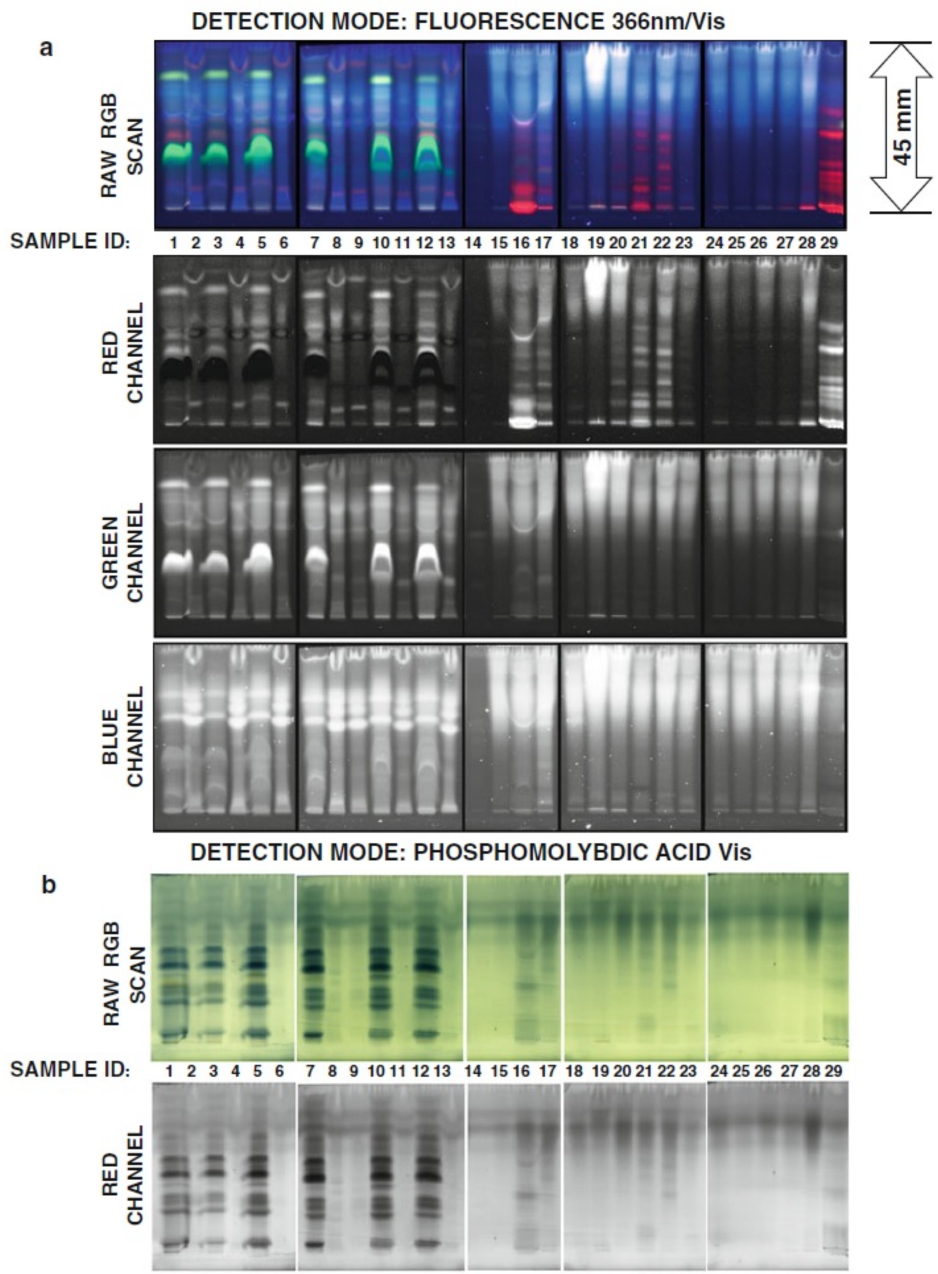
Separation of C60 and C70 fullerenes at different temperatures (−20 ◦C left, +20 ◦C right). Chromatographic conditions: stationary phase: HPTLC RP18WF254; mobile phase: 100% *n*-hexane; chamber saturation mode: unsaturated; sample application mode: manual (micro-syringe); sample volume: 1.0L; analytes mass: 200 ng/spot (C60), 50 ng/spot (C70); plate temperature equilibrium before developing: 15 min; developing time: 8 min (−20.0 ◦C), 5 min (+20.0 ◦C); spots visualization method: direct scan under visible light conditions.

Temperature:

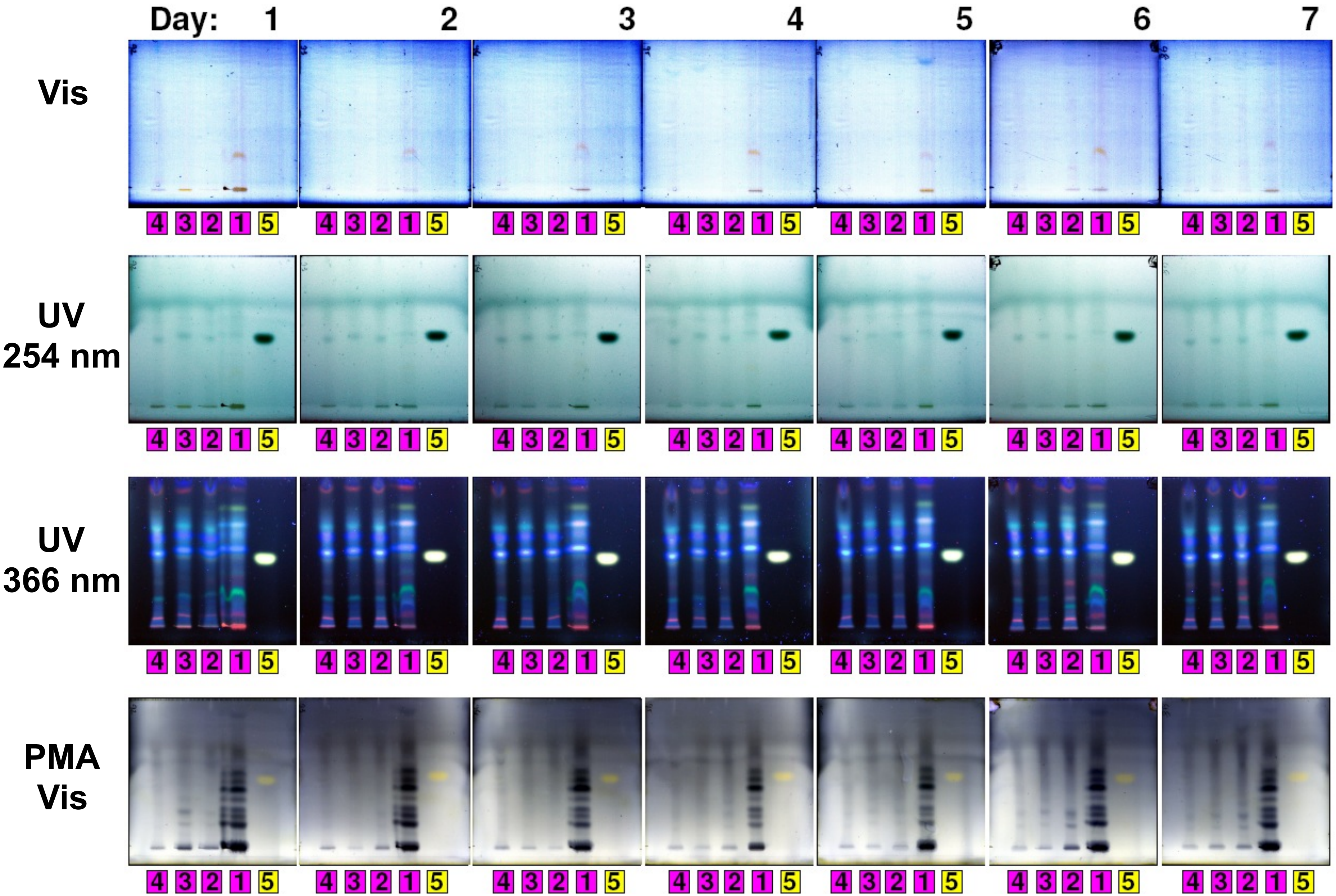


Adapted from: P.K. Zarzycki; „Simple horizontal chamber for thermostated micro-thin-layer chromatography"; Journal of Chromatography A, 1187 (2008) 250–259



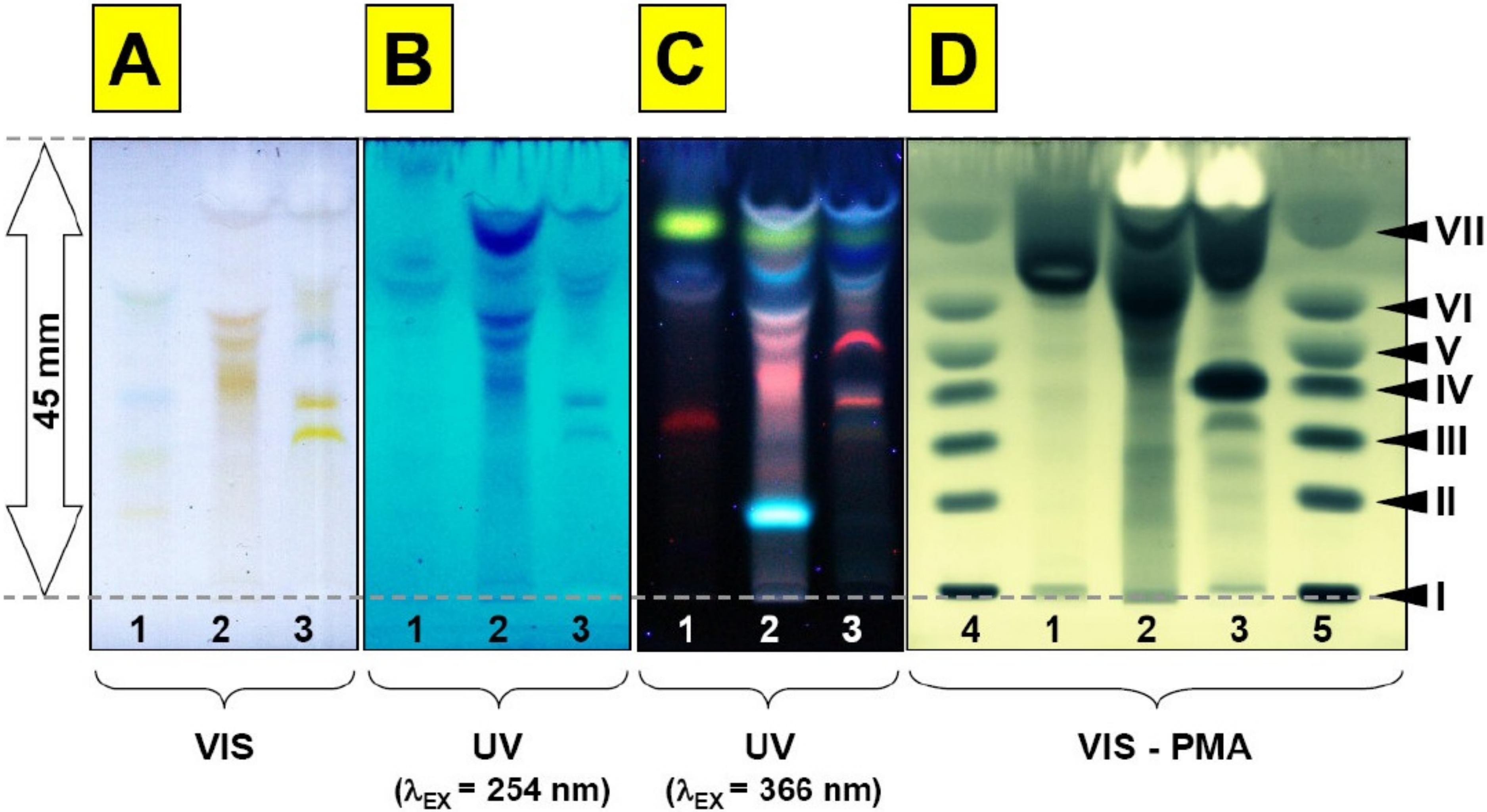


Source: P.K. Zarzycki, M.M. Ślączka, E. Włodarczyk, M. J. Baran; “Micro-TLC approach for fast screening of environmental samples derived from surface and sewage waters”; Chromatographia (2013) published online DOI 10.1007/s10337-013-2445-3.

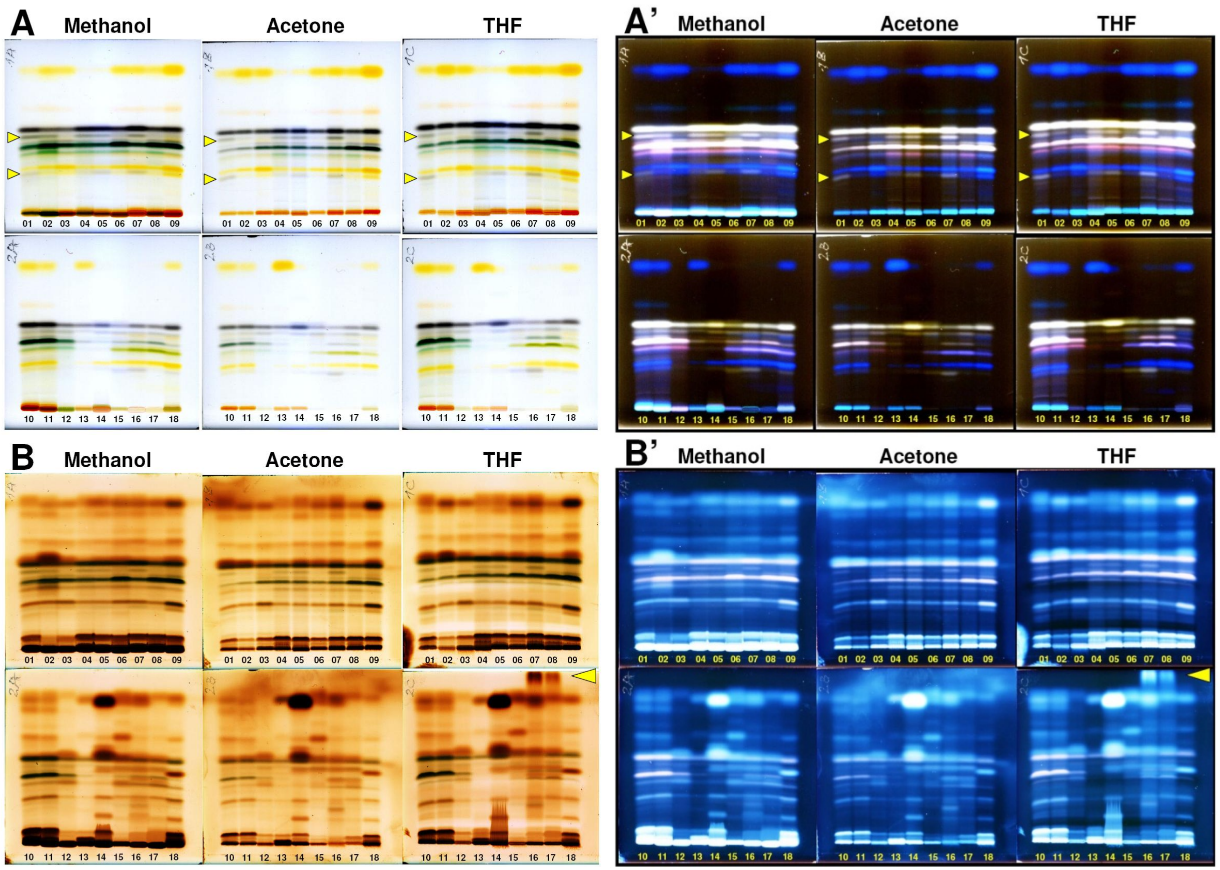


Micro-TLC separation of SPE extracts within polarity range from estetrol to progesterone (free steroids); samples were derived from sewage treatment plant JAMNO): screening of free steroids fraction within polarity range from estetrol to progesterone (Source: M. Ślączka Ph.D. Thesis; „*Biodegradation of selected hormonal modulators under technological processes and natural environmental conditions*", Koszalin 2013.)

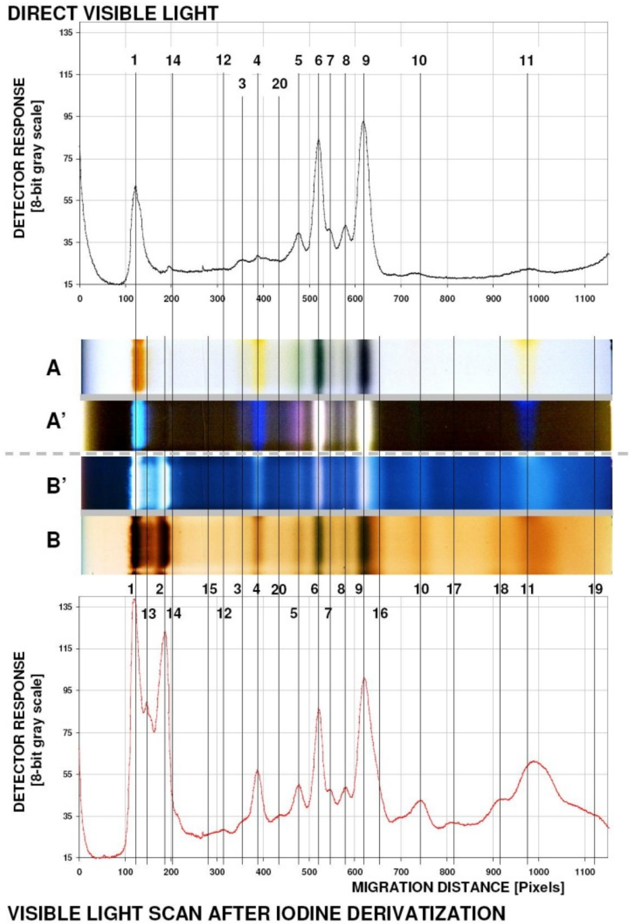
Micro-TLC separation of main components of sea trout raw bile (deproteinized methanolic solution; lane No 1) as well as acid (lane No 2) and base (lane No 3) type hydrolyzed bile samples, detected under different UV-Vis light exposure and visualization reagent conditions. Lanes No 4 and 5 correspond to cholesterol (I) and litocholic (II), deoxycholic (III), cholic (IV), glycodeoxycholic (V), glycocholic (VI) acids as well as taurodeoxycholic acid sodium salt (VII) chromatographic standards mixture, separated at quantity of 1 and 2μg/spot, respectively.



Adapted from: P.K. Zarzycki, M.M Ślączka, M.B. Zarzycka, M.A. Bartoszuk E. Włodarczyk, M.J. Baran; "Temperature-controlled micro-TLC: a versatile green chemistry and fast analytical tool for separation and preliminary screening of steroids fraction from biological and environmental samples", Journal of Steroids Biochemistry and Molecular Biology; DOI: 10.1016/j.jsbmb.2011.05.007.



Micro-TLC arrays of dyes and low-molecular mass compounds of cyanobacteria cells and herbs leafsextracted from pharmaceutical formulations and commercial food products. Extraction was performed using organic solvents characterized by relatively low parachor values includingmethanol, acetone and tetrahydrofuran.



Typical densitometric profiles and peaks ID of raw dyes (top) and after iodine exposure (bottom) corresponding to lane 4 located within acetone plate. Raw scans (A, B), inverted colors (A' , B' 426).

Adapted from: P.K. Zarzycki, M.B. Zarzycka, V.L. Clifton, J. Adamski, B.K. Głód, Low parachor solvents extraction and thermostated micro-TLC separation for fast screening and classification of spirulina from pharmaceutical formulations and food samples; accepted in Journal of Chromatography A.

CHOLESTEROL AND BILE ACIDS

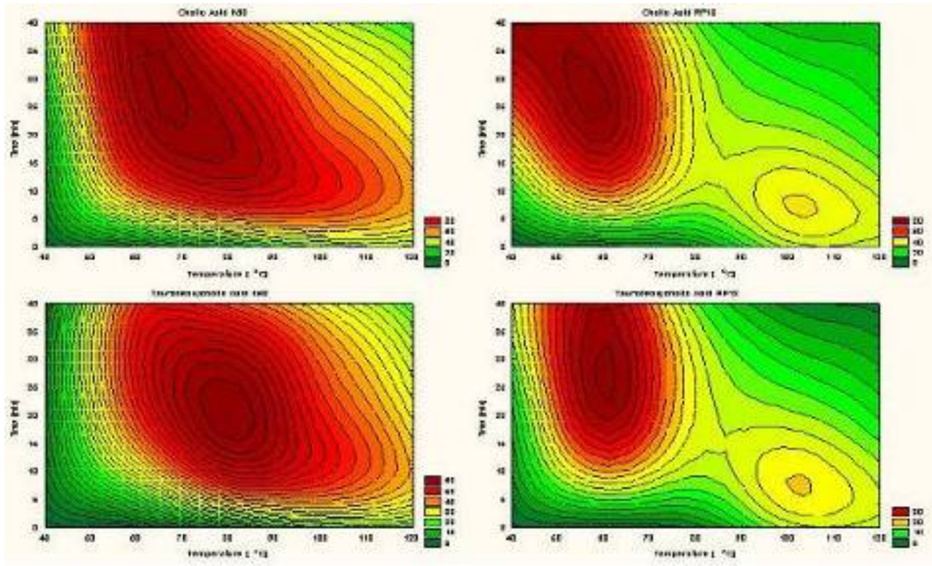
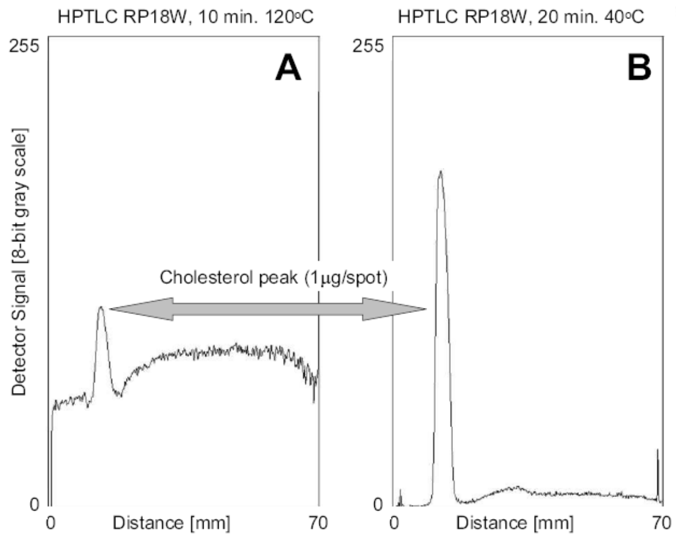
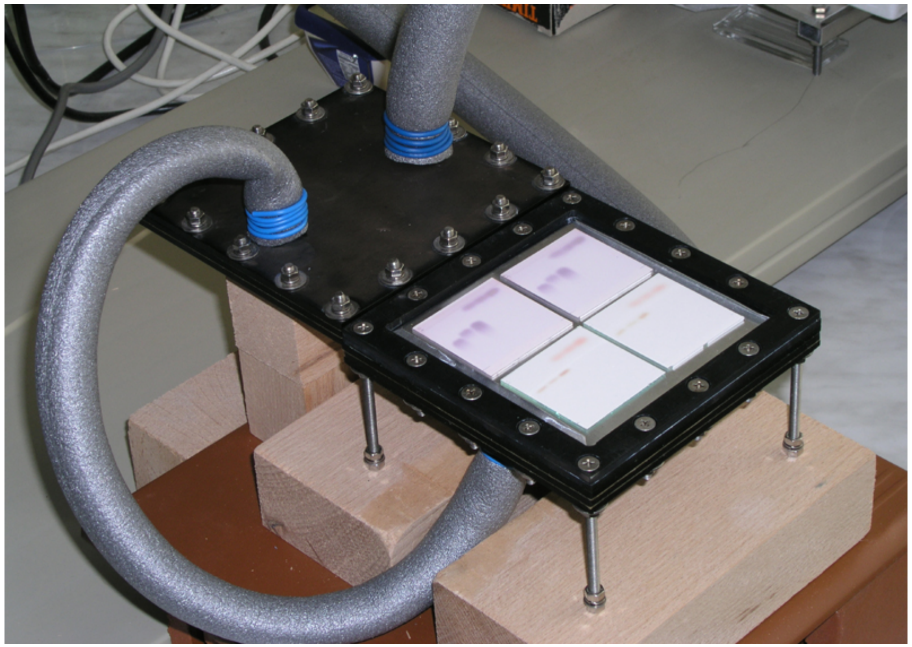


Figure 2





Source: P.K. Zarzycki, M. A. Bartoszuk, A. I. Radziwon, „Optimization of TLC Detection by Phosphomolybdic Acid Staining for Robust Quantification of Cholesterol and Bile Acids", J. Planar Chromatogr., 19 (2006) 52-57. T. Modzelewski, E. Włodarczyk, F. B. Harasimiuk, M. B. Zarzycka, M. Baran, P.K. Zarzycki, „Simple device for TLC spots visualisation at elevated temperatures"; Measurement Automation and Monitoring (Pomiary Automatyka Kontrola), 54(4) (2008) 184-186

DATA PROCESSING SCHOOL - FOCUSING ON NEW APPROACHES FOR DETECTION IN SEPARATION SCIENCE

KOSZALIN UNIVERSITY OF TECHNOLOGY
KOSZALIN 2015, POLAND

CONTACT PERSONS:

A/Prof. Zbigniew Suszyński, Chair of Multimedia Systems and Artificial Intelligence, Faculty of Electronics and Computer Sciences.
Ph. +48 600 244 048, E-mail address: zbigniew.suszynski@tu.koszalin.pl,
web page: ksmisi.tu.koszalin.pl

A/Prof. Paweł K. Zarzycki, Section of Toxicology and Bioanalytics, Department of Civil and Environmental Engineering.
Ph. +48 94 3478671; mob: +48 507 516 486
E-mail address: pkzarz@wp.pl, web page: http://www.wilsig.tu.koszalin.pl/labtox

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Section of Toxicology and Bioanalytics, Department of Civil and Environmental Engineering, Koszalin University of Technology, Śniadeckich 2,75-453 Koszalin, Poland

www.wilsig.tu.koszalin.pl/labtox/

HPTLC 2014, Lyon, France

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