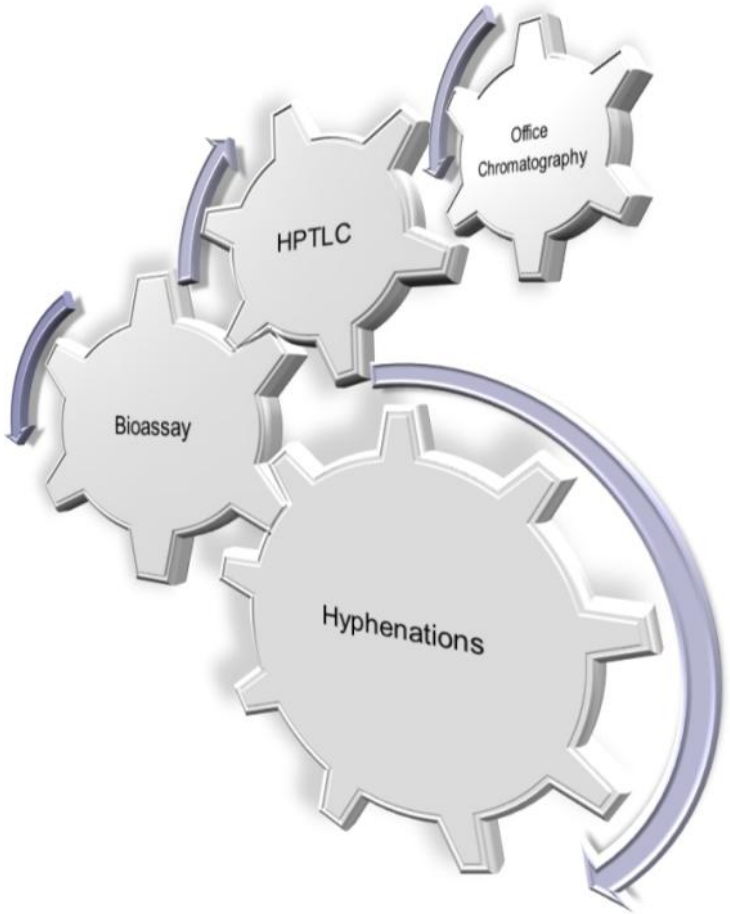
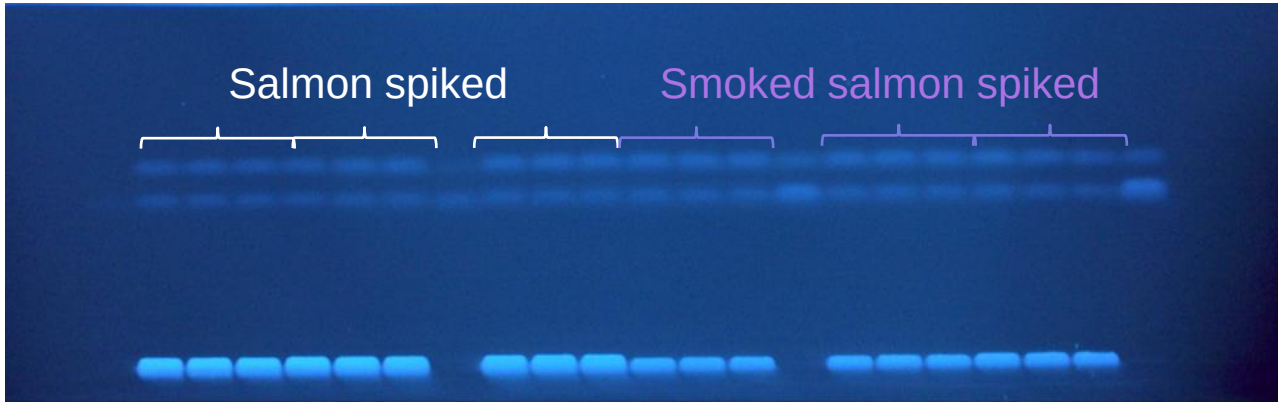
Overview on the power of hyphenated HPTLC analysis of food and herbs

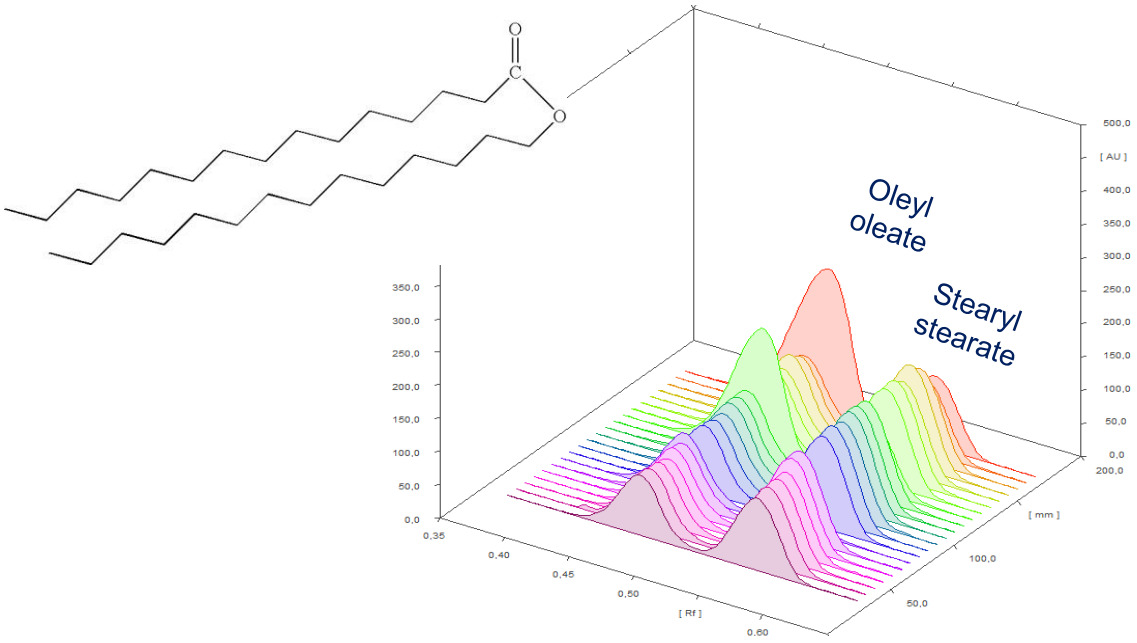


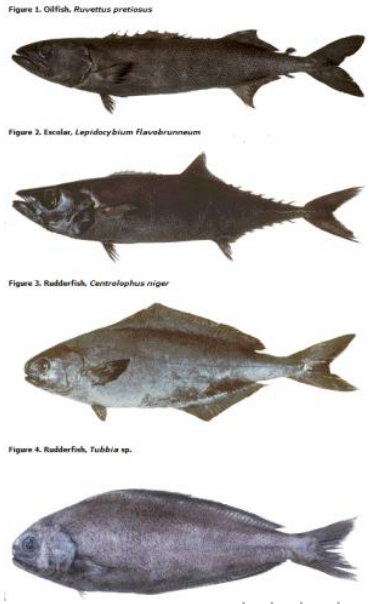
Gertrud Morlock, Chair of Food Science
Justus Liebig University Giessen

Analysis of wax ester in fish (*Gemypylidae*)

Stearyl stearate
Oleyl oleate

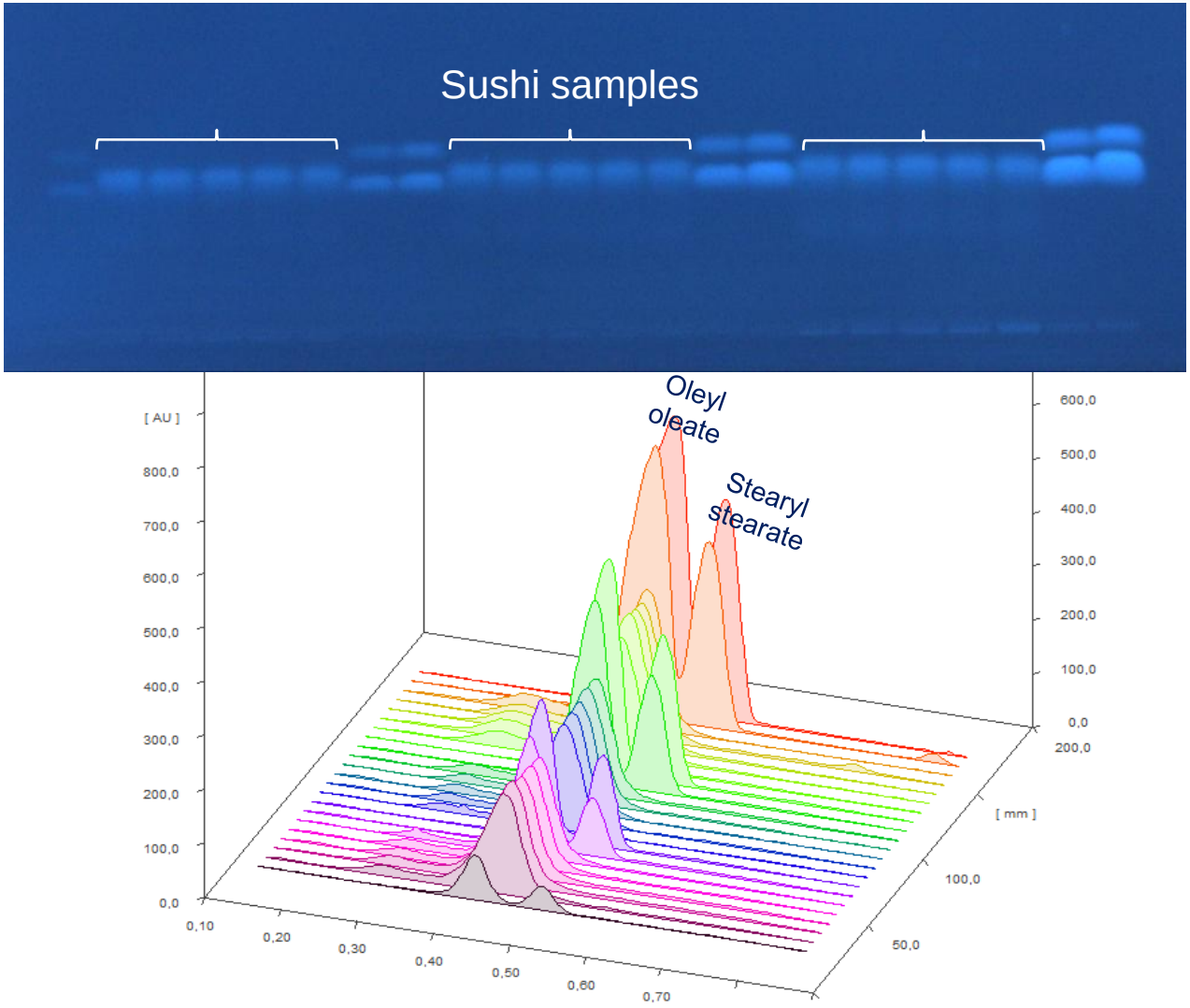






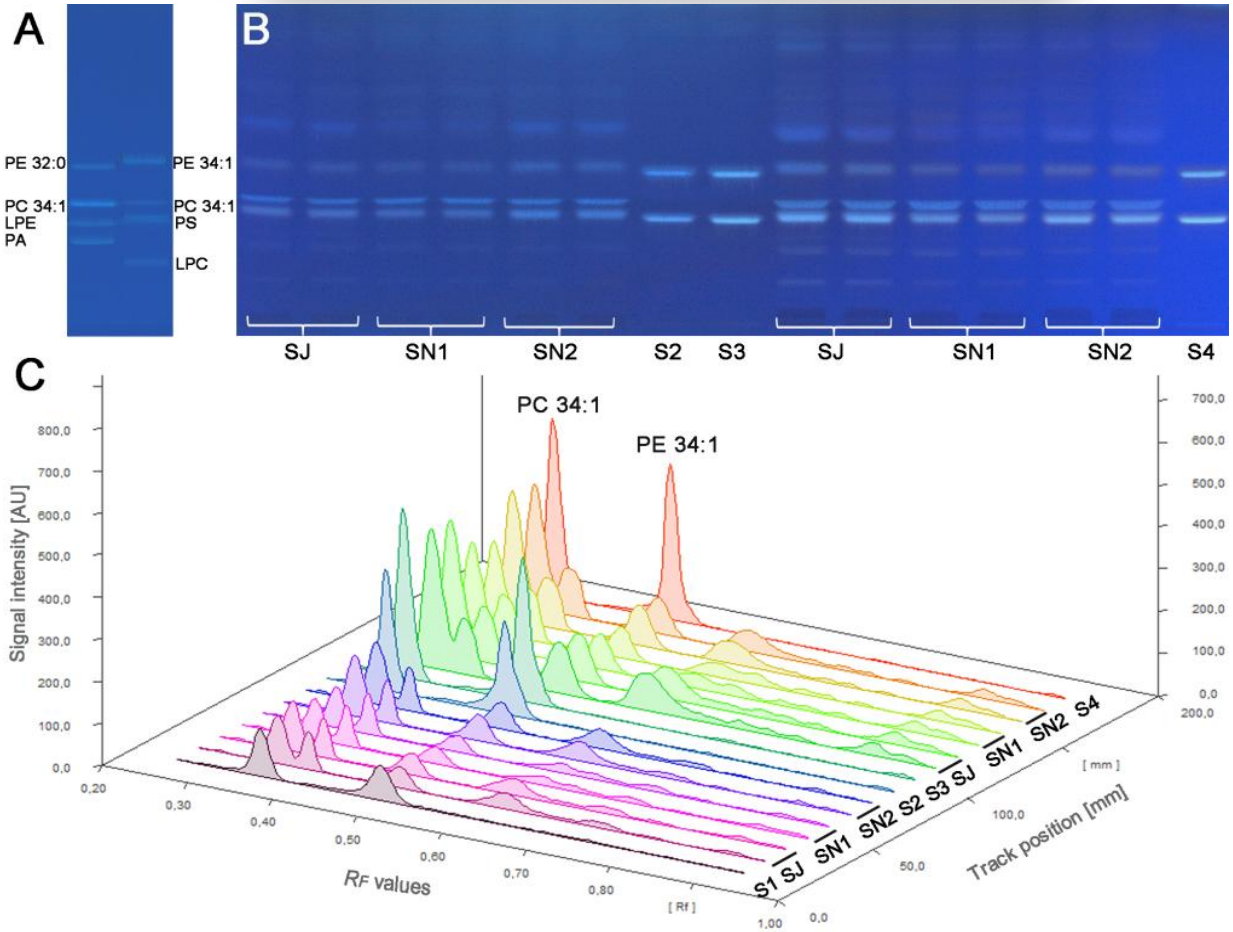
www.kuirejo.de

Analysis of wax ester in fish (*Gemypylidae*)



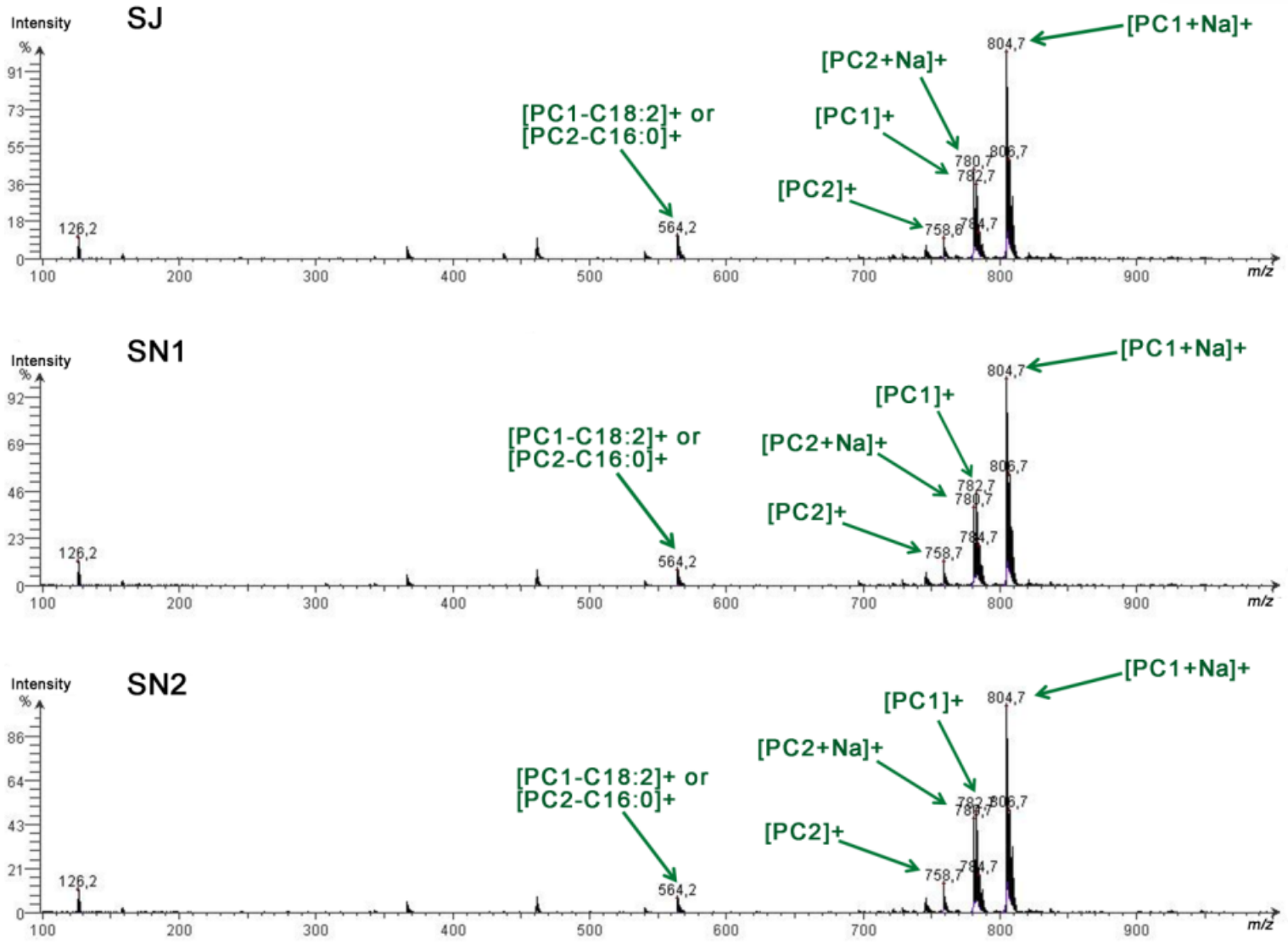
L. Winheim, A. Miller, G. Morlock, in preparation

Analysis of phospholipids in lecithins

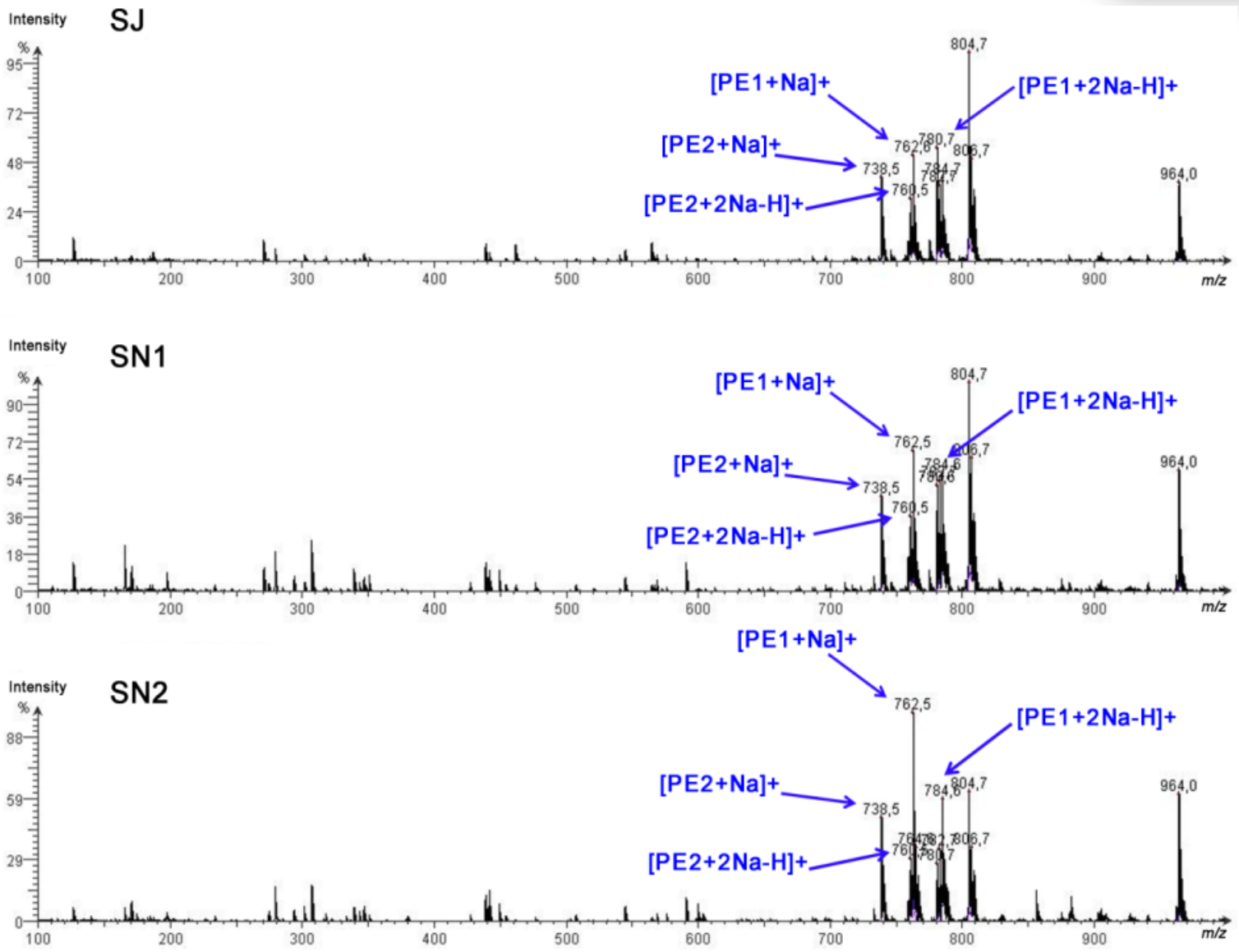


S. Krüger, L. Bürmann, G. Morlock, *J Agric Food Chem,* in print

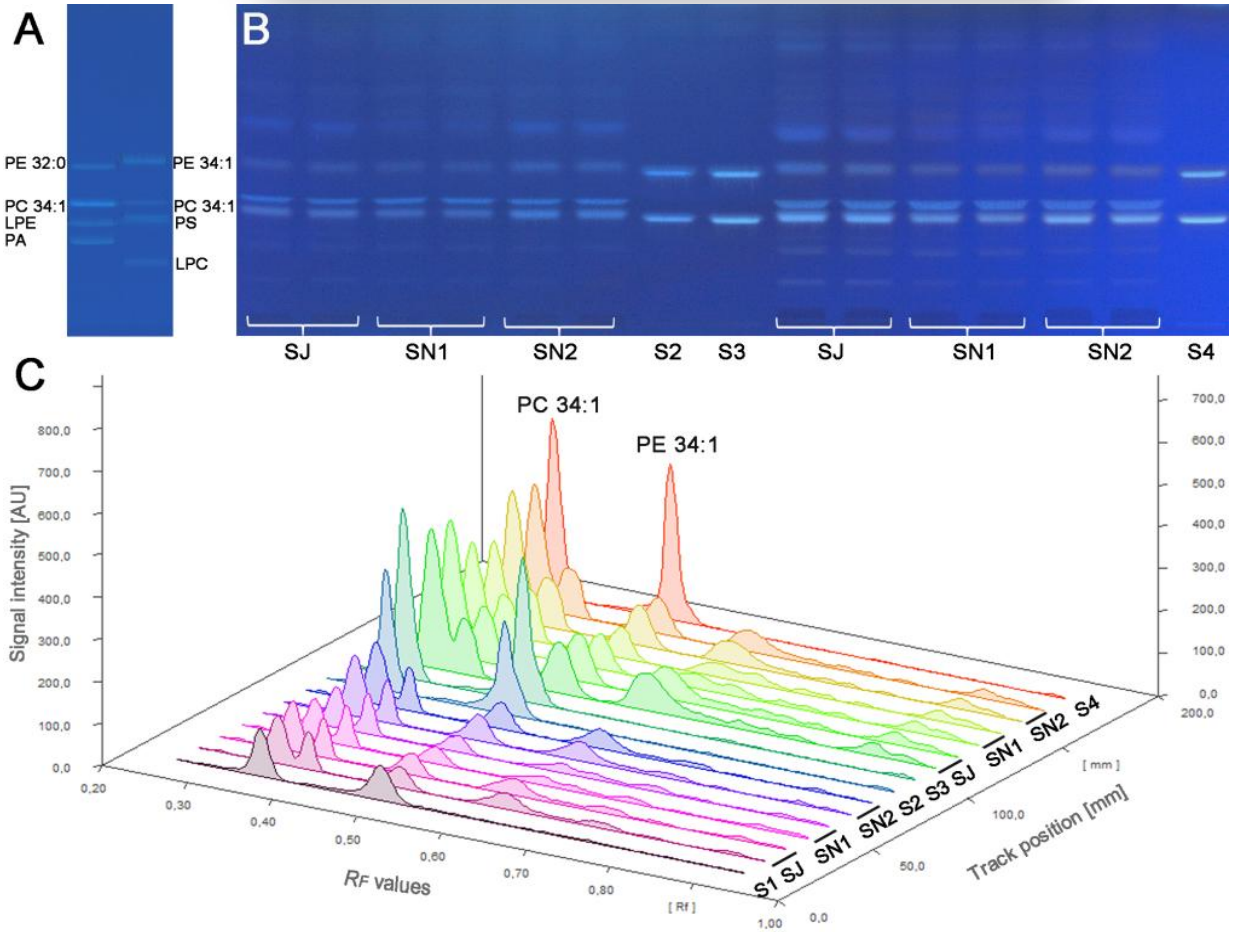
Characterization: soy bean *vs.* sunflower lecithin



Characterization: soy bean *vs.* sunflower lecithin



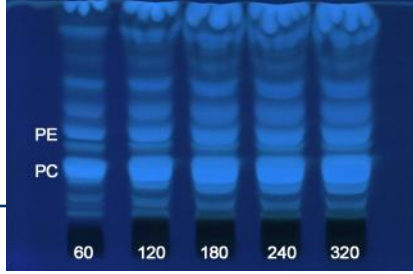
Analysis of phospholipids in lecithins

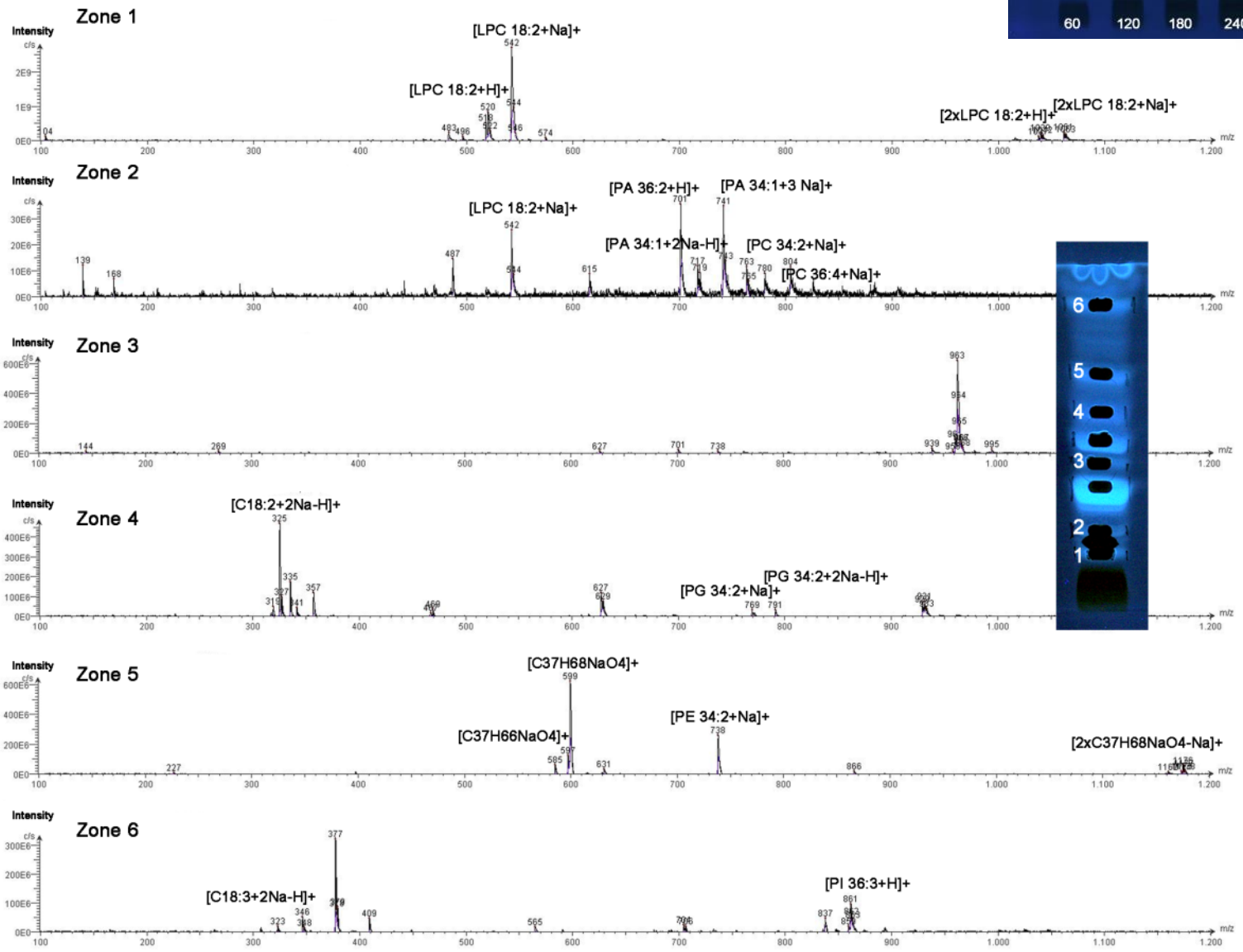


S. Krüger, L. Bürmann, G. Morlock, *J Agric Food Chem,* in print

G. Morlock Food Science JLU Gießen

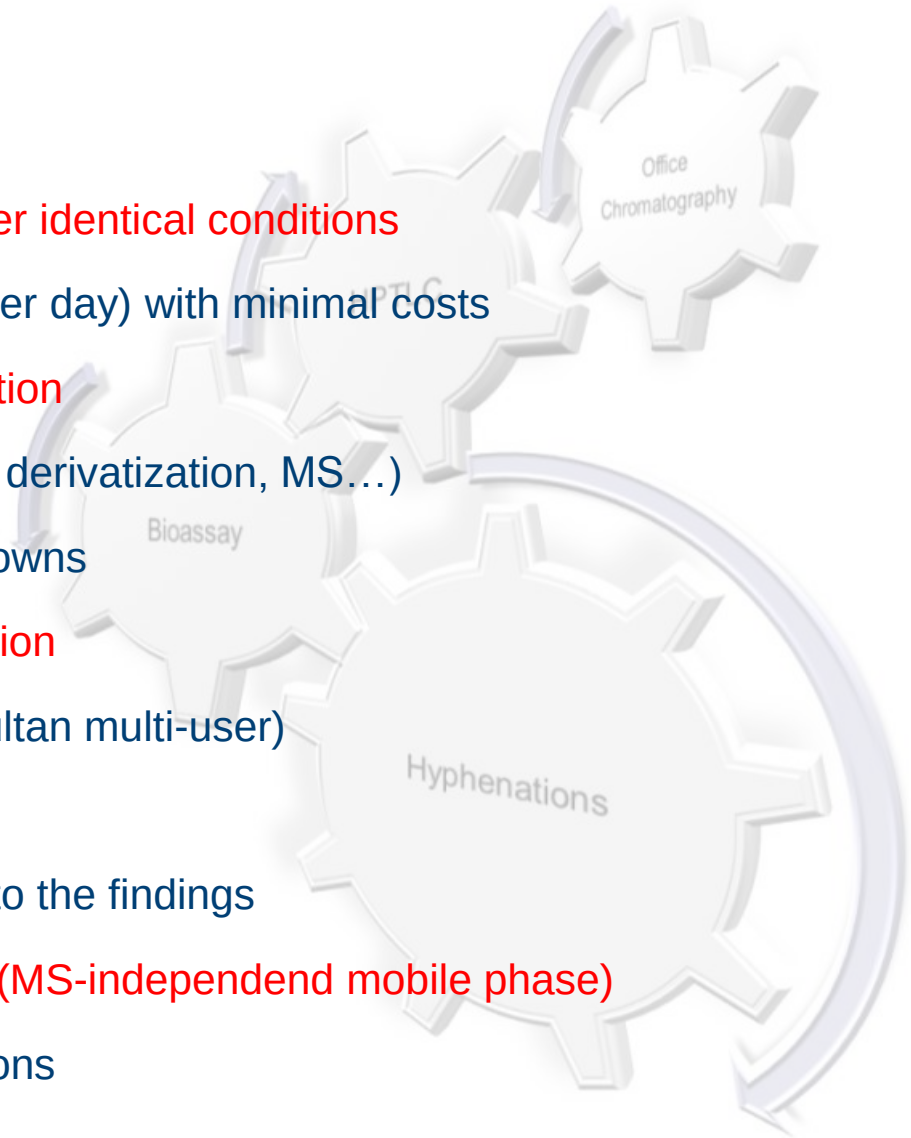
Minor phospholipids?





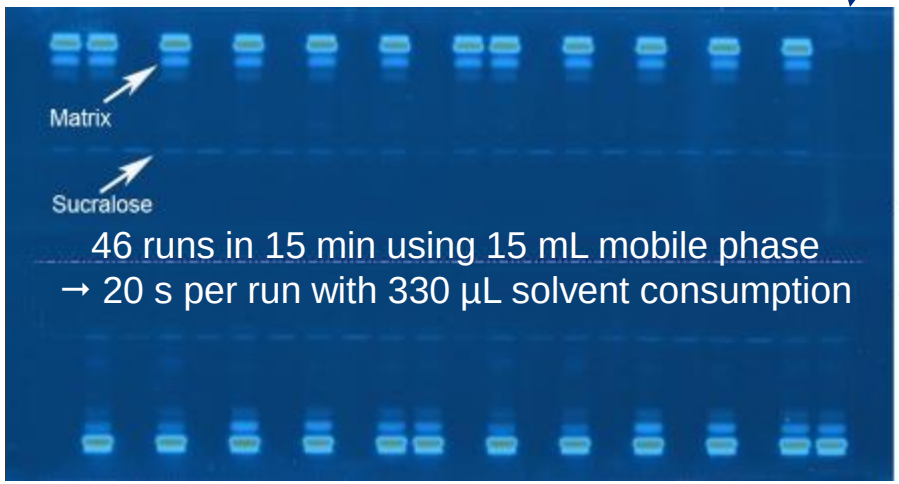
The power of HPTLC

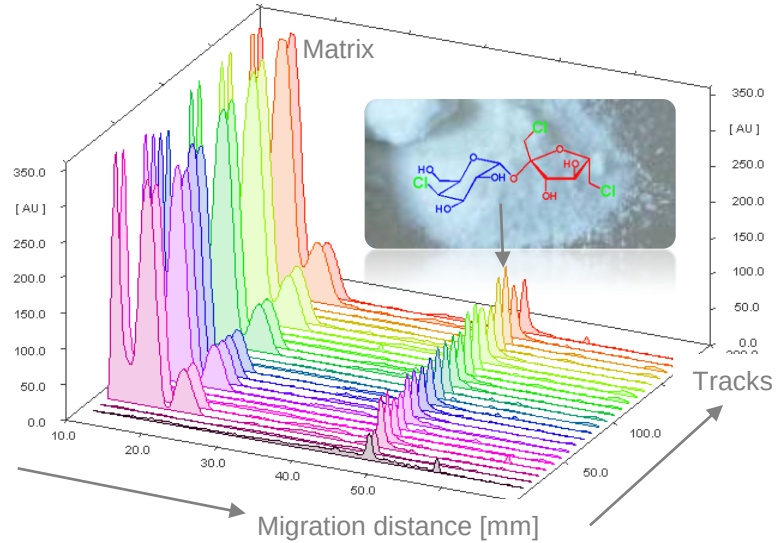
1. Reduced sample preparation
2. Matrix-tolerant method
3. Parallel chromatography under identical conditions
4. High throughput (1000 runs per day) with minimal costs
5. Selective, simultan derivatization
6. Multi-detection (UV/Vis, FLD, derivatization, MS…)
7. More information about unknowns
8. Concentration during application
9. Flexible working station (simultan multi-user)
10. Effect-directed analysis
11. Analytical workflow adjusted to the findings
12. Targeted mass spectrometry (MS-independend mobile phase)
13. The ease of super-hyphenations



Sucralose in milk-based confection (*Burfi*)

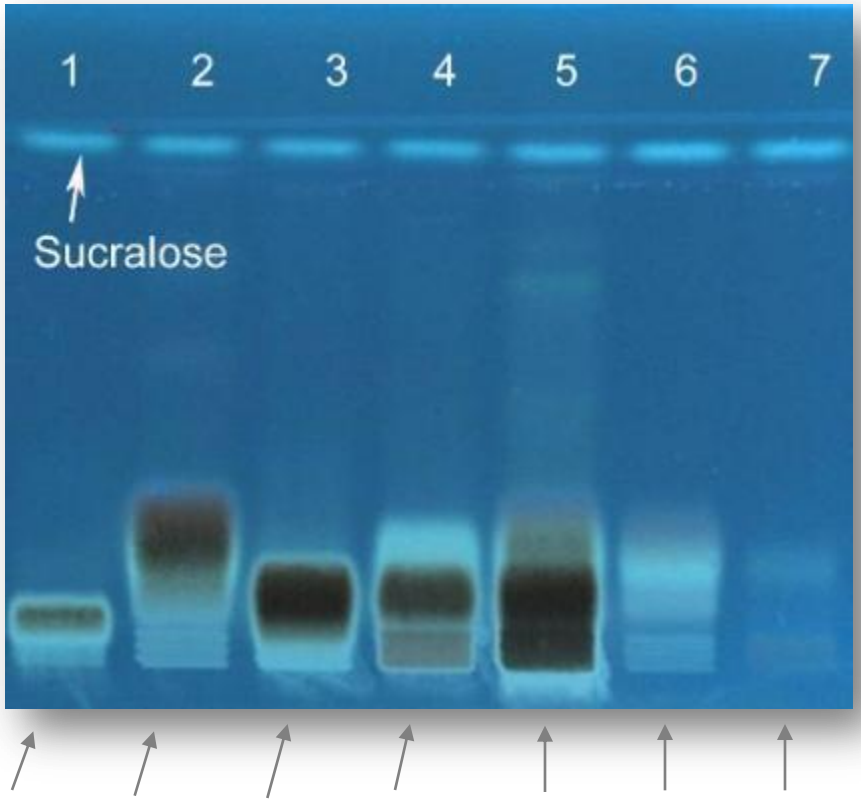
extracted in MeOH, filtered





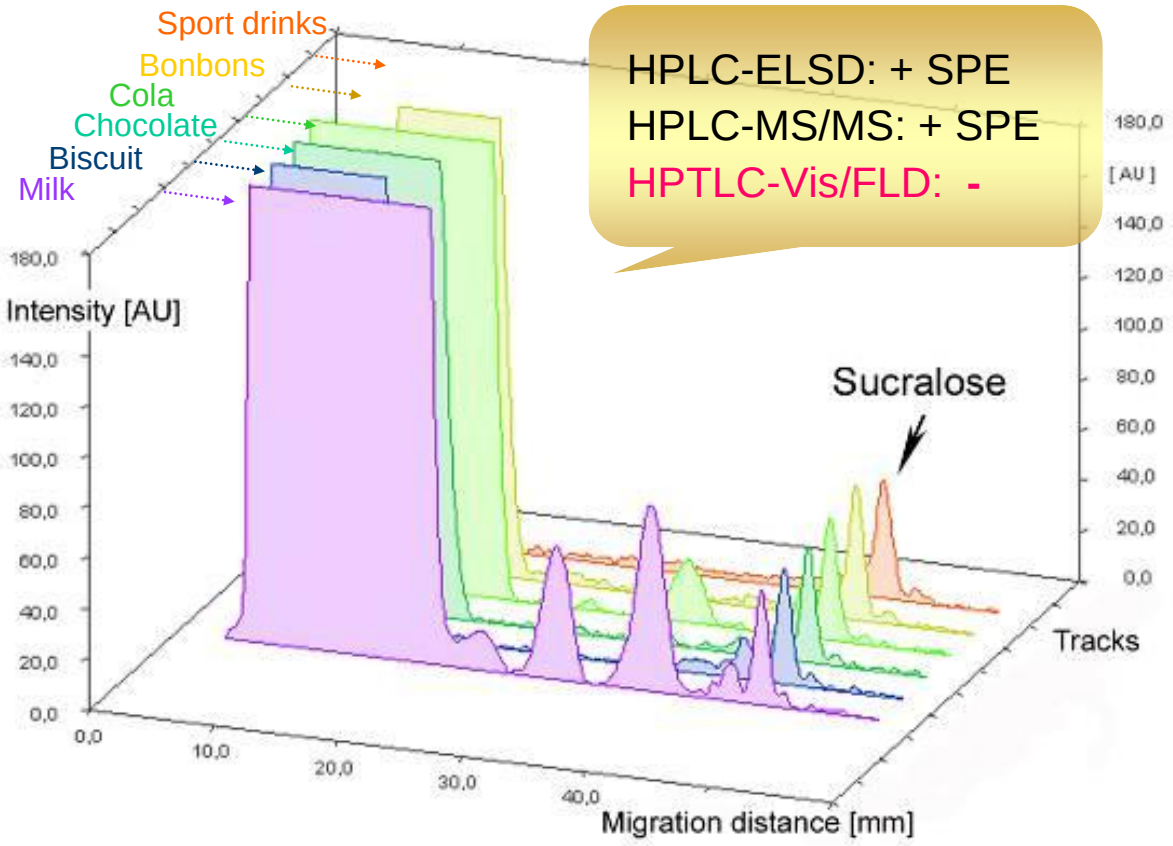
G. Morlock, S. Prabha, *J Agric Food Chem* 55 (2007) 7217-7223

… in further matrices



Milk, biscuit, chocolate, cola, bonbons, energy/sport drinks

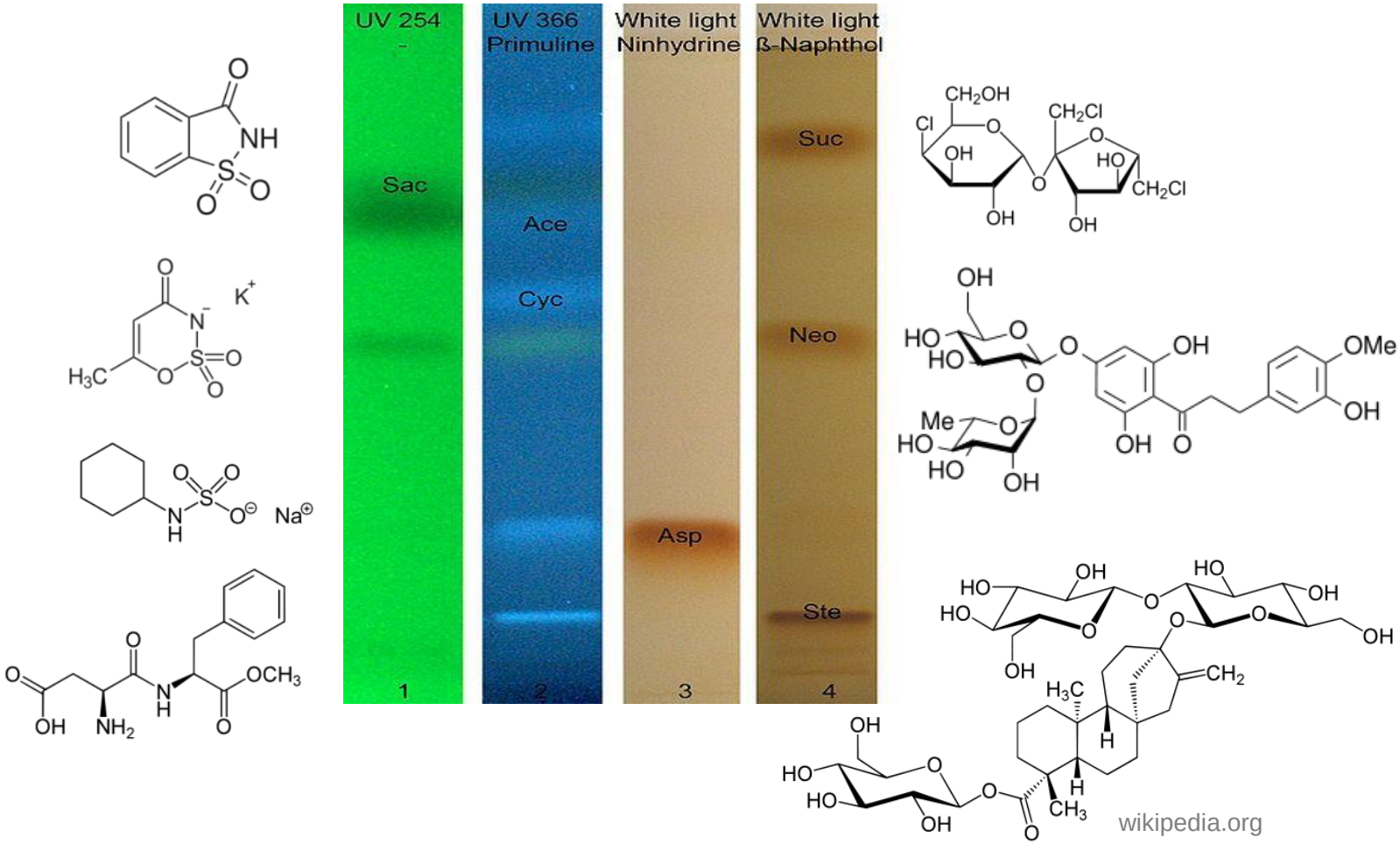
Sample preparation and chromatography



G. Morlock, M. Vega, *J Planar Chromatogr* 20 (2007) 411-417

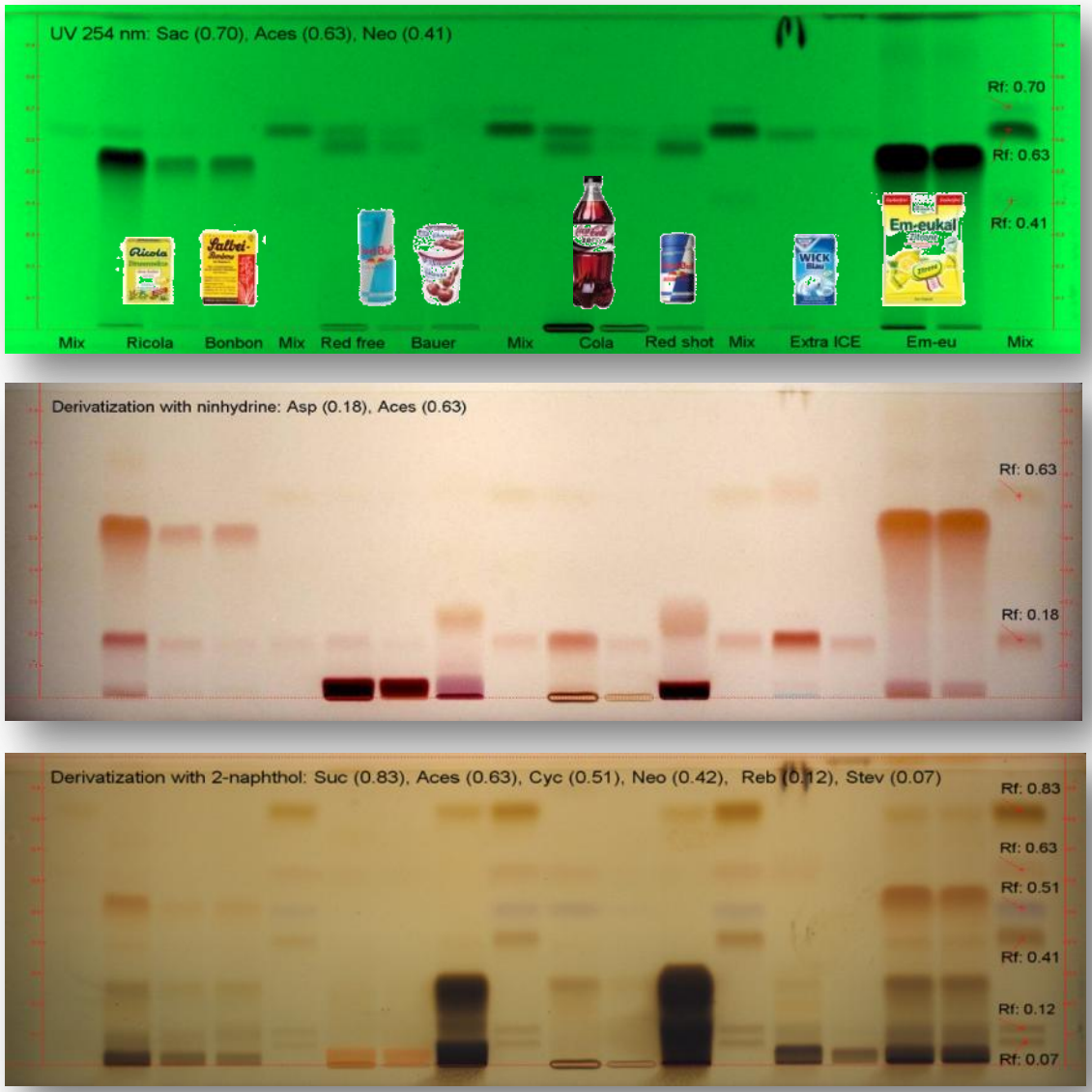
Analysis of sweeteners

→ Reagent sequence: 4 reagents on the same plate



G. Morlock, G. Sabir, in preparation

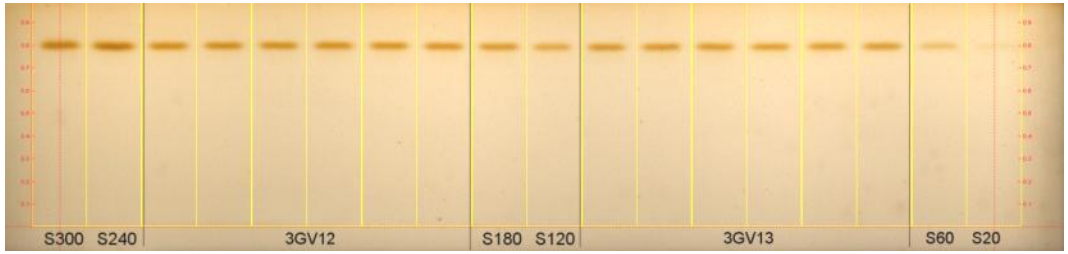
Food safety → Sweeteners



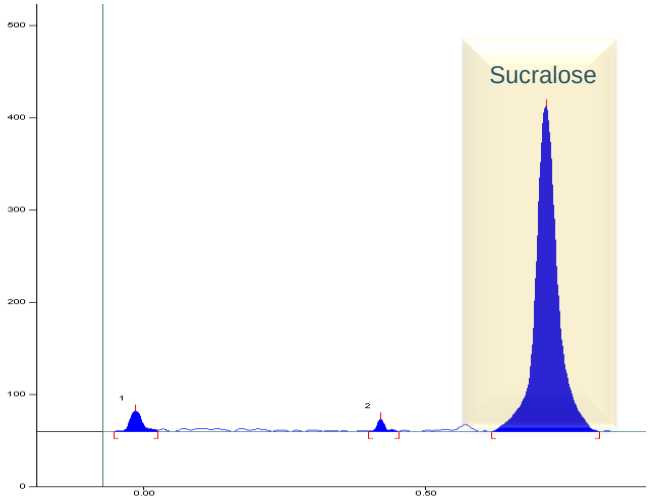
G. Morlock, G. Sabir, in preparation

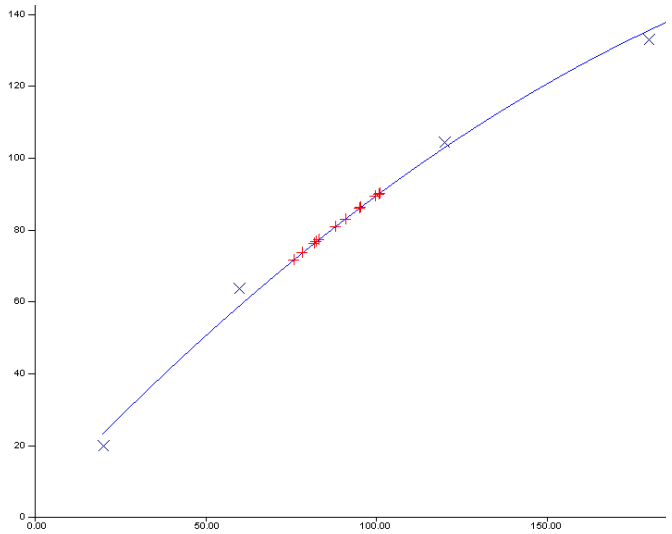
Sucralose in saliva

→ Release study from chewing gum

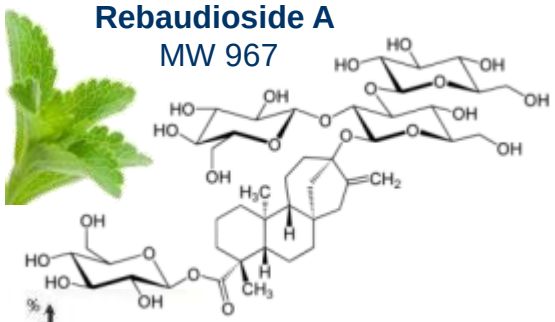


www.media.news.de/resources

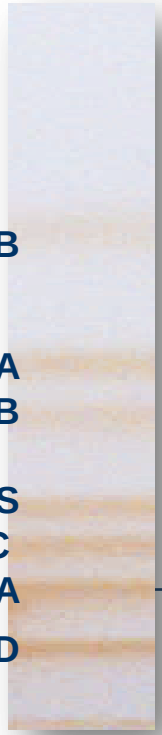


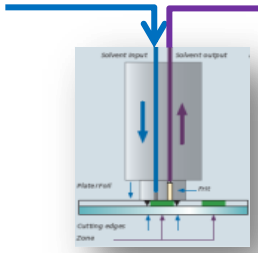


Analysis of steviol glycosides



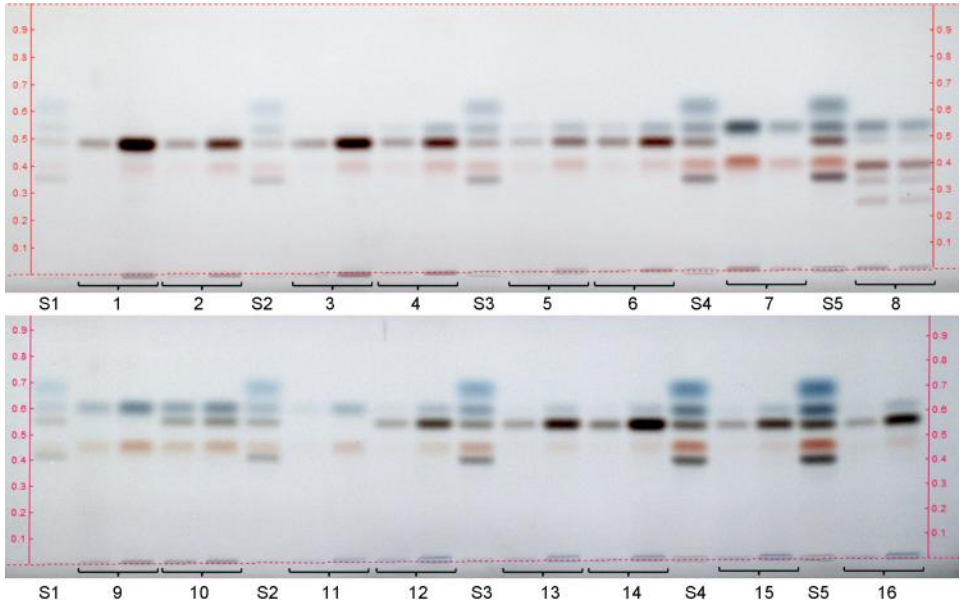
Rebaudioside A
MW 967

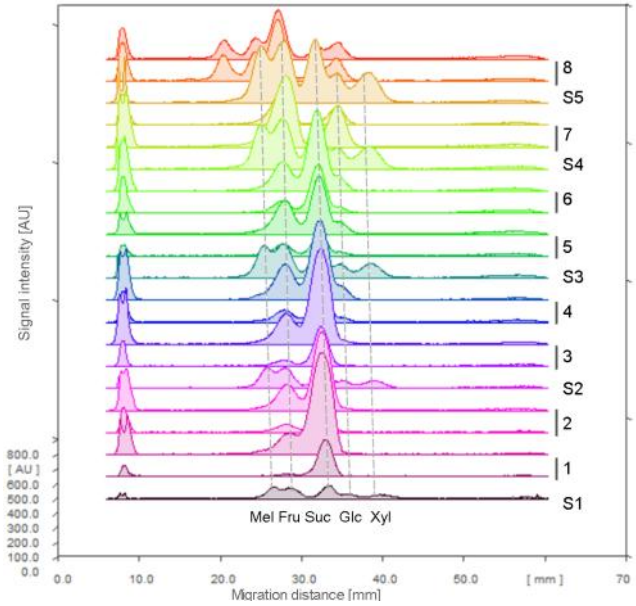




G. Morlock *et al., J Chromatogr A* 1350 (2014) 102-111

Carbohydrates in *Marantaceae* nectars





	C. lutea So	*C. lutea S*	*C. crotalifera So*	*C. crotalifera S*	*C. picturata S*	*C. hylaeanthoides S*	*C. platystachya S*	*C. lasiophylla So*	*C. lasiophylla S*	*C. donnell-smithii S*	*C. marantifolia S*	*H. hoffmannii S*	*Ct. Dasycarpa S*	*Ma. Purpurea S*	*P. pruinosa So*	*P. pruinosa S*
N	10	12	5	6	1	1	6	5	7	6	7	7	3	6	6	7
Suc	24.85	28.92	22.57	18.47	3.21	16.32	26.38	17.34	21.52	41.55	43.21	41.14	24.03	19.87	41.4	31.25
Glc	8.06	6.10	2.99	3.06	0.00	1.42	1.41	1.83	1.63	1.13	1.36	0.00	1.02	0.00	4.22	2.68
Fru	10.66	7.22	4.40	4.40	0.00	3.17	1.90	2.56	2.25	2.58	3.10	0.00	3.62	0.00	8.05	4.76
Suc / Glc + Fru	1.327	2.170	3.053	2.473	/	3.548	7.970	3.945	5.527	11.157	9.659	/	5.167	/	3.371	4.196
Glc / Fru	1.322	1.183	1.471	1.440	/	2.225	1.348	1.399	1.399	2.279	2.279	/	3.534	/	1.905	1.778

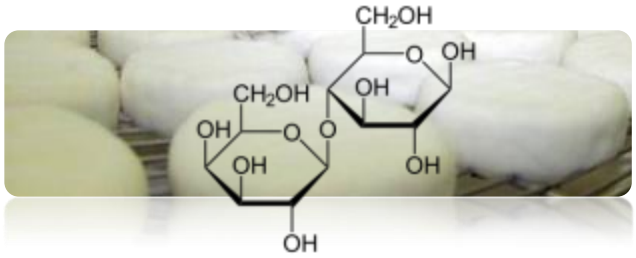
A. Ruppel, G. Morlock, *J Planar Chromatogr* 28 (2015) 162–166

Analysis of lactose-free food: HPLC-CAD

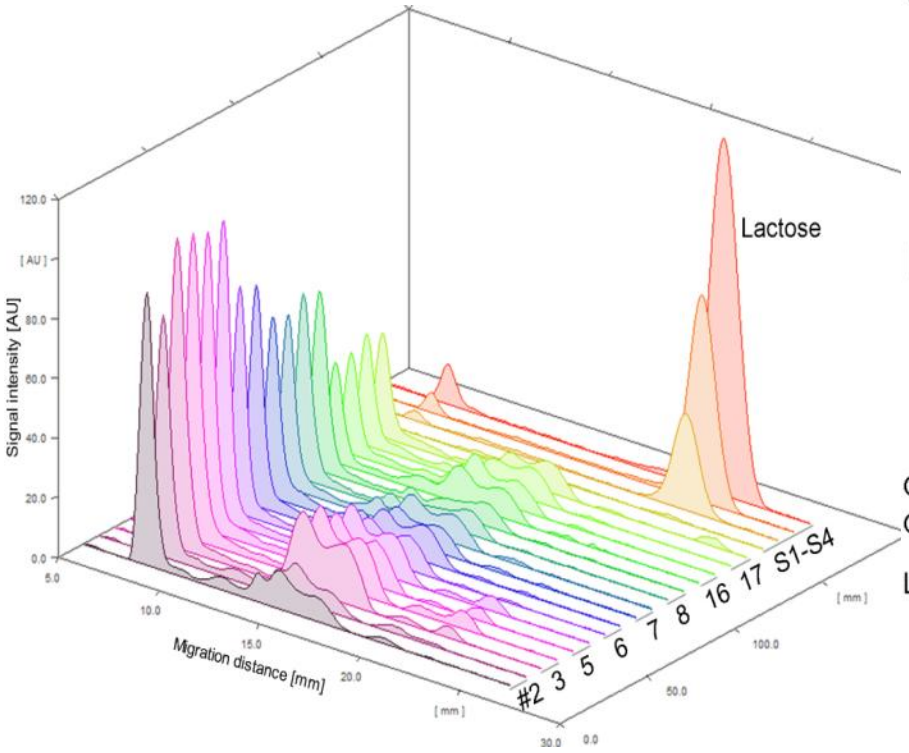
www.einfach-laktosefrei-leben.de

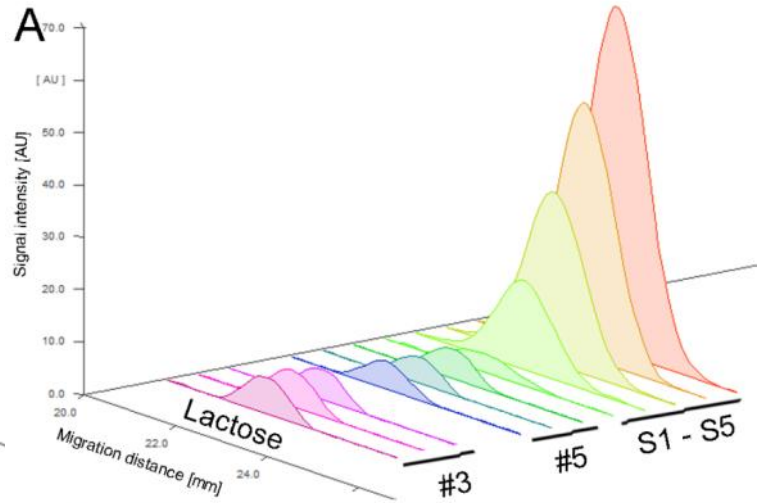
ESA, Dionex, application note 2011: Lactose in milk - a validated method

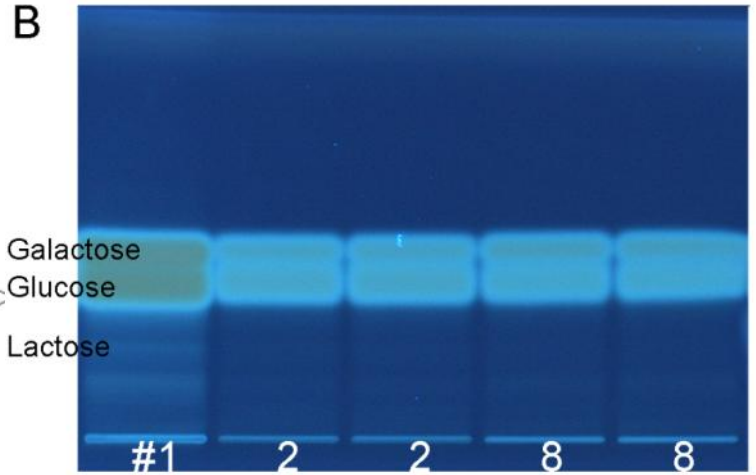
Analysis of lactose-free food



LOD → 0.000004 % (0.04 mg/L)





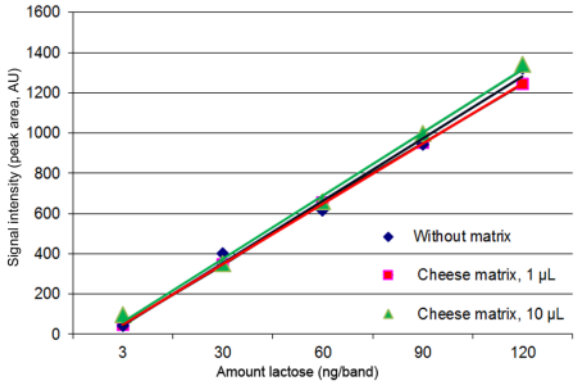


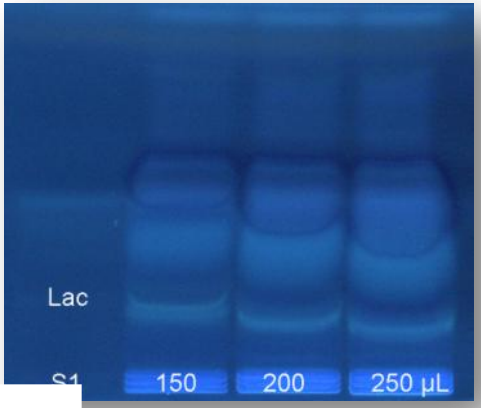
G. Morlock, L. Morlock, C. Lemo, *J Chromatogr A* 1324 (2014) 215–223

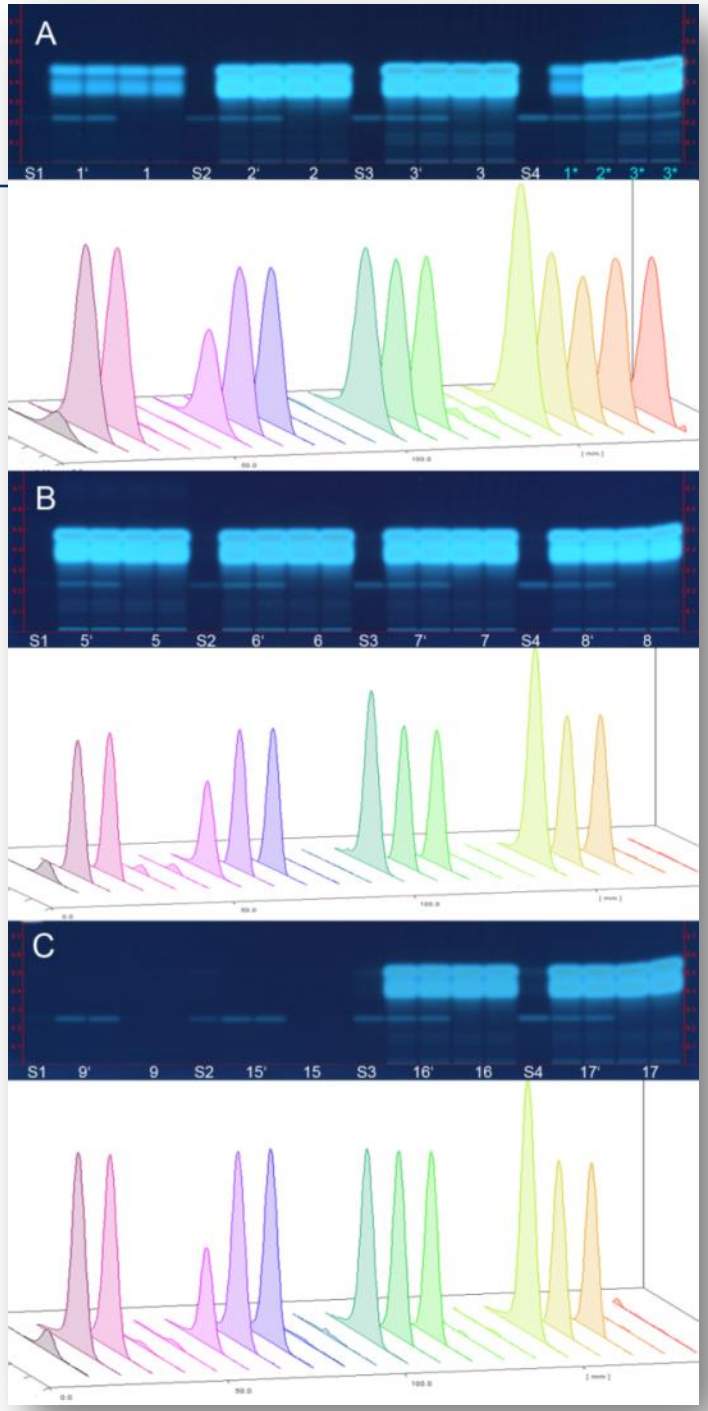
Rectangular application

Lactose-free dairy products

LOD → 0.000004 % (0.04 mg/L)



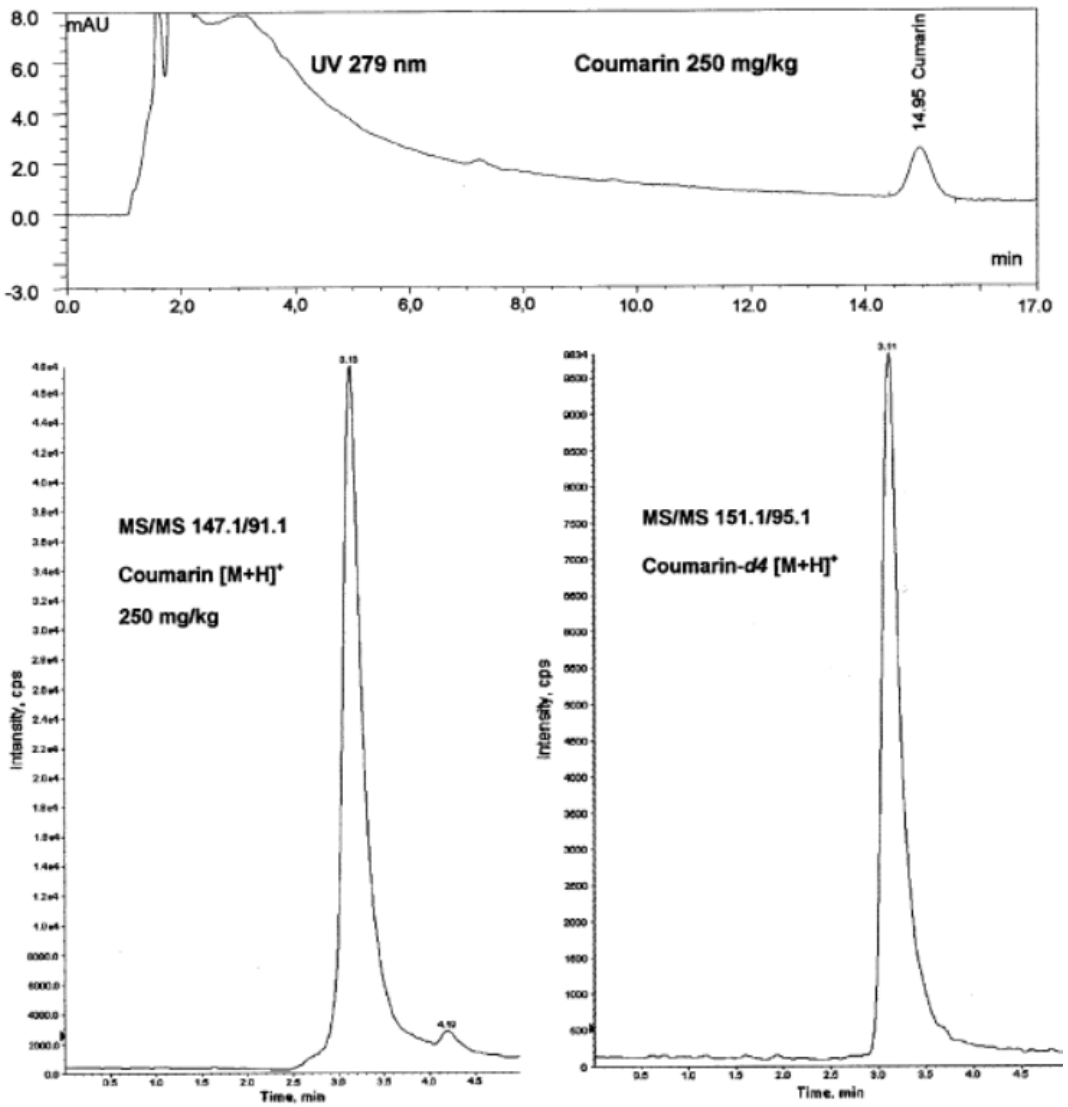




Recovery rates in lactose-free food

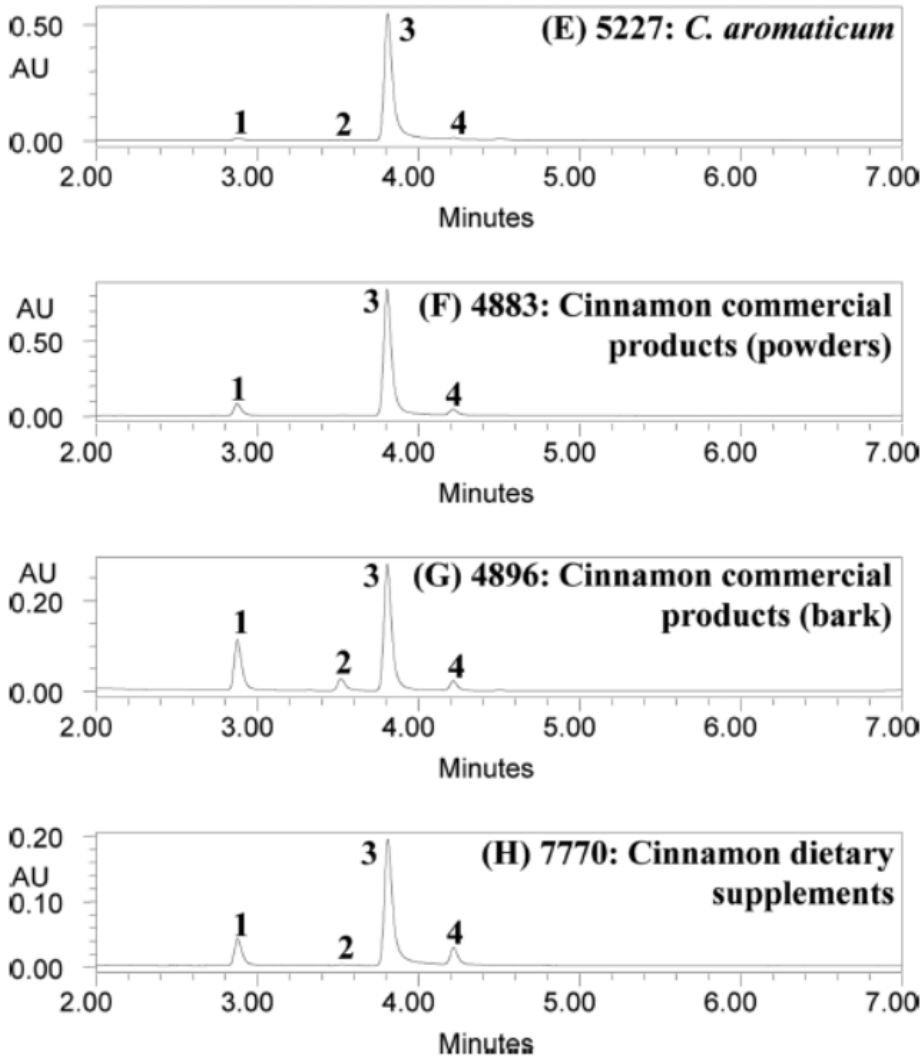
Lactose-free dairy product	#	Mean recovery of lactose (%)	Repeatability (*%RSD*, *n* = 2)
Butter	1	105.3	1.9
Yoghurt	2	87.7	0.7
Milk	3	87.7	0.6
Evaporated milk	5	76.2	3.1
Buttermilk	6	81.2	0.2
Sour cream	7	78.9	2.9
Cream	8	82.8	0.2
Goat cheese	9	103.6	1.4
Cheese	15	102.4	1.2
Cream cheese	16	99.2	0.5
	17	90.3	1.8
Mean ± *%RSD* (n = 11)		90.5 ± 10.5	1.3 ± 1.0

Cumarine analysis: HPLC-UV *and* -MS



M. Raters, R. Matissek, *Eur Food Res Technol* 227 (2008) 637–642

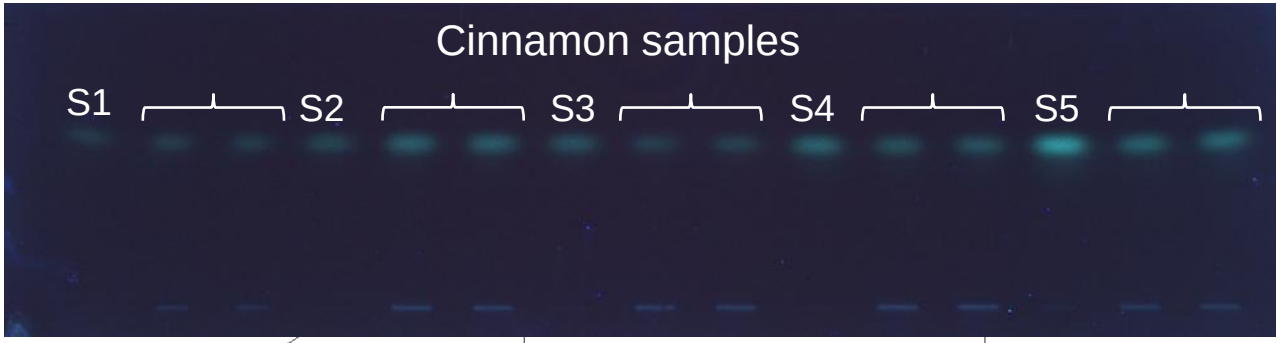
Cumarine analysis: UPLC-UV



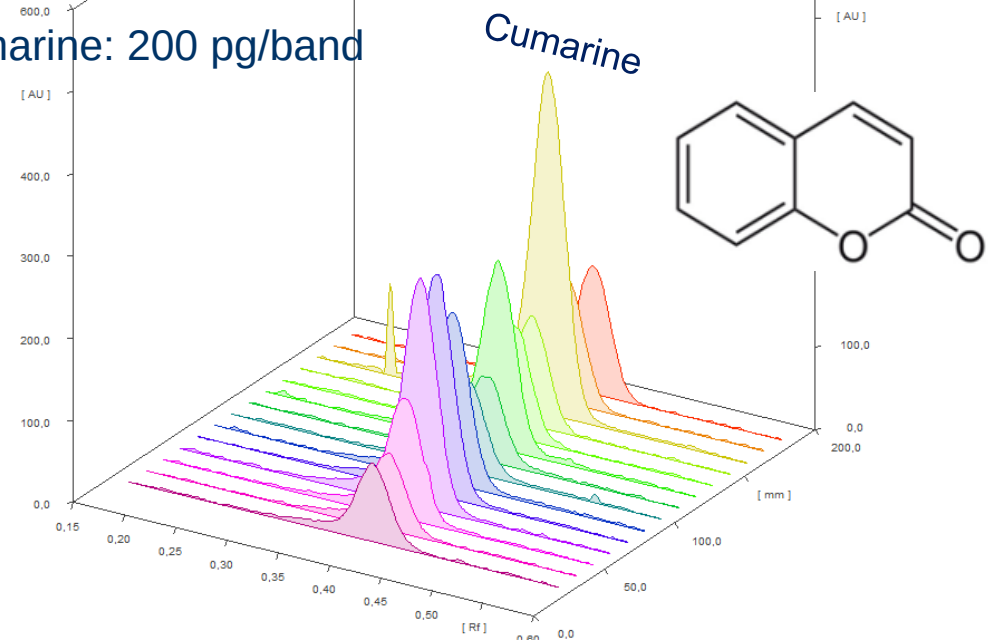
J Agric Food Chem 61 (2013) 4470−4476

Analysis of cumarine

5 different cinnamom spice samples containing cumarine



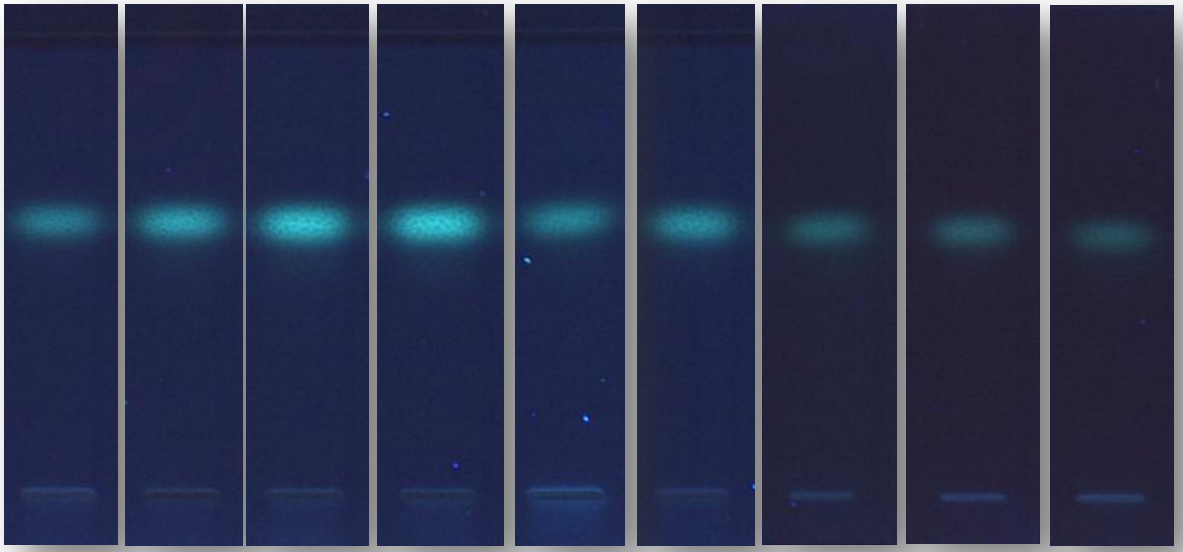
LOD of cumarine: 200 pg/band



L. Winheim, S. Krüger, G. Morlock, in preparation

Analysis of cumarine

9 different food samples containing cumarine



www.wikipedia.de

www.colourbox.de

www.deutsche-wirtschafts-nachrichten.de

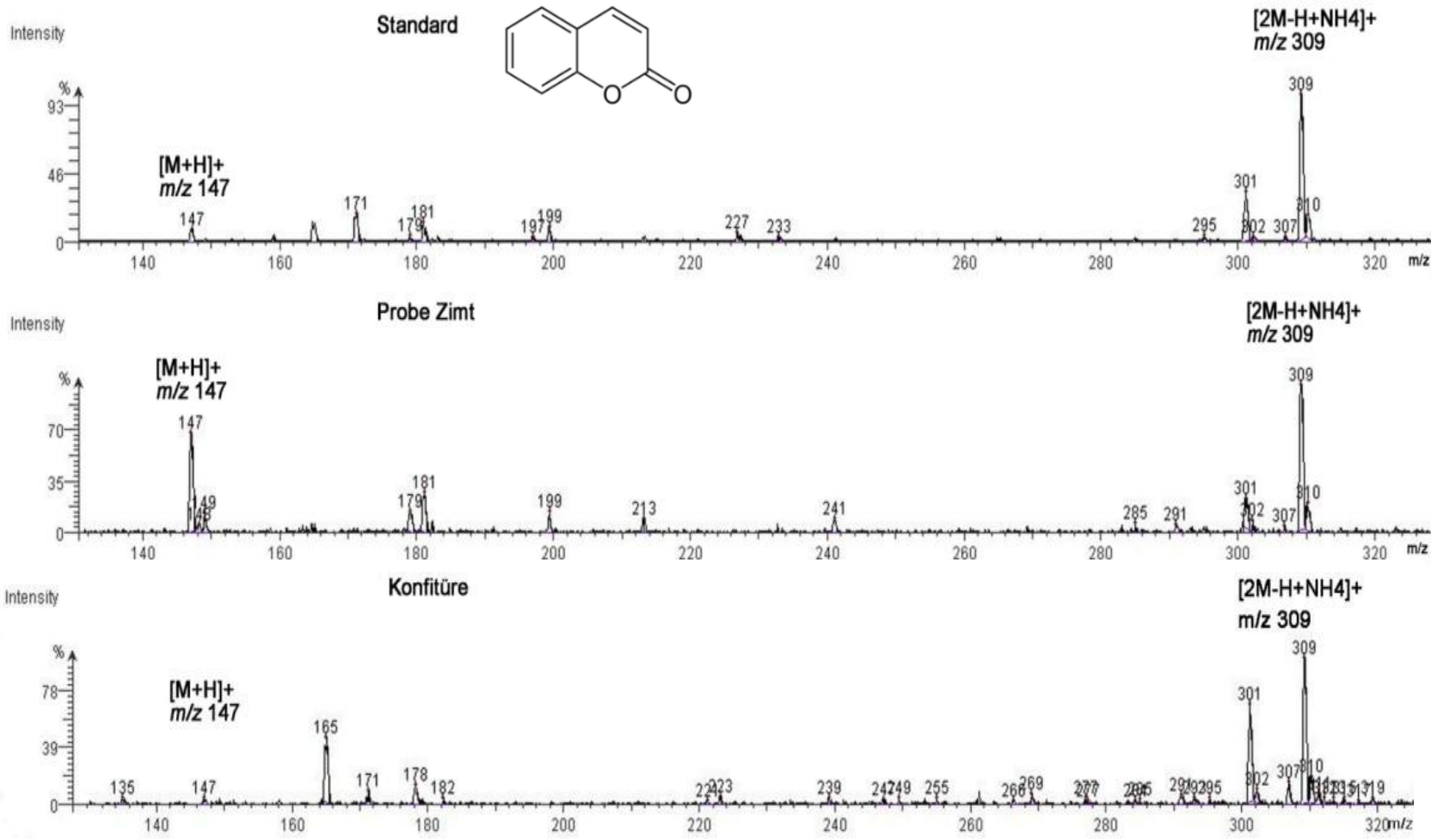
www.institut-fresenius.de

L. Winheim, S. Krüger, G. Morlock, in preparation

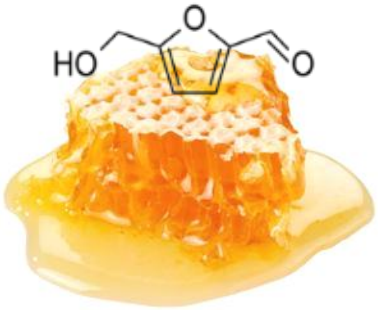
Cumarine contents

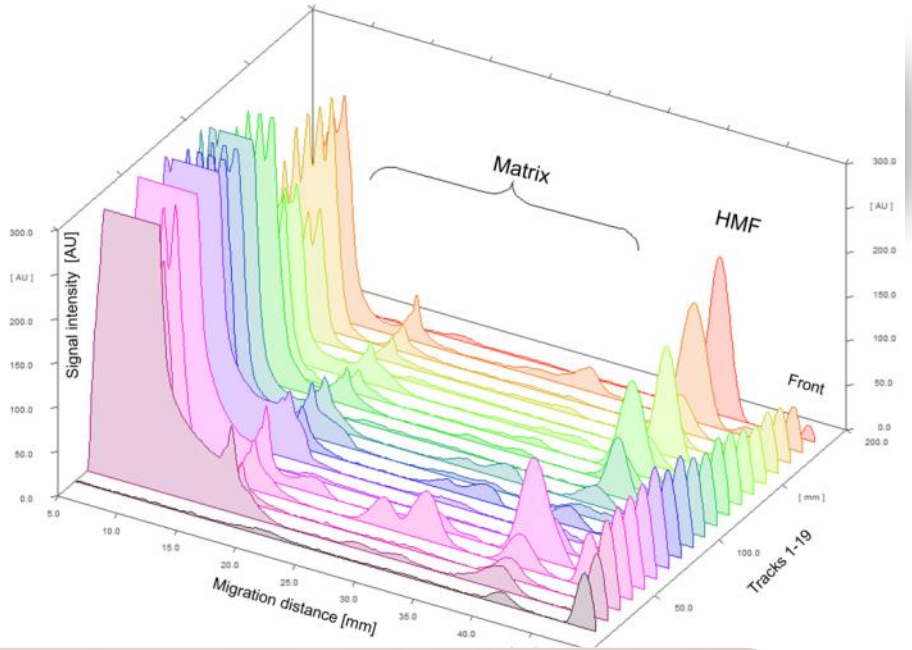
Sample	Manufacturer	Cumarine (mg/kg)	Repeatabilities *(%RSD, n=2)*	Reproducibility *(%RSD, n=2)*
Spice	Sonnentor - gemahlen	3732	0.4/1.2	1.0
	Lidl Kania - gemahlen	1615	0.4/0.3	2.4
Tea	YogiTea - Frauenpower	19	1.4/3.2	4.5
	TeeGeschwendner – Kaminfeuer	22	3.3/2.0	0.5
Cereals	Aldi Knusperone - Zimt Chips	1	3.8/5.9	0.4
Cinnamon bun	IKEA - Kanelbullar	16	1.4/4.5	6.6
Jam	Grafschafter Konfitüre – Winterzauber	4	1.6/1.6	1.6

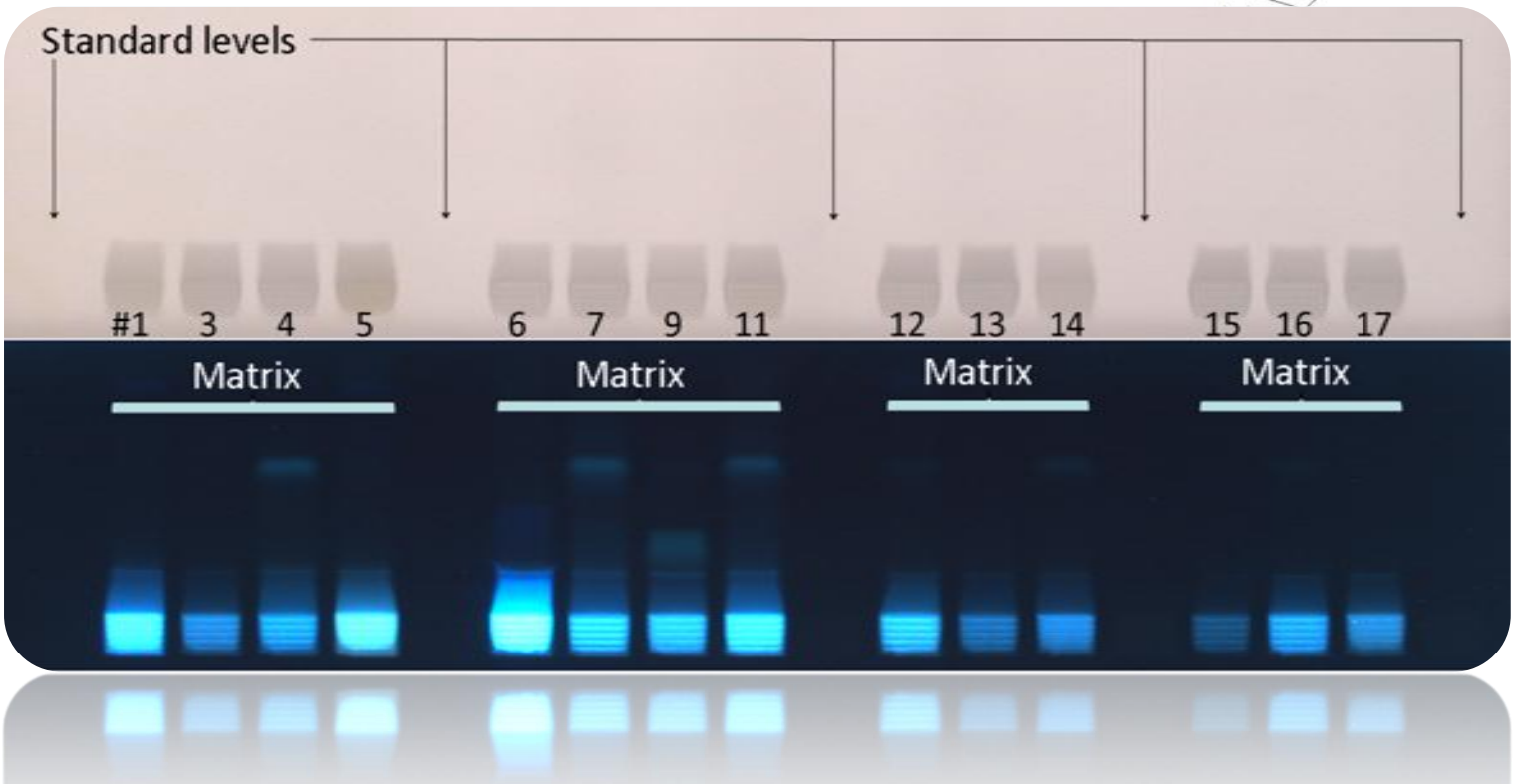
Confirmation by HPTLC-ESI-MS



Quality of honey → HMF







Method comparison

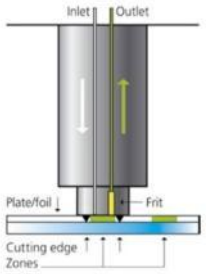
→ Traditional methods (photometry, HPLC) *versus* HPTLC

Concentration of HMF in honey						
Honey sample #	Winkler	HPLC-UV		HPTLC-UV		
	HMF in honey, mg/kg	HMF in honey, mg/kg	Deviation to Winkler method, %	Mean HMF in honey, mg/kg	Deviation to Winkler method, %	**Deviation to HPLC, mg/kg**
1	95.3	n/a	n/a	75.2	-22	**n/a**
2	41.8	n/a	n/a	30.8	-30	**n/a**
3	46.1	38.5	-16	39.3	-16	**0.8**
5	17.6	13.5	-23	13.7	-20	**0.2**
7	21.6	18.1	-16	18.8	-13	**0.7**
8	40.2	30.4	-24	28.7	-26	**1.7**
10	23.9	n/a	n/a	25.1	+5	**n/a**
Mean			20		19	**0.9**

Cooperation with Apicultural Institute, Celle and Stuttgart

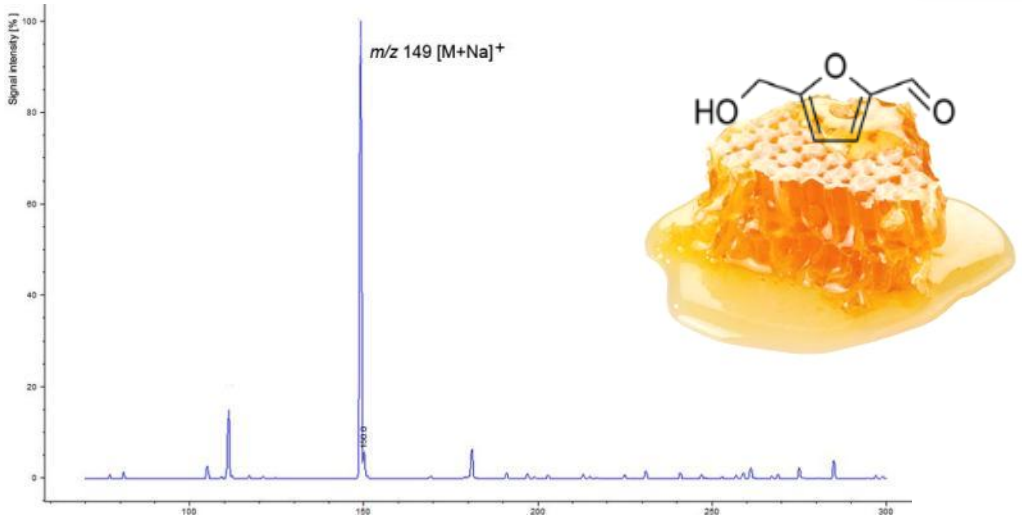
E. Chernetsova, A. Revelsky, G. Morlock, *Anal Bioanal Chem* 401 (2011) 325-332

Method comparison



→ HPTLC: UV *versus* MS

Sample#	HPTLC-UV			HPTLC-ESI-MS, SIM		Deviation UV *versus* MS
Plate 1	Mean HMF in honey, mg/kg	*%RSD* (n = 3)	Reprod. (*%RSD*, n=2)	Mean HMF in honey, mg/kg	*%RSD* (n = 3)	HMF in honey, mg/kg
1	37.7	3.7	4.4	36.4	18	1.3
2	50.1	2.8	2.3	57.1	10	-7.0
3	78.4	1.5	1.9	72.0	2.5	6.4
4	35.0	2.0	3.3	29.2	7.5	5.8
Mean			**3.0**			**5.1**

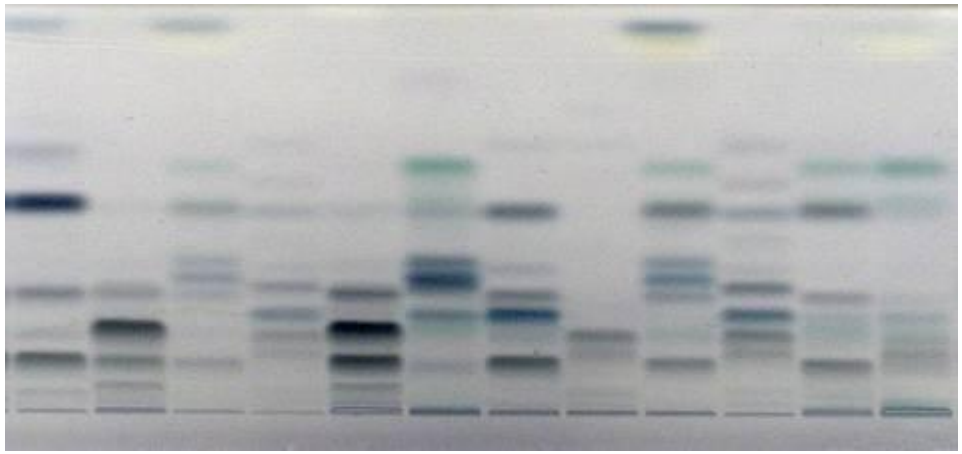


Method comparison

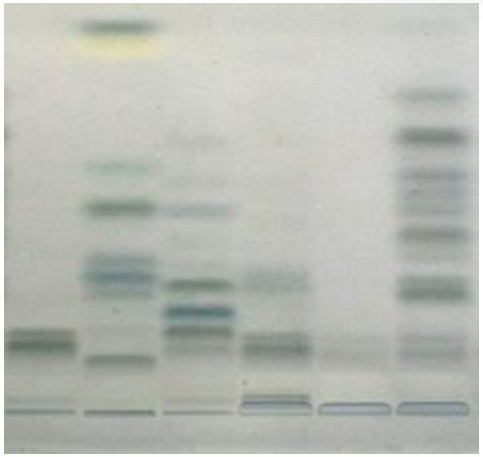
→ Fast methods: HPTLC *versus* Reflectoquant

A. Hostalkova, I. Klingelhöfer, G. Morlock, *Anal Bioanal Chem* 405 (2013) 9207–9218

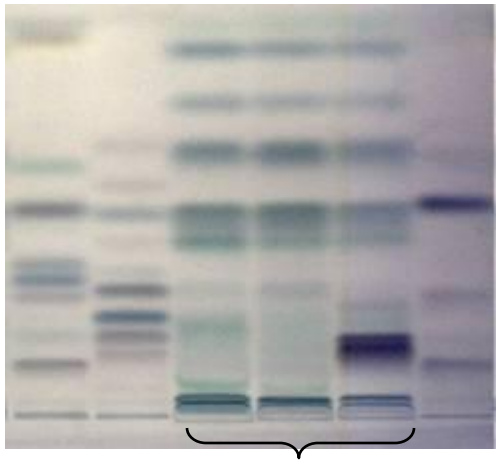
Analysis of biopolymers → monomeric units



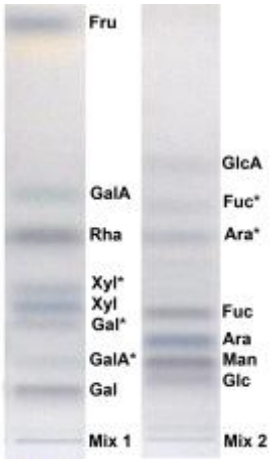
Carr JBKM Mix 1 Mix 2 Guar Trag Gum Xan Mix 1 Mix 2 Kara Pect

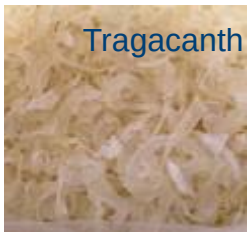


Starch Mix 1 Mix 2 CMC Cell HPMC



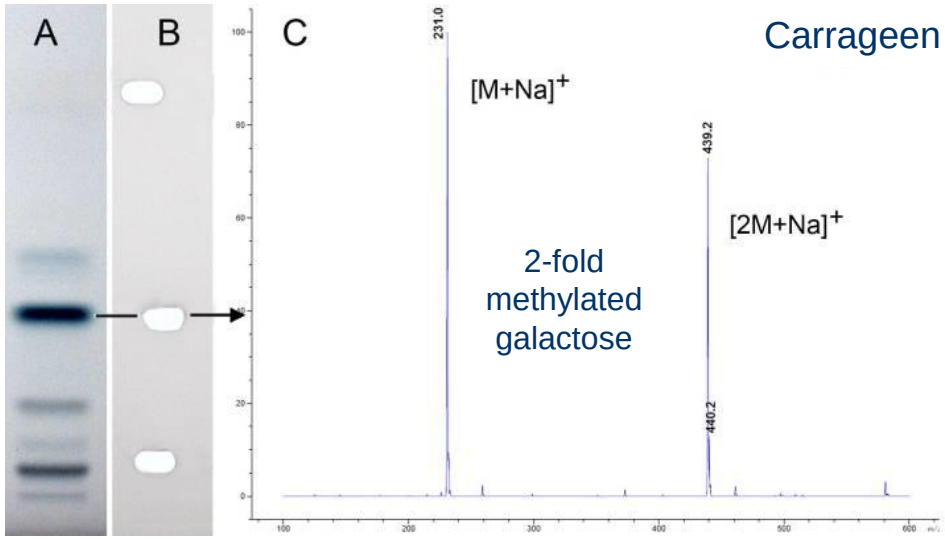
Mix 1 Mix 2 Alginate Agar





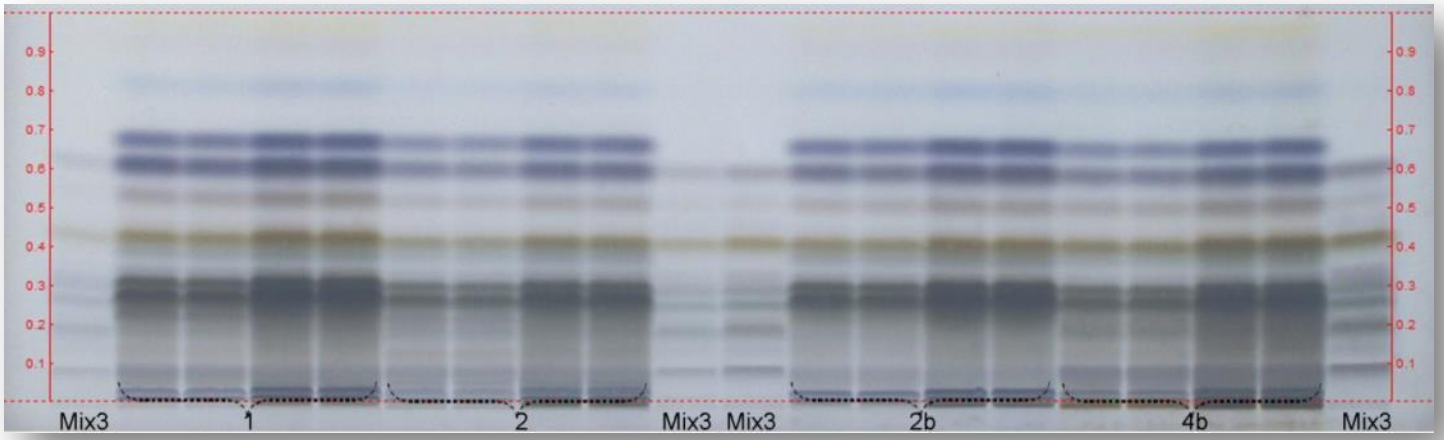
G. Morlock, F. Gamlich, *J Planar Chromatogr* 25 (2012) 244-250

Analysis of biopolymers → monomeric units



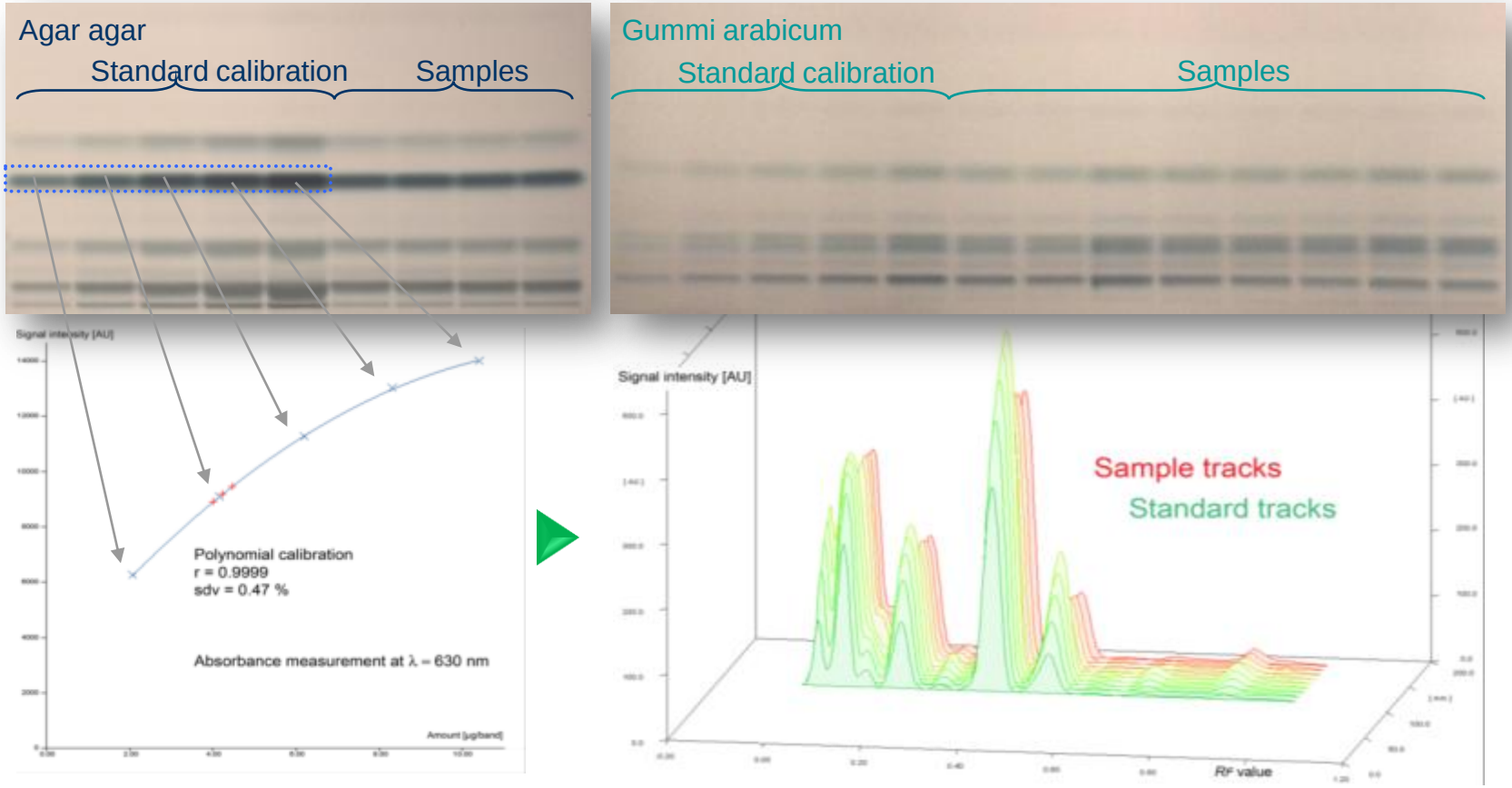
Carrageen

Ocimum basilicum



A. Yili *et al.*, *J. Planar Chromatogr.* 27 (2014) 11-18

Quantitation



G. Morlock, D. Schick, W. Schwack, in preparation

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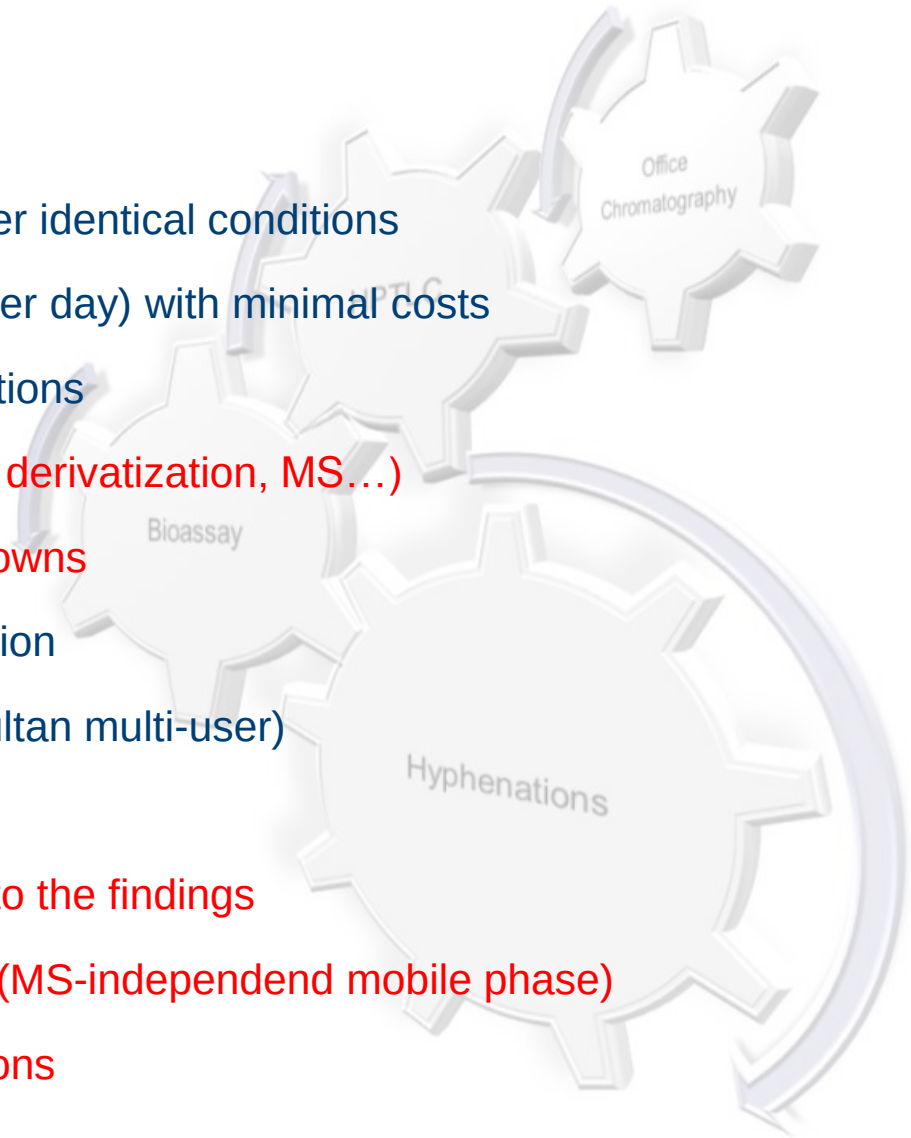
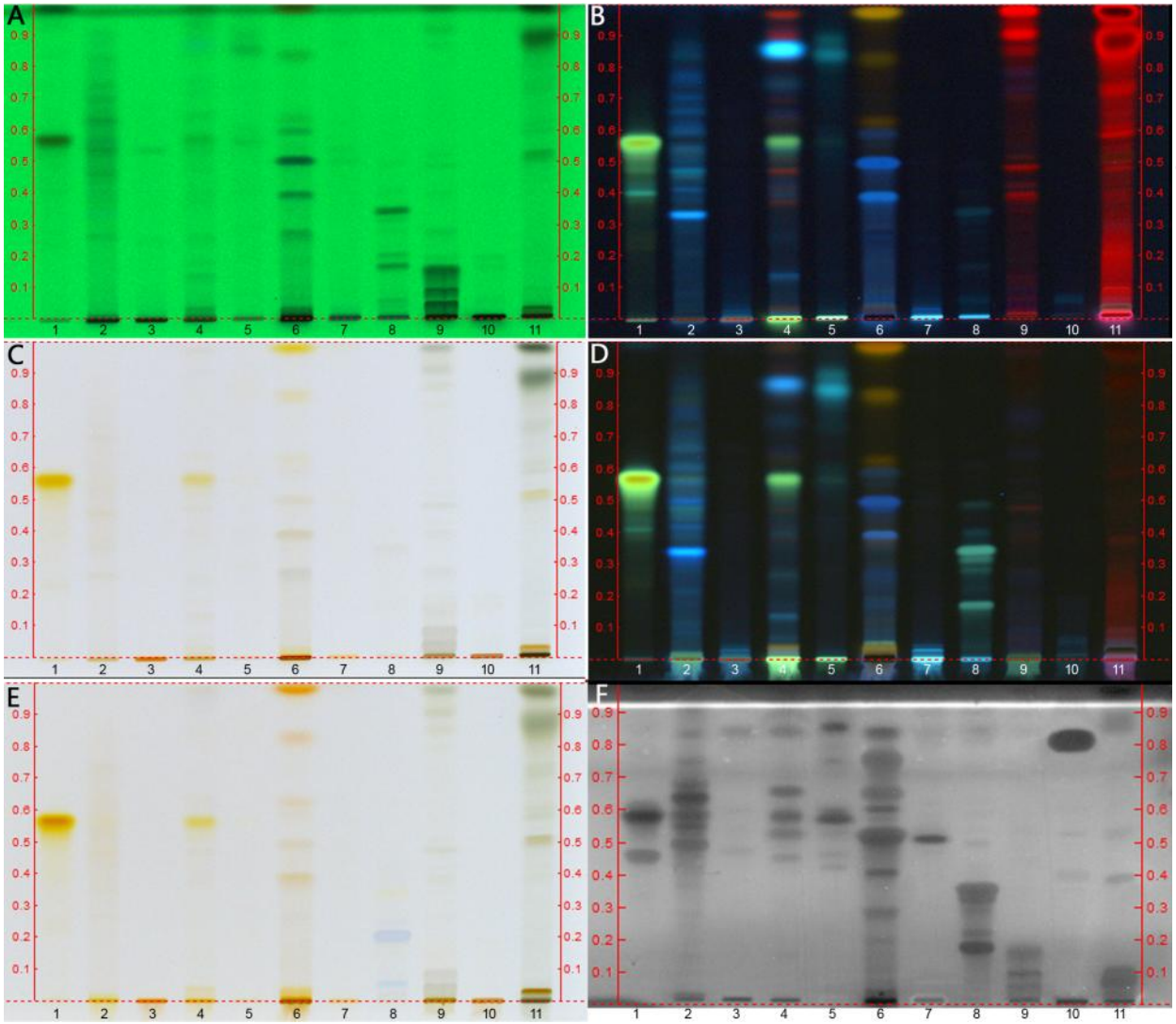


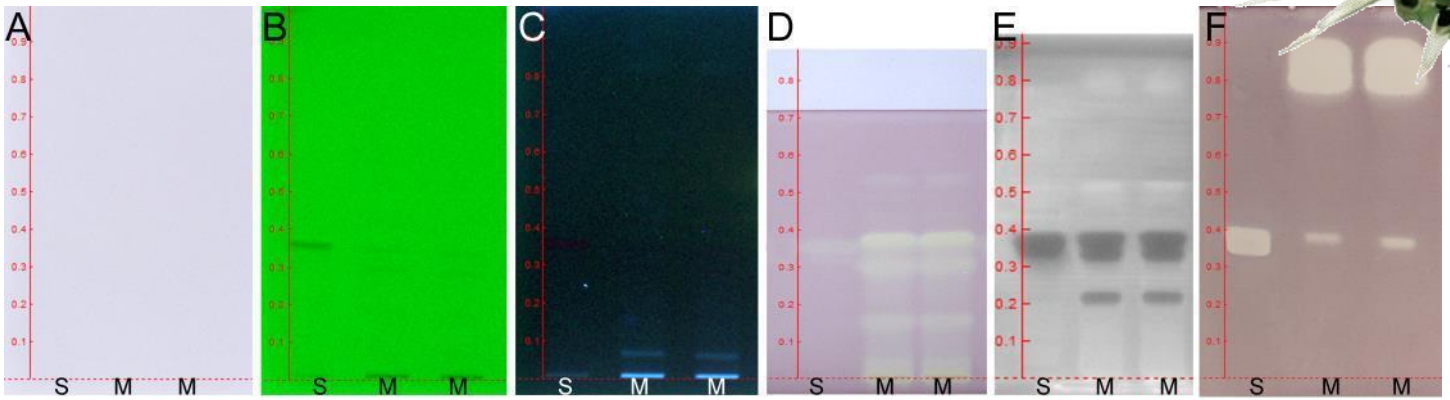
EDA of 11 plant species

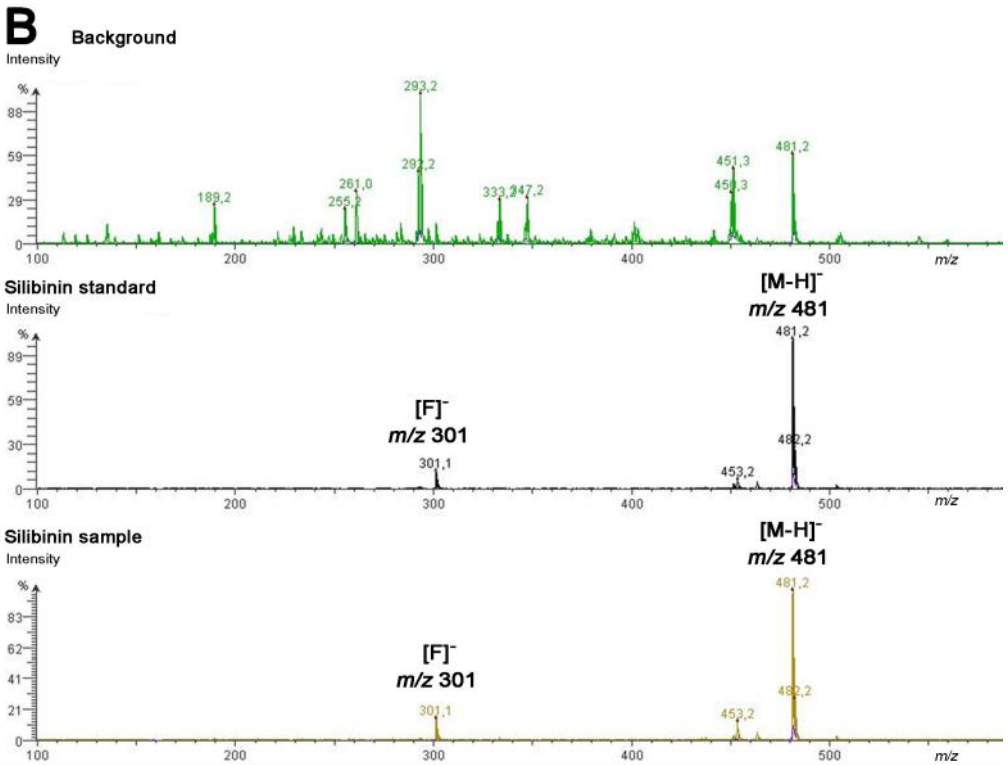


1 turmeric
2 licorice
3 saffron
4 mixed herbs I
5 ginger
6 mixed herbs II
7 chilli pepper
8 milk thistle
9 green tea
10 dates seed
11 spirulina

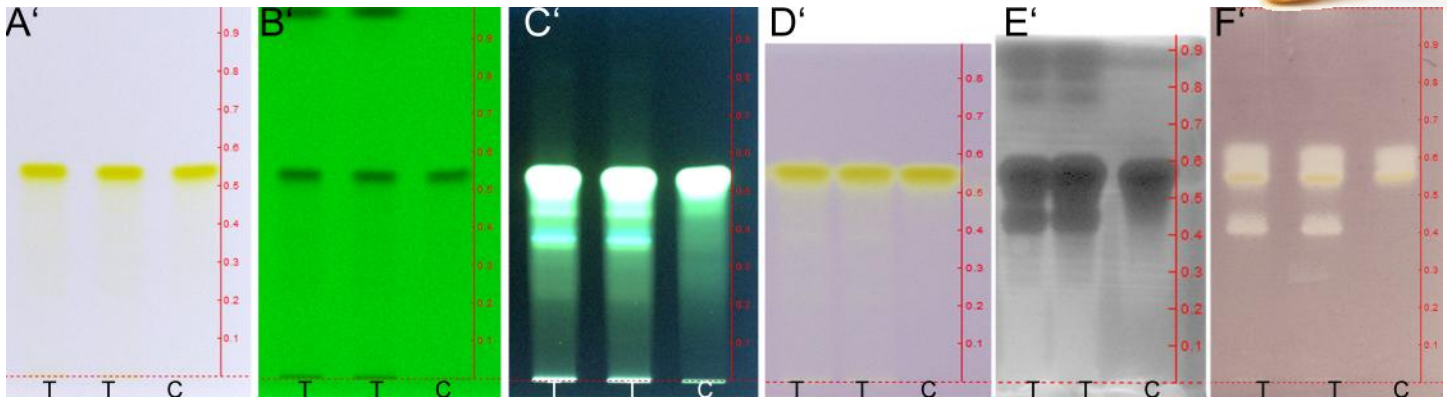
M. Taha, M. Krawinkel, G. Morlock, *J Chromatogr A*, in print

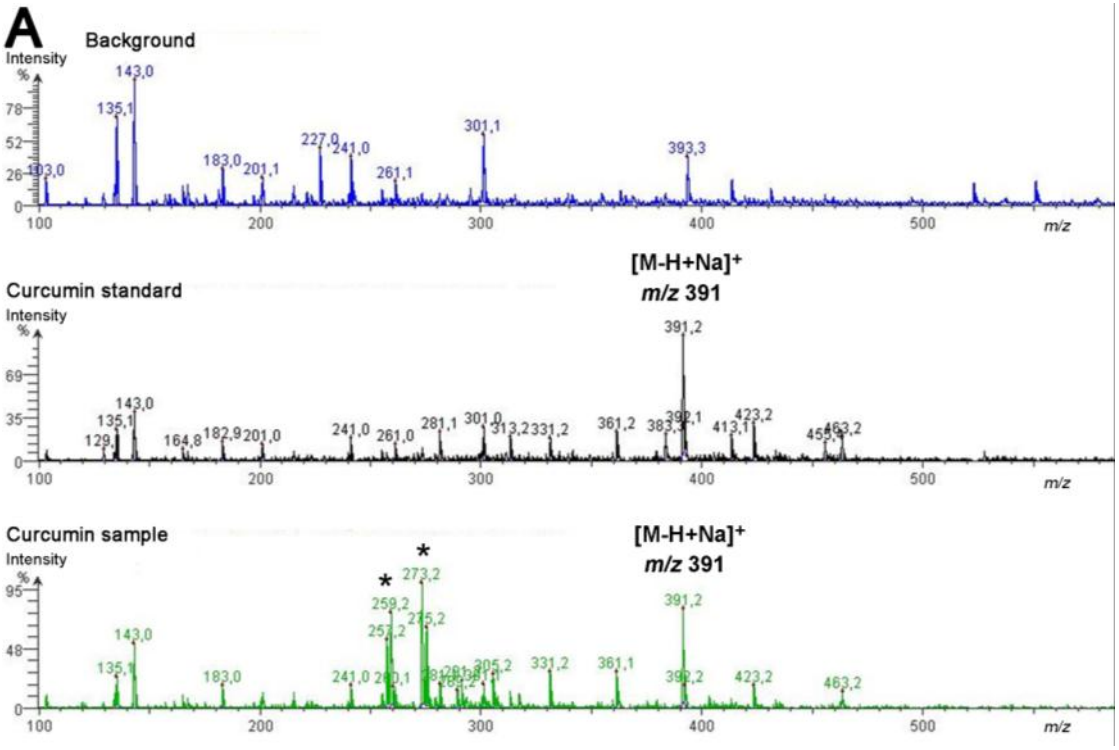
EDA of milk thistle (*Silybum marianum*) seeds





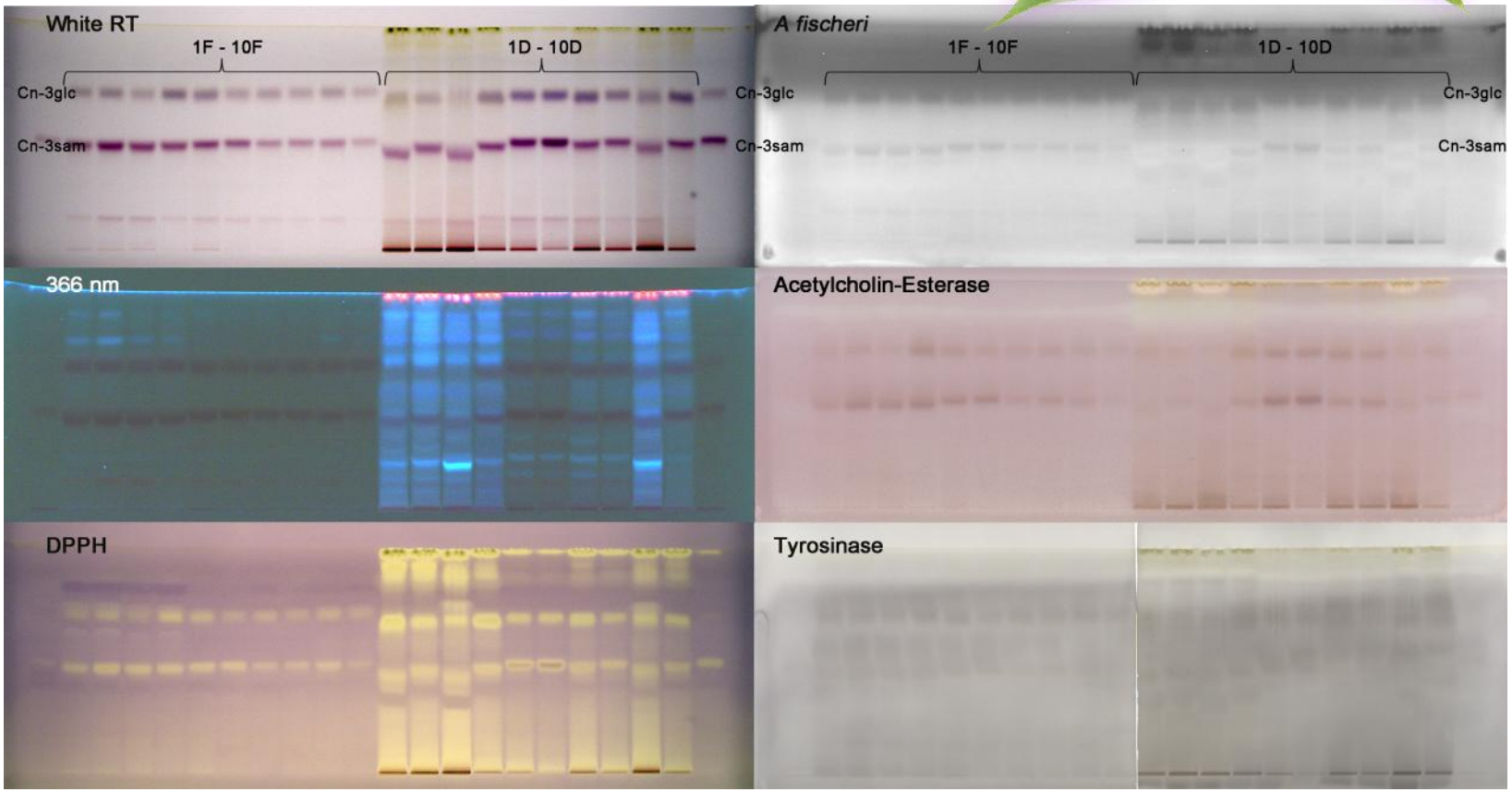
EDA of turmeric (*Curcuma longa*) rhizomes



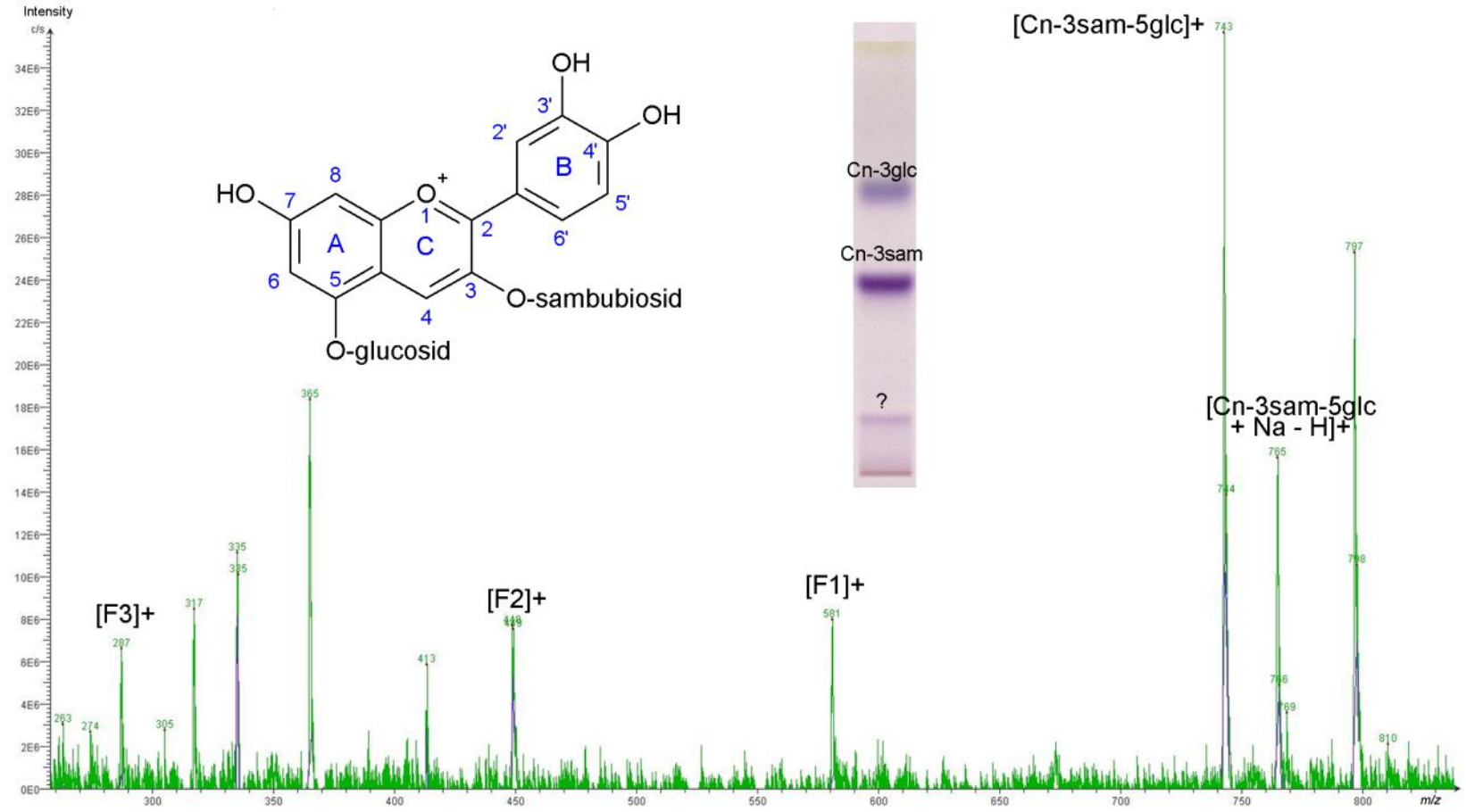


EDA of elderberry (*Sambucus nigra* L.)

→ fresh *versus* dried fruit samples

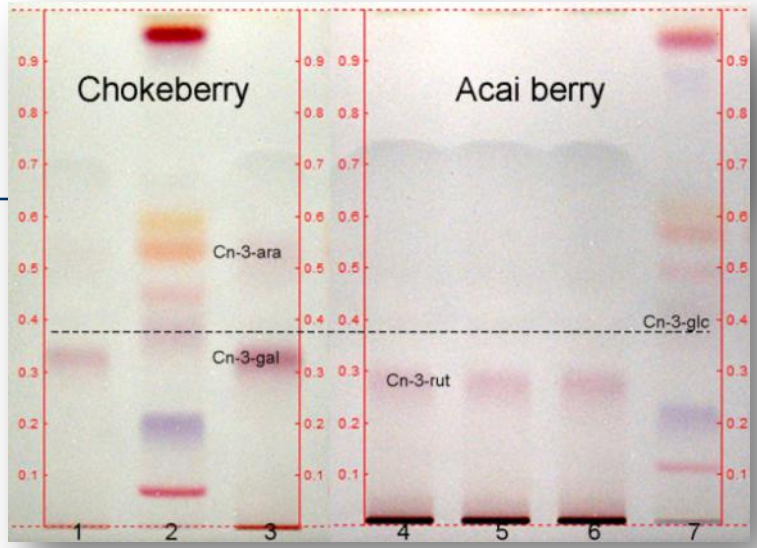


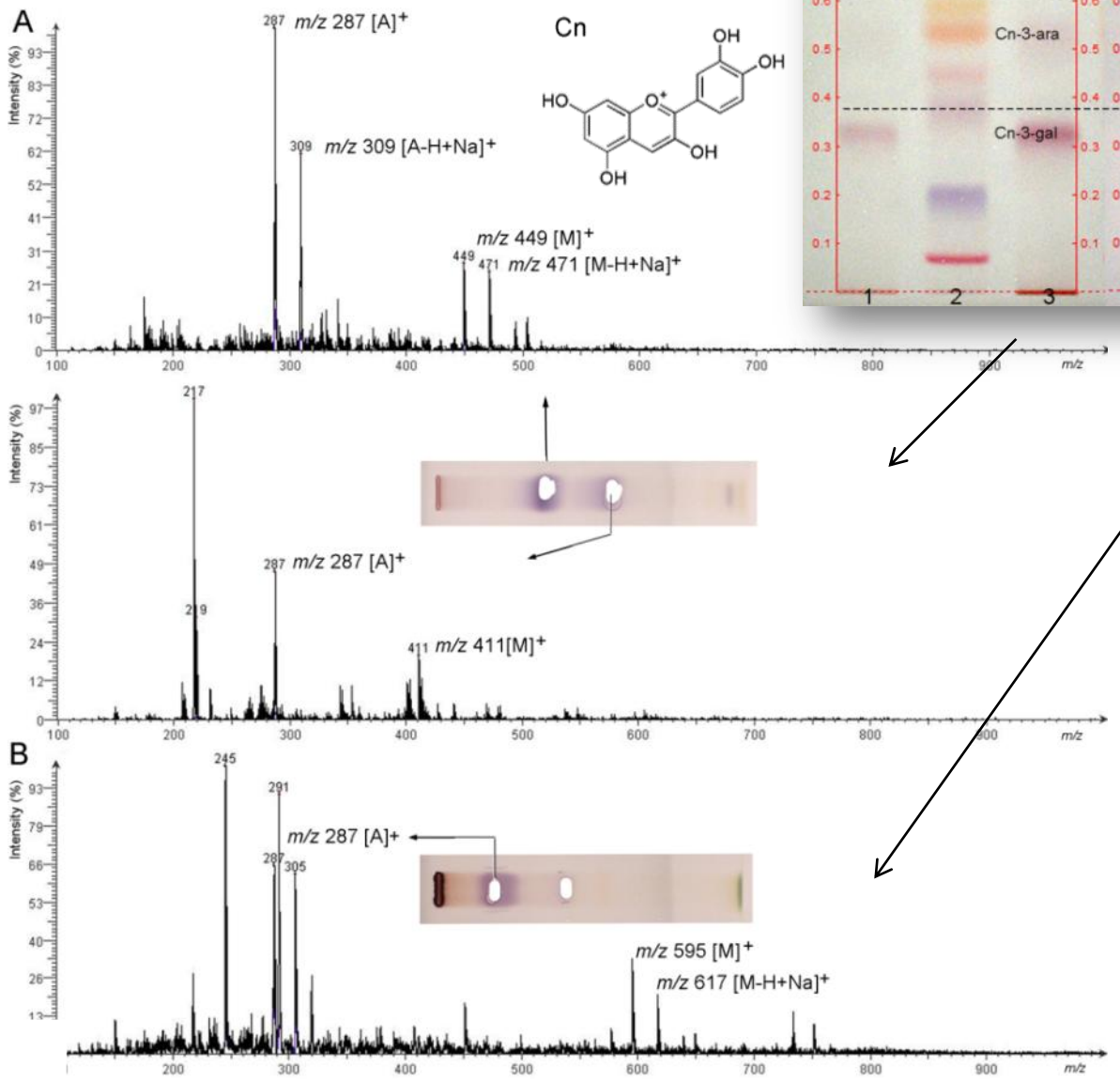
HPTLC-ESI$^+$-MS of unknown zone at hR_F 10



S. Krüger, M. Mirgos, G. Morlock, in preparation

Analysis of anthocyanes



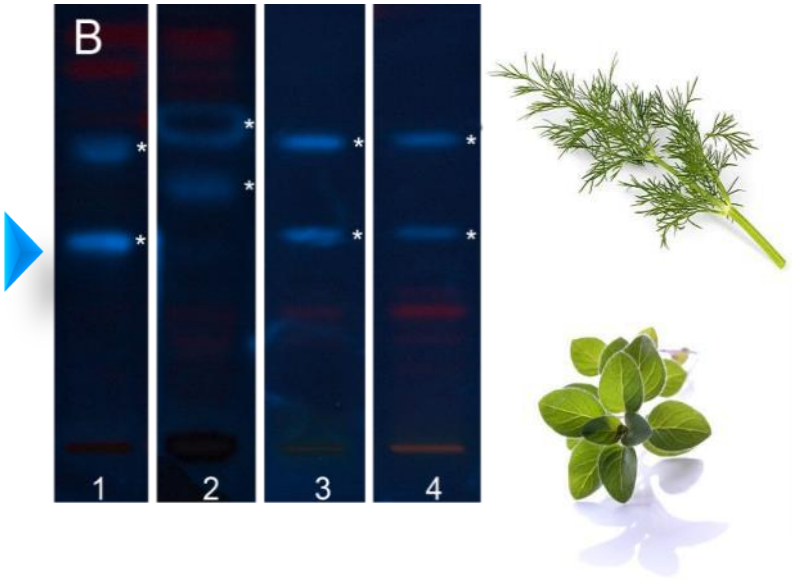


G. Cretu, G. Morlock, *Food Chem* 146 (2014) 104-112

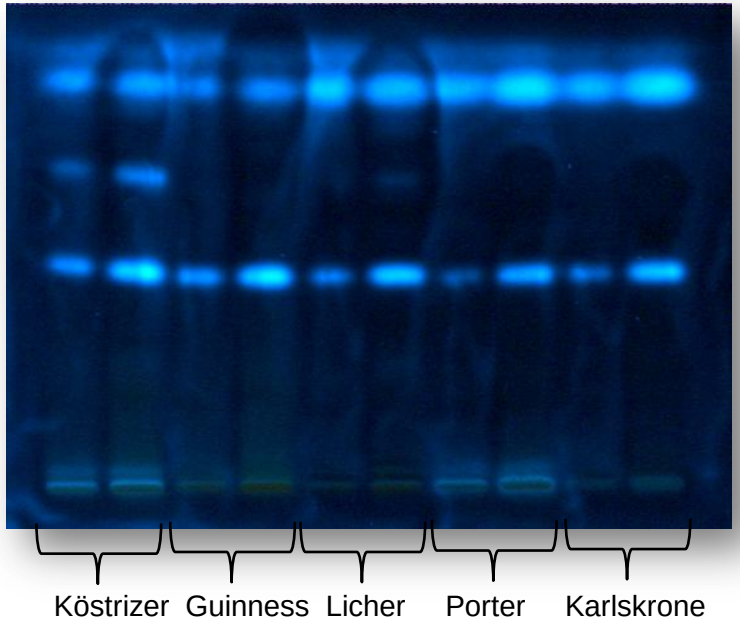
www.wikipedia.de

EDA of estrogen-effective compounds

→ Spices



→ Beer samples



I. Klingelhöfer, G. Morlock, *J Chromatogr A* 1360 (2014) 288-295

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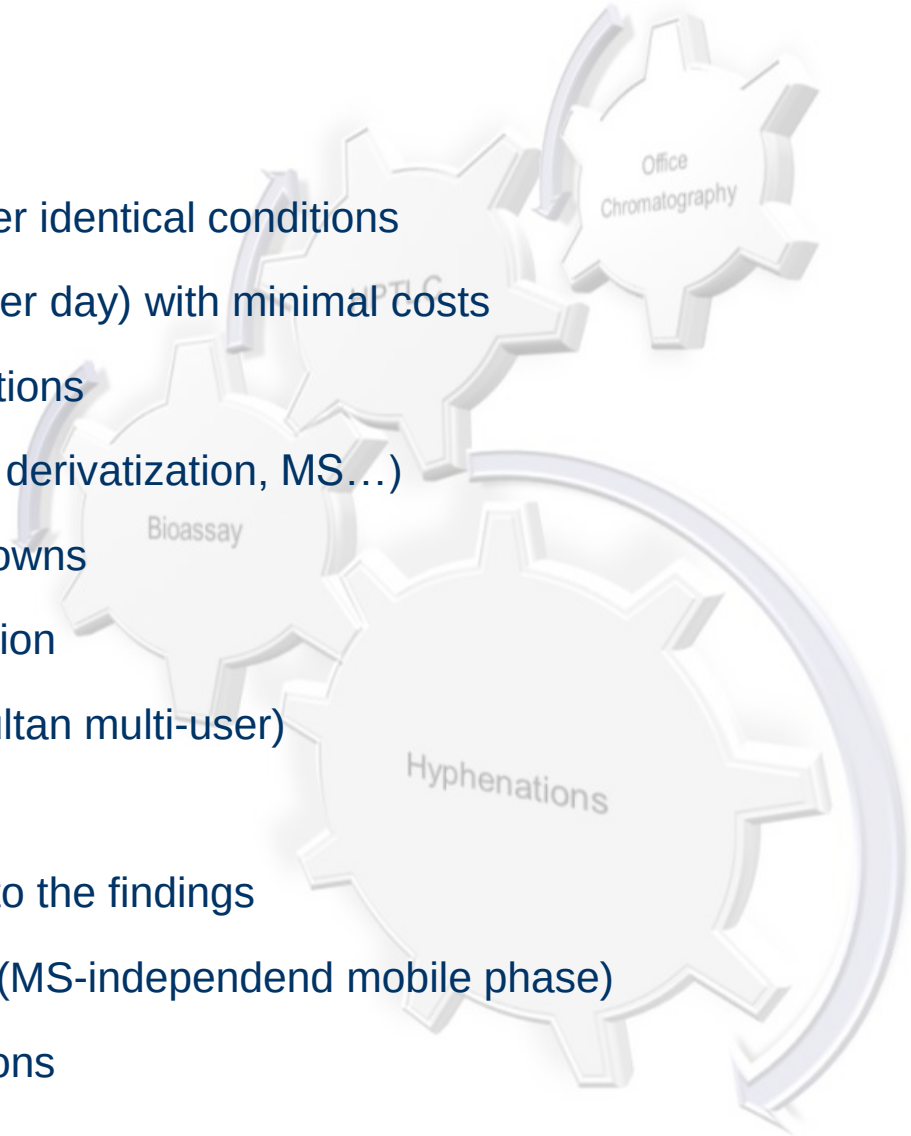
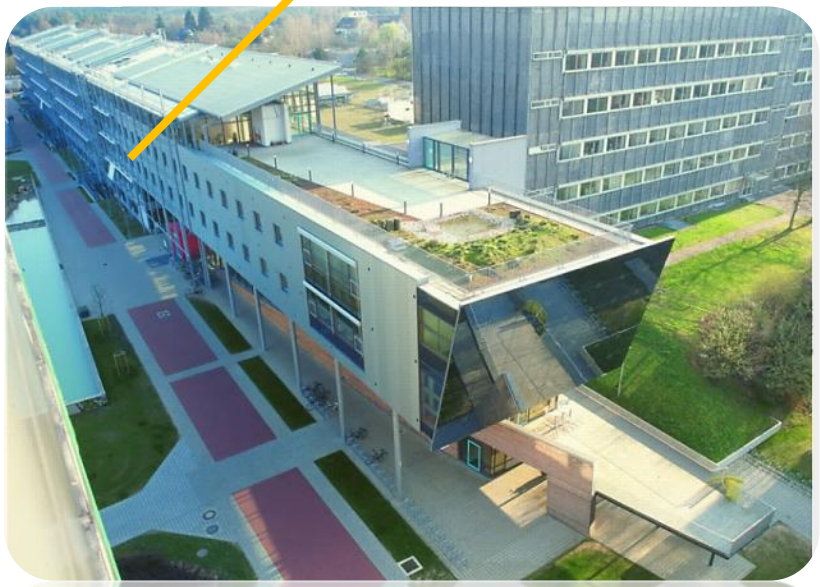
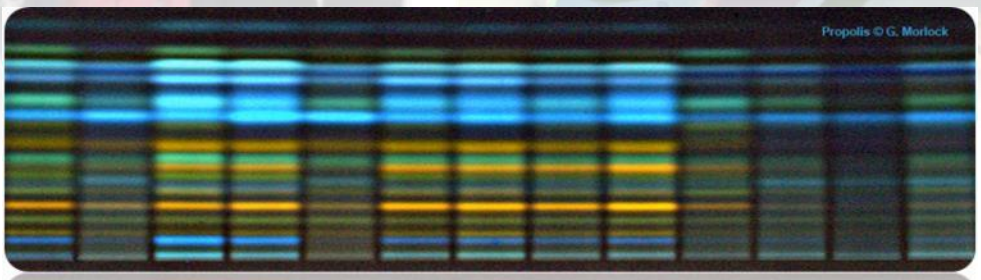


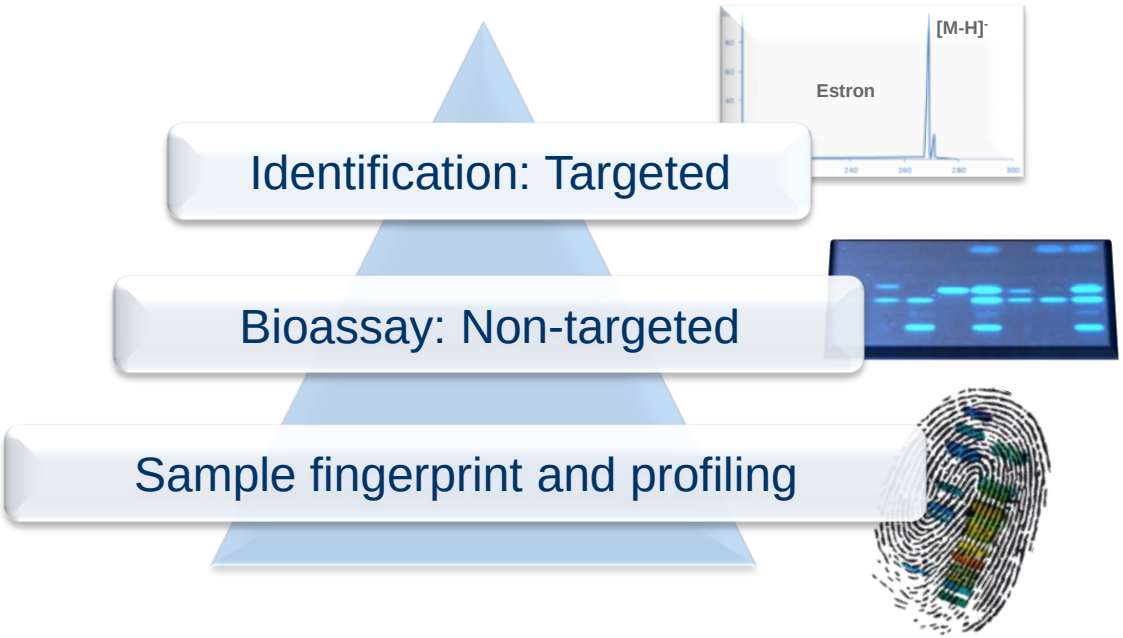
Thank you!

JUSTUS-LIEBIG-UNIVERSITÄT GIESSEN
Food Science





Breakthrough in the hyphenation HPTLC-EDA-MS – a must-have method in the analyst´s toolbox



Gertrud Morlock, Chair of Food Science
Justus Liebig University Giessen

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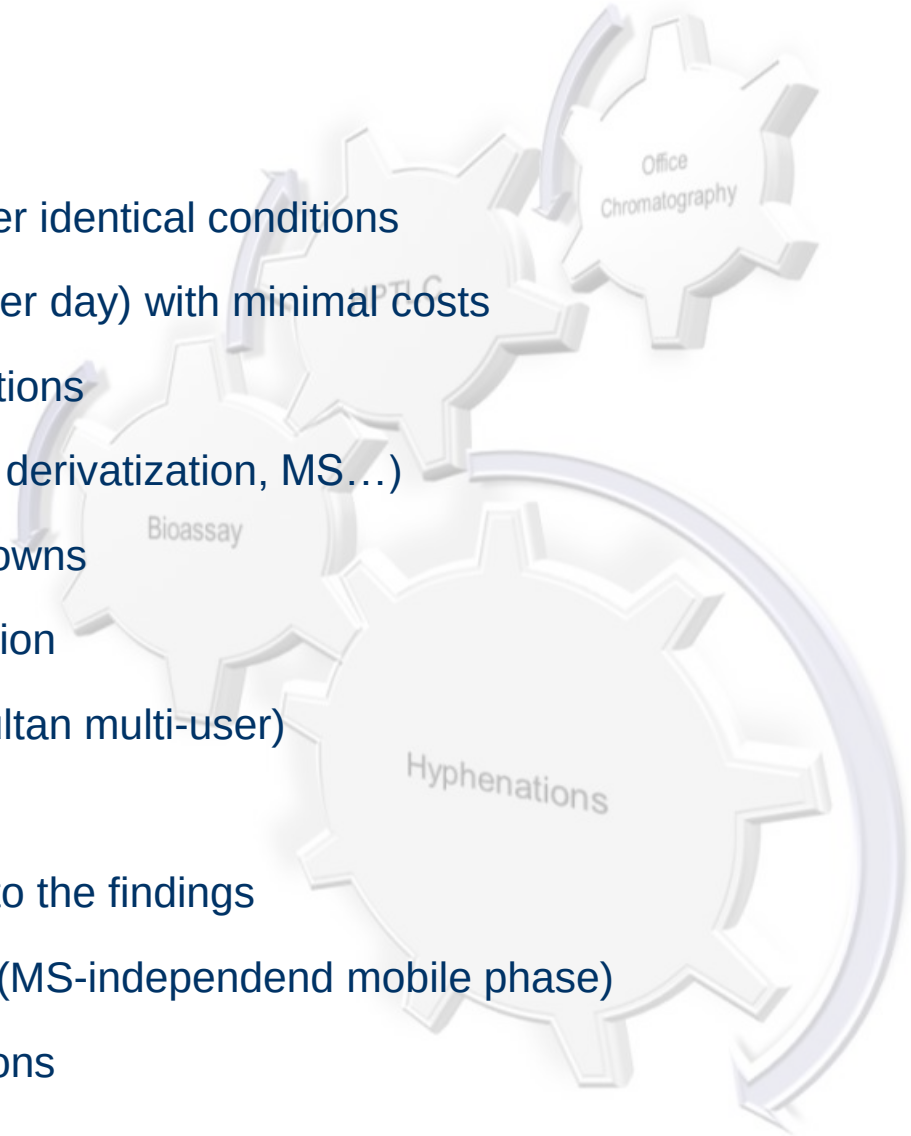
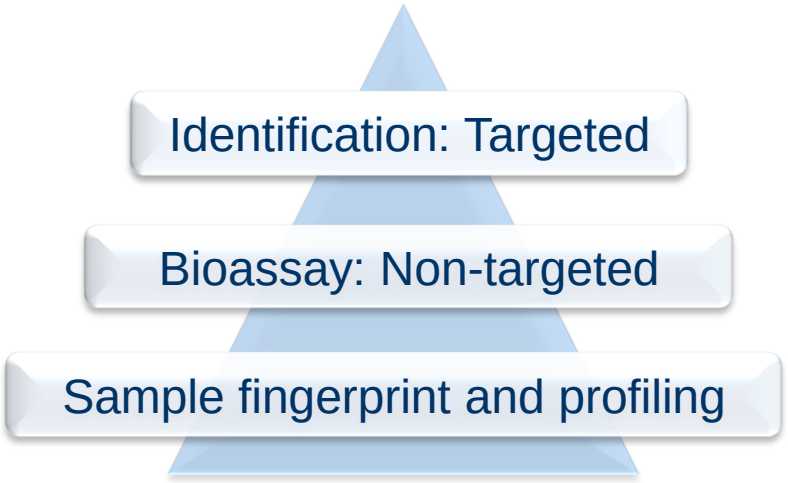


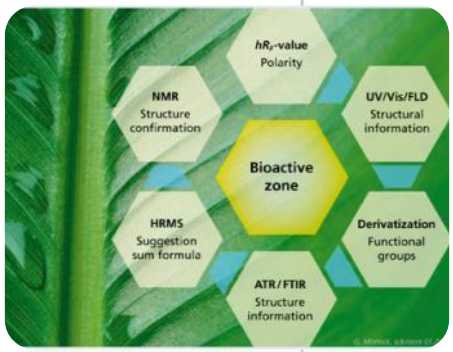
Impact

- *Only a part is represented by target analysis or multi-methods*
- *Complex samples contain thousands of compounds*
- X *It remains largely unclear which peaks are bioactive*
- X *Samples to be identified as risky or bioactive*



Novel effect-directed profiling

Effect-directed link to the compound



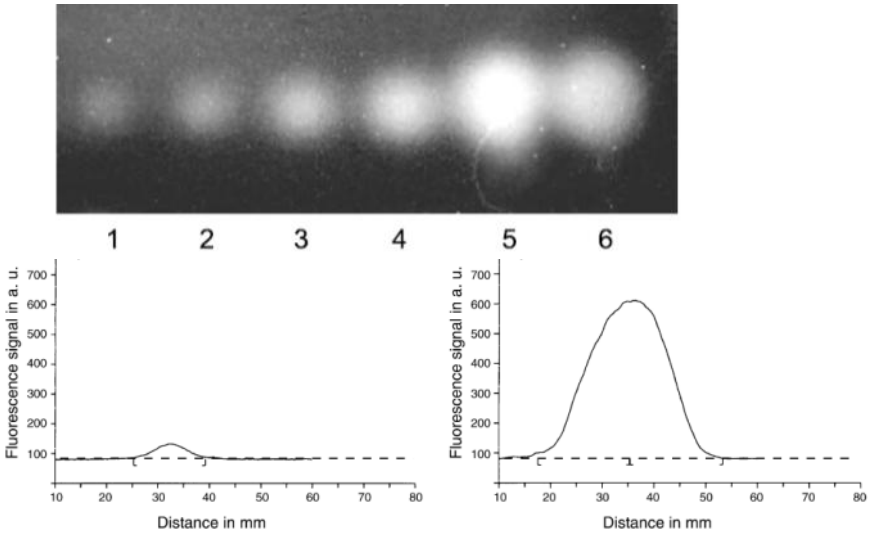
TLC/HPTLC-EDA

- Microchemical derivatization
  - DPPH* reagent - radical scavenging test
  - ABTS reagent - radical scavenging test
  - Bleaching of ß-carotene - antioxidant test
- Microbiological detection
  - Algae
    - *Pseudokirchneriella subcapitata*
  - Fungi
    - *Candida albicans*
    - *Trichophyton rubrum*
    - *Cryptococcus neoformans*
    - *Cladosporium cucmerinum*
    - *Apergillus niger*
    - *Colletotrichum acutatum/gloeosporioides/fragaria*
  - Bacteria
    - *Pseudomonas savastanoi*
    - *Bacillus subtilis*
    - *Aliivibrio fischeri*
    - *Escherichia coli*
    - *Pseudomonas syringae*
    - *Xanthomonas campestris*
    - *Erwinia carotovora/herbicola*
    - *Cryptococcus neoformans*
  - Yeast
    - Planar yeast estrogen screen (pYES)
- Biochemical assays
  - (Cholin)esterase assay
  - α/ß-Glucosidase assay
  - Xanthine oxidase assay
  - Tyrosinase oxidase assay

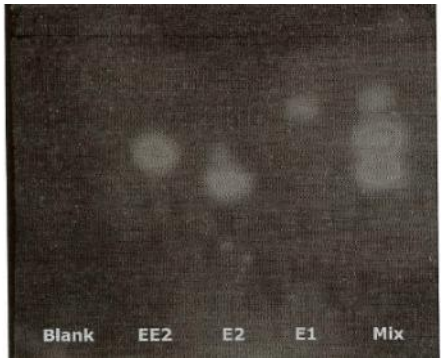
G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

Challenge: Avoid zone broadening

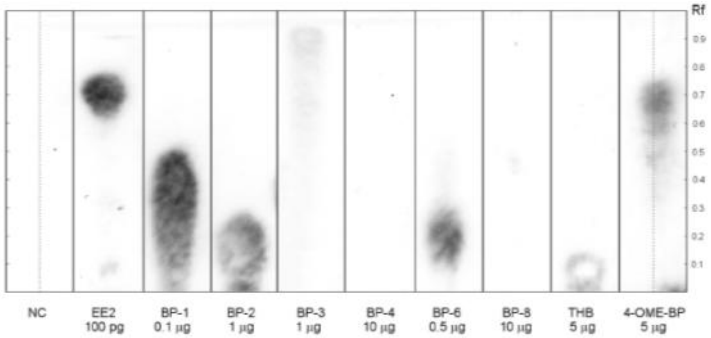
Goodall & Levi, Nature 158, 675–676 (1946)



Mueller *et al*. *Chromatographia* 60, 207-211 (2004)

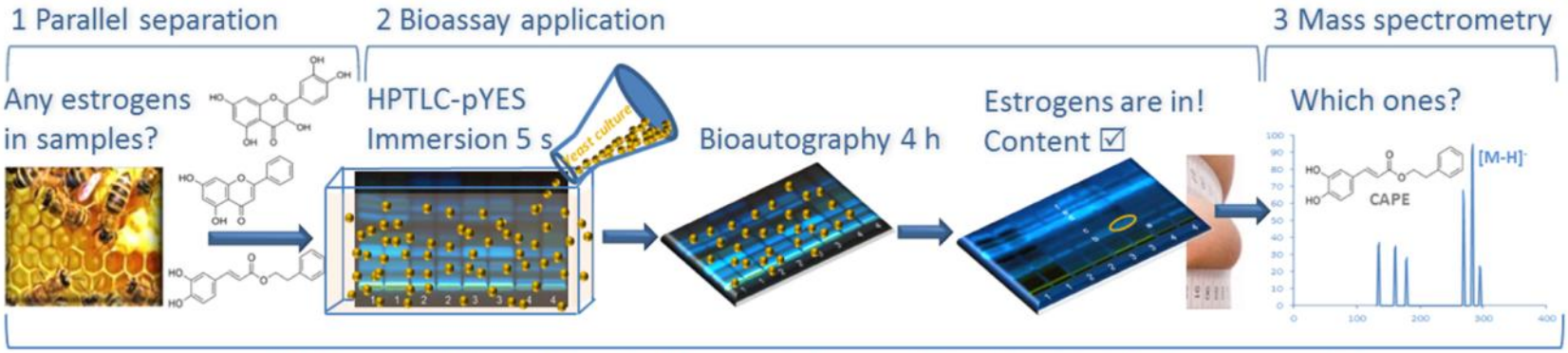


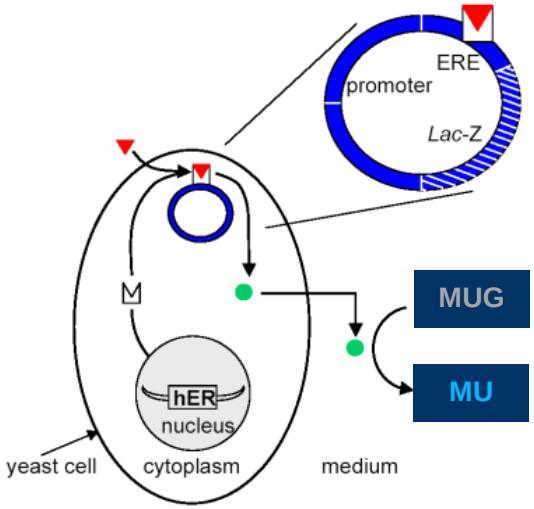
Schönborn & Grimmer,
J. Planar Chromatogr. 26, 402-408 (2013)



Buchinger *et al.*
Anal. Chem. 85, 7248-7256 (2013)

Detection of endocrine disrupting compounds (EDCs)





Modified from draft of pYES expert group

Planar yeast estrogen screen (pYES)

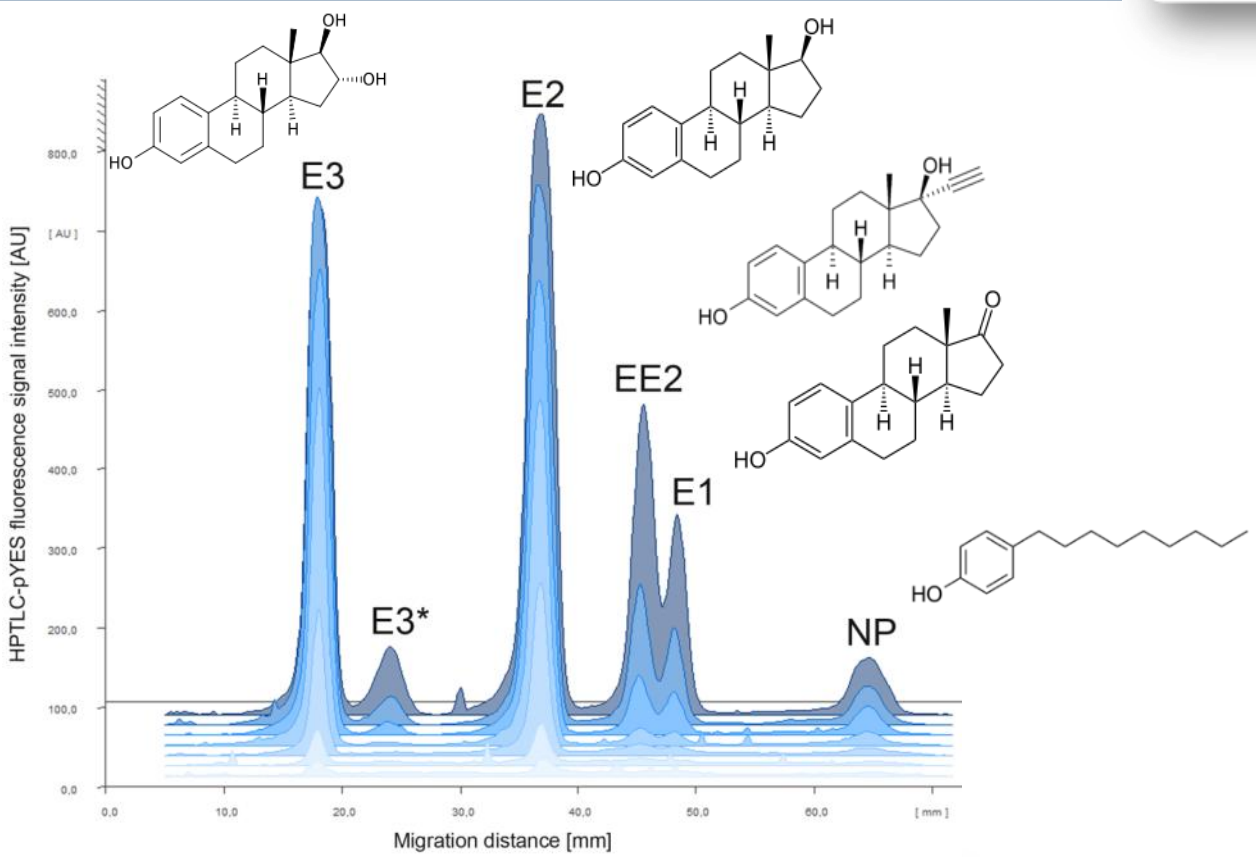
→ using human estrogen receptor hERα

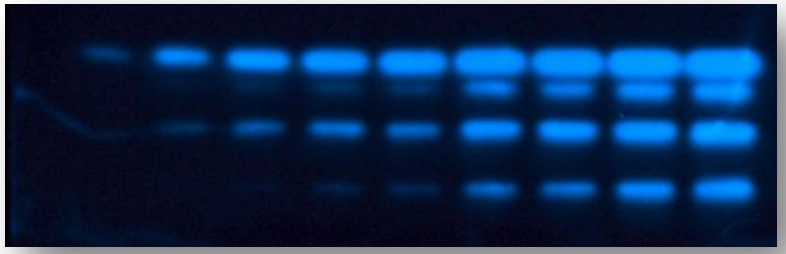
→ in *Saccharomyces cerevisiae*

1. Routledge & Sumpter, Environ. Toxicol. Chem. 15 (1996) 241
2. McDonnell *et al.*, J. Steroid Biochem. Mol. Biol. 39 (1991) 291

→ blue fluorescent 4-methylumbelliferone

Detection of EDCs



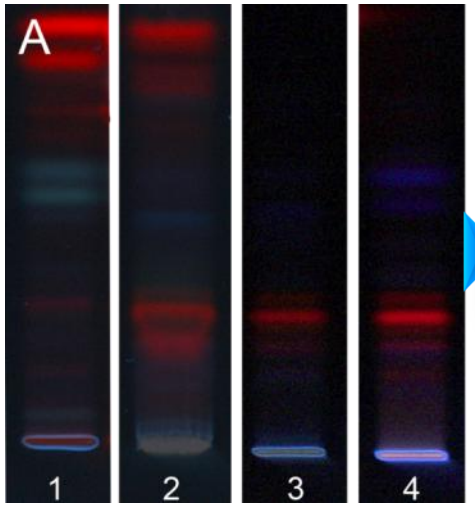


	pg/band
E1	25 – 1000
EE2	0.5 – 20
E2	0.5 – 20
E3	25 – 1000

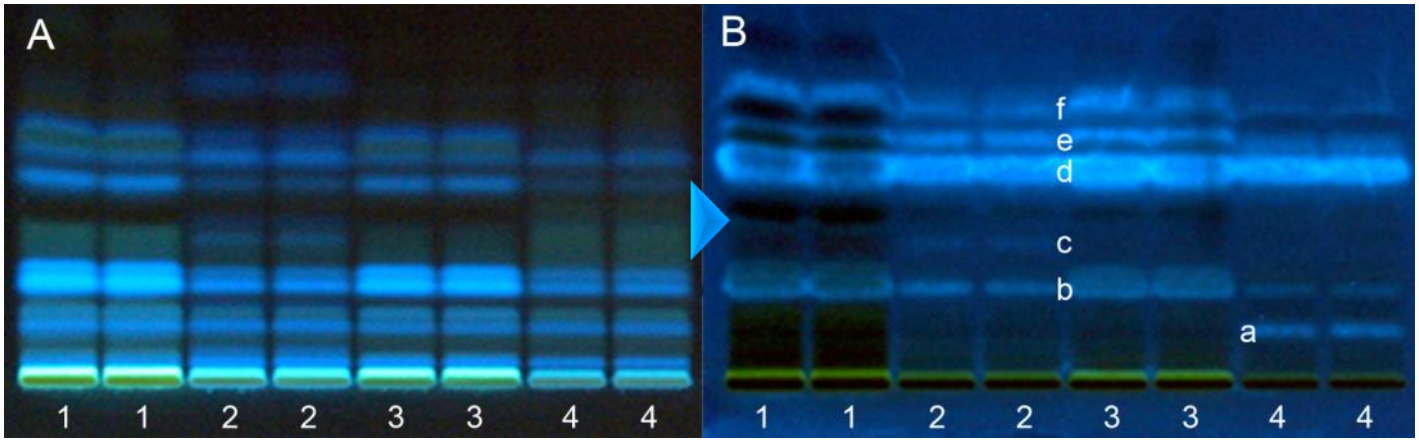
I. Klingelhöfer, G. Morlock, *J. Chromatogr. A* 1360 (2014) 288-295

Detection of EDCs

→ Spices/tea

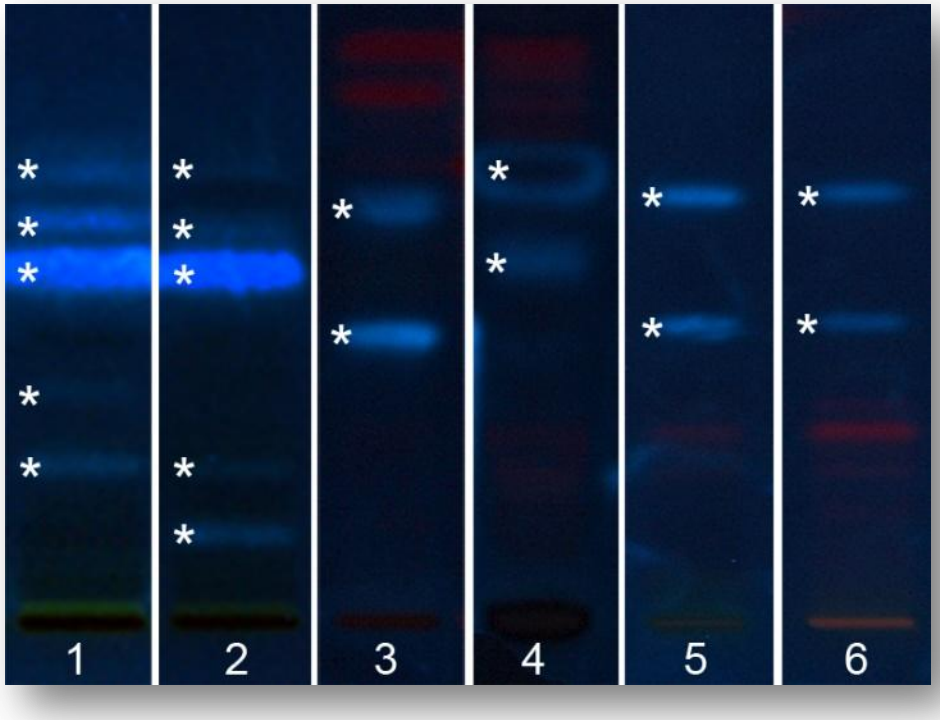


→ Propolis samples

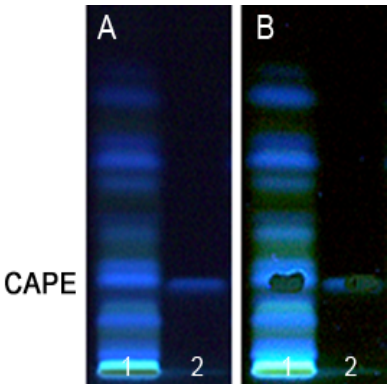


G. Morlock, I. Klingelhöfer, *Anal. Chem*. 86 (2014) 8289–8295

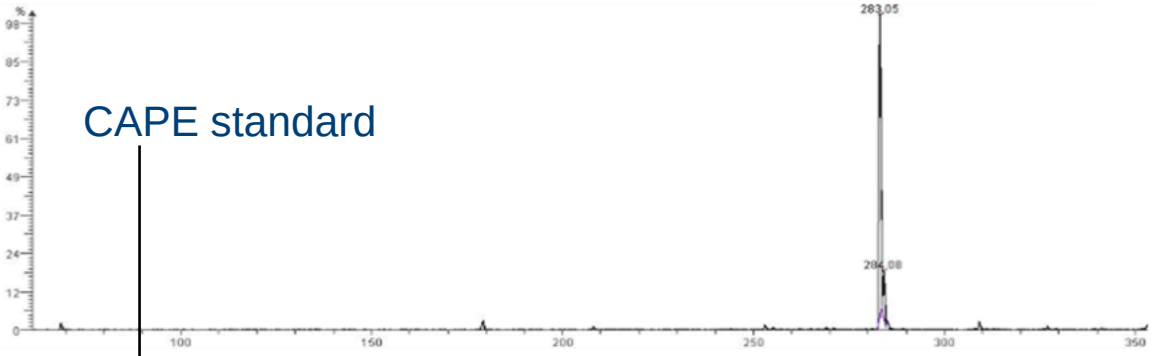
What is it?



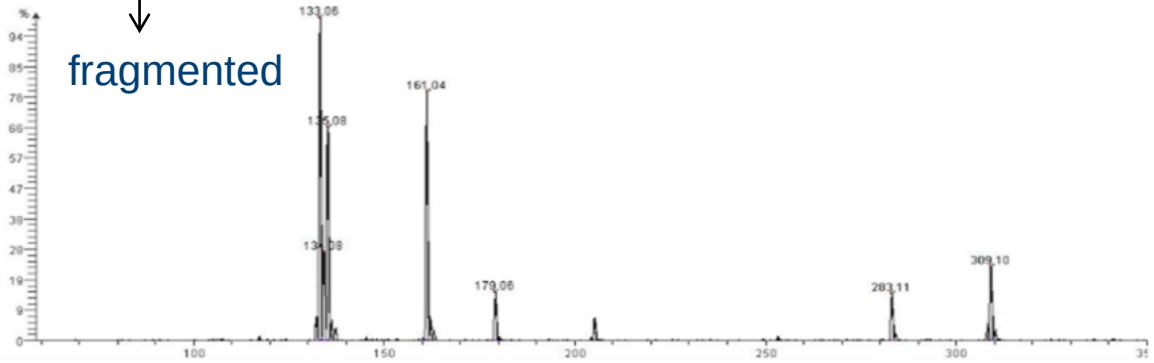
CAPE?



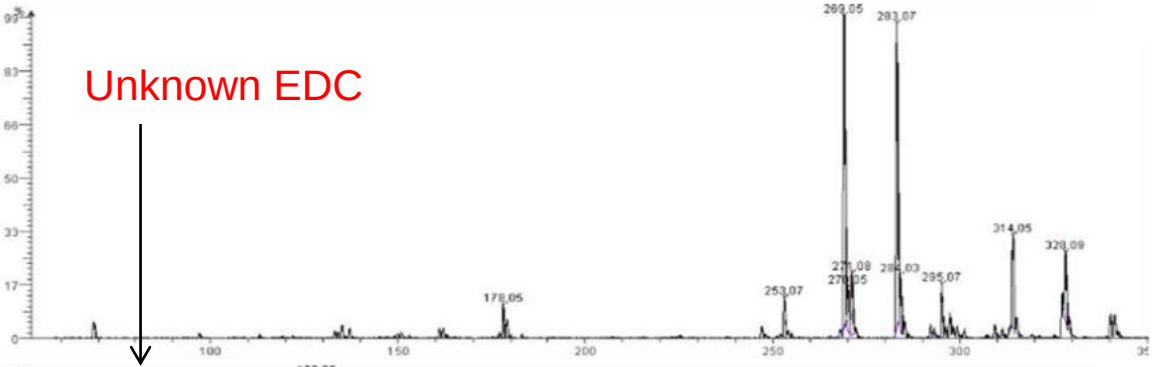
CAPE standard



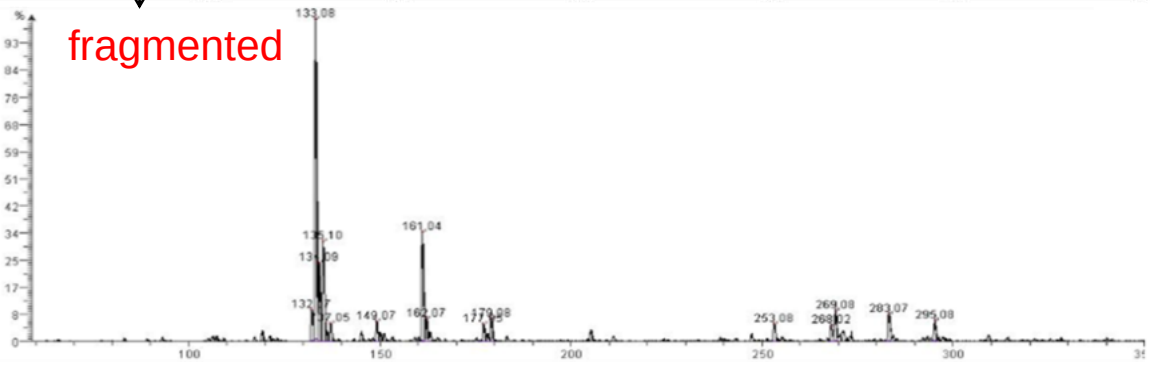
fragmented



Unknown EDC



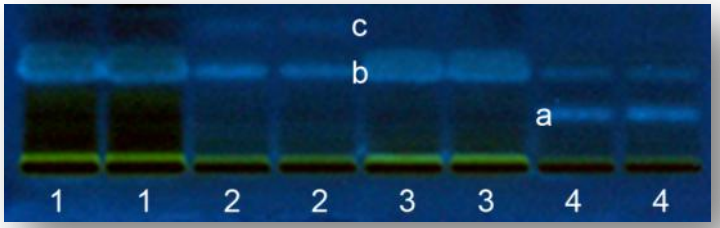
fragmented

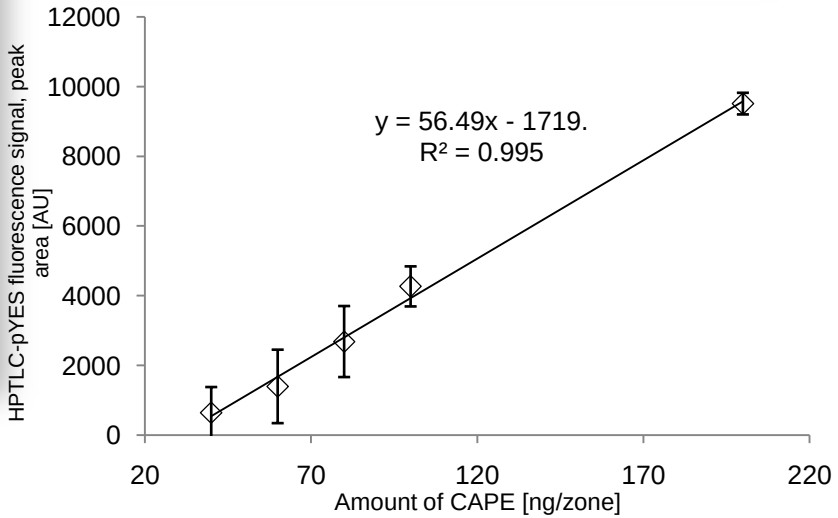


Quantitation of CAPE in propolis

Propolis sample	CAPE content in sample [µg/mL]	CAPE content [µg/g] referred to propolis weight (n=2)
P1 (30 %)	481	2028
P2 (30 %)	476	2009
P3 (25 %)	471	2387
P4 (62 %)	348	710
P5 (not specified)	380	380[3]
P6 (250 mg/capsule)	359[1]	1435
P7 (30 mg/lozenge)	22[2]	1089

[1]µg/capsule, [2]µg/pastille, [3]µg/mL



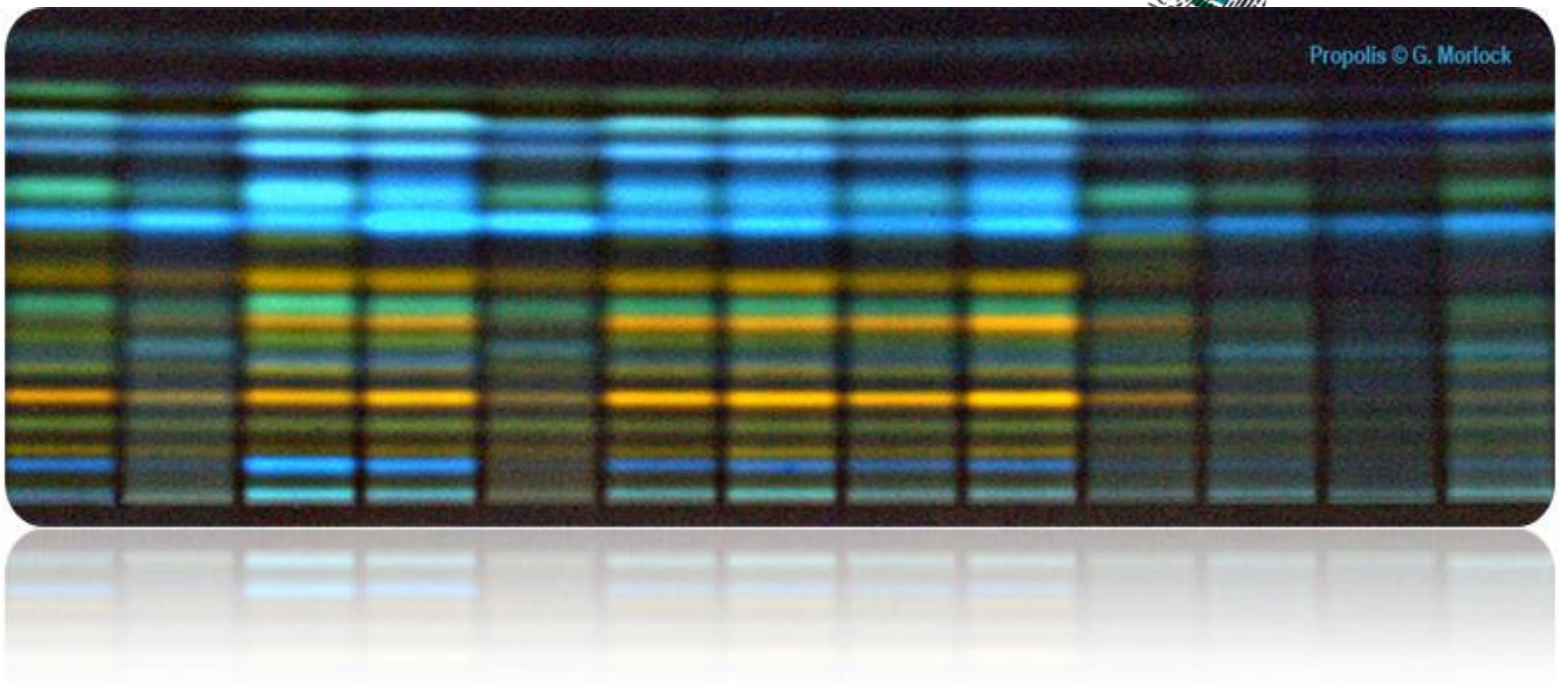


G. Morlock, I. Klingelhöfer, Anal. Chem. 86 (2014) 8289–8295

Fingerprint of phenolic compounds in propolis

→ Screening of >100 samples showed characteristic marker compounds

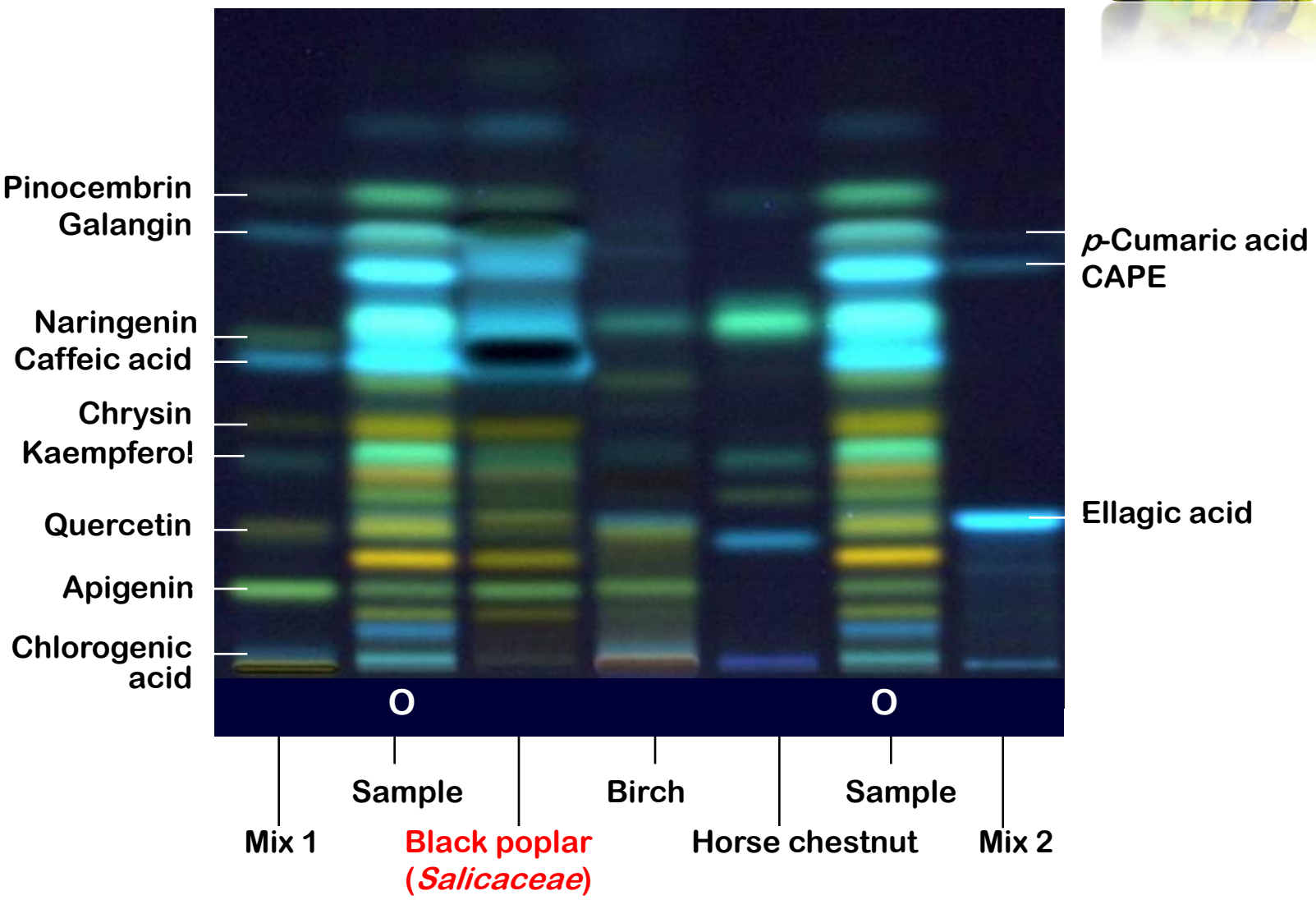
→ Mainly 2 types of German propolis



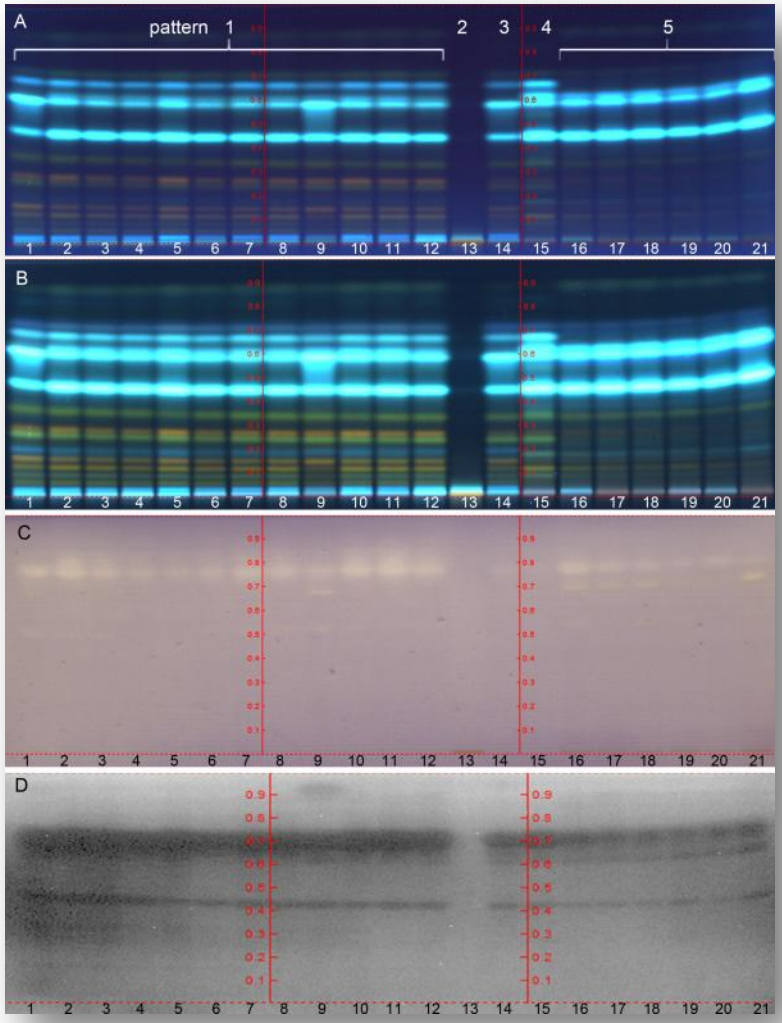
G. Morlock *et al.*, in preparation

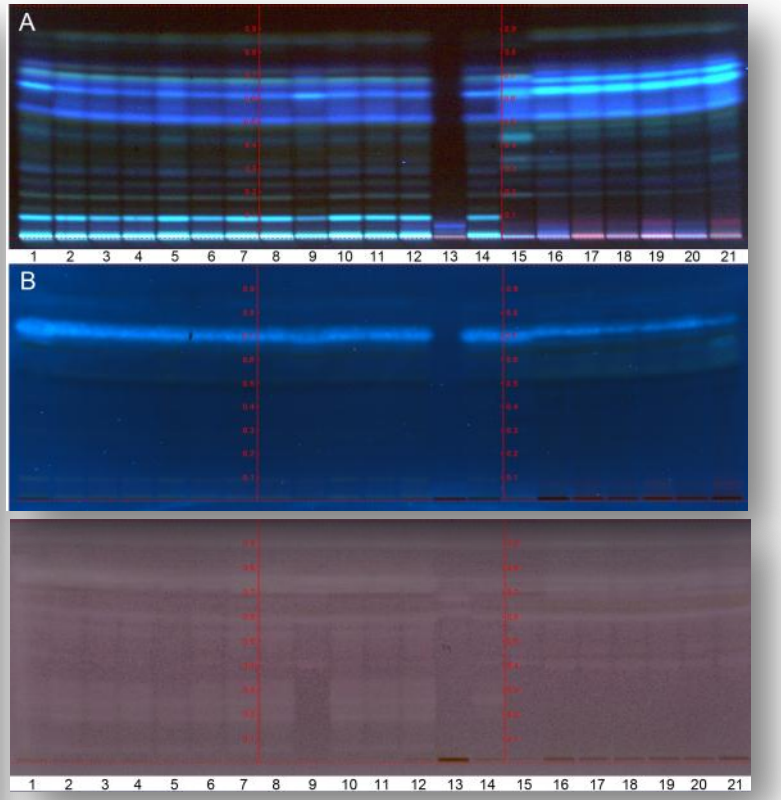
Cooperation with Wala and Apicultural State Institute, Stuttgart

Plant origin of O-type propolis?



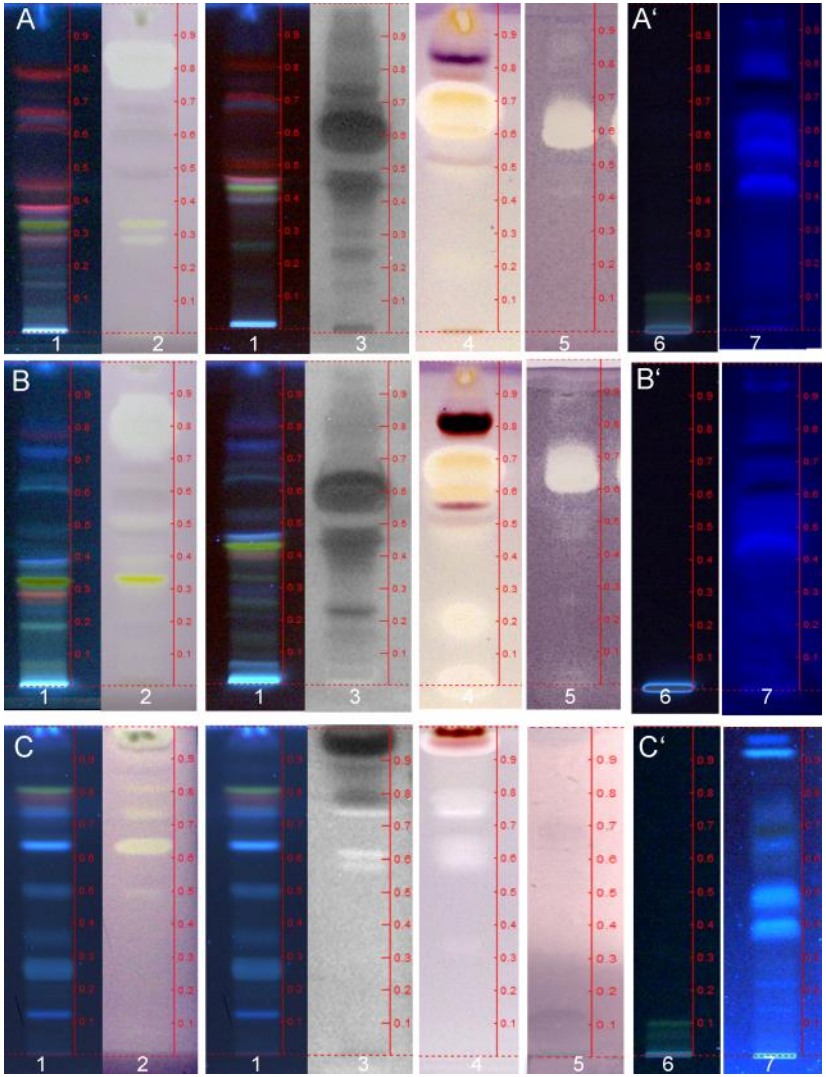
EDA of *Salicaceae* bud extract samples





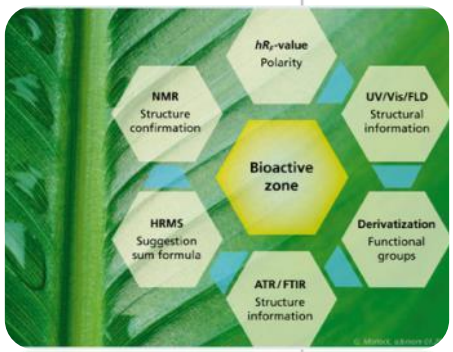
S. Hage *et al.*, in preparation

EDA of oilseed extracts



S.-S.Teh, G. Morlock, *Food Chem* in revision

Effect-directed link to the compound

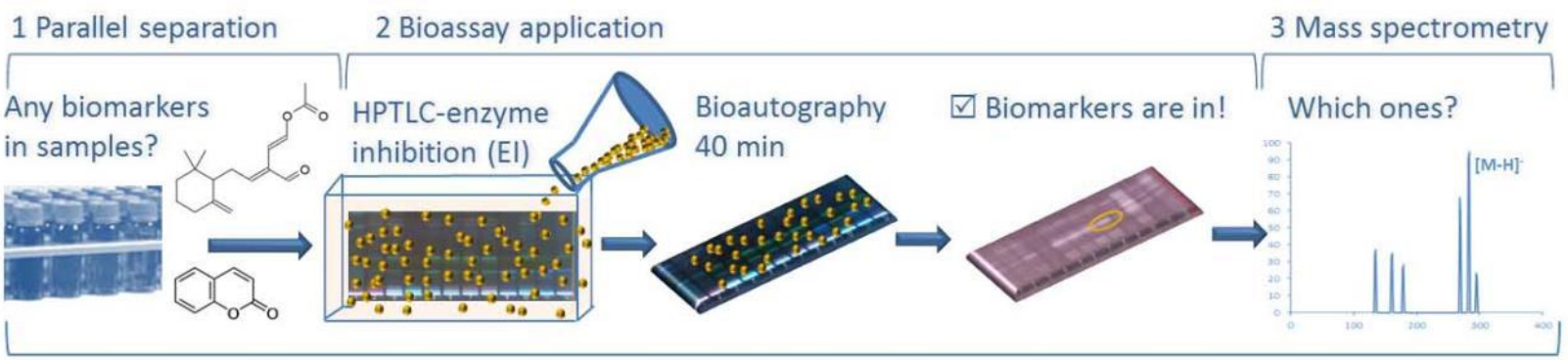


TLC/HPTLC-EDA

- Microchemical derivatization
  - DPPH* reagent - radical scavenging test
  - ABTS reagent - radical scavenging test
  - Bleaching of ß-carotene - antioxidant test
- Microbiological detection
  - Algae
    - *Pseudokirchneriella subcapitata*
  - Fungi
    - *Candida albicans*
    - *Trichophyton rubrum*
    - *Cryptococcus neoformans*
    - *Cladosporium cucmerinum*
    - *Apergillus niger*
    - *Colletotrichum acutatum/gloeosporioides/fragaria*
  - Bacteria
    - *Pseudomonas savastanoi*
    - *Bacillus subtilis*
    - *Aliivibrio fischeri*
    - *Escherichia coli*
    - *Pseudomonas syringae*
    - *Xanthomonas campestris*
    - *Erwinia carotovora/herbicola*
    - *Cryptococcus neoformans*
  - Yeast
    - Planar yeast estrogen screen (pYES)
- Biochemical assays
  - (Cholin)esterase assay
  - α/ß-Glucosidase assay
  - Xanthine oxidase assay
  - Tyrosinase oxidase assay

G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

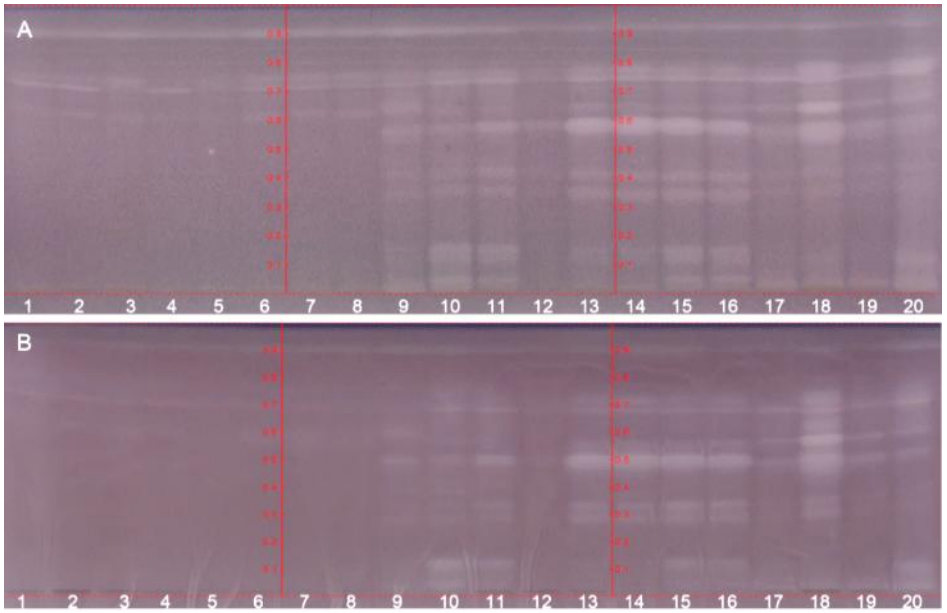
Detection of cholinesterase inhibitors



Rosaceae

AChE

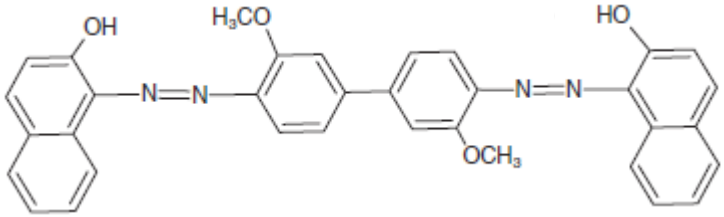
BChE



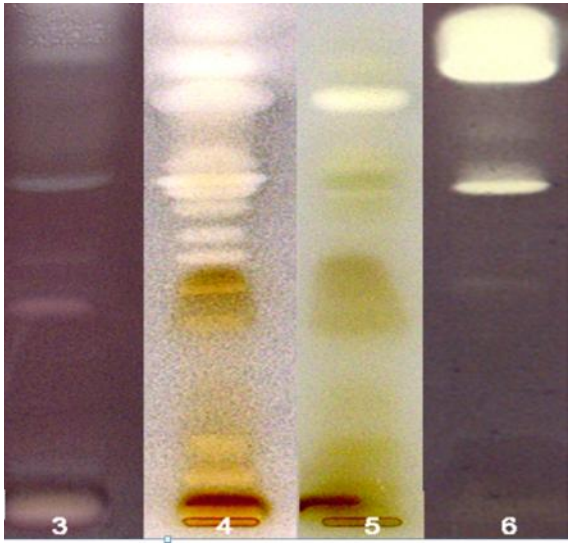
S. Hage *et al.*, in preparation

Detection of α-/β-glucosidase inhibitors

→ Immersion in α-/β-glucosidase solution and incubation
→ Immersion in 2-naphthyl-α/ß-D-glucopyranoside and Fast Blue Salt as substrate for visualization (5 min)
→ White zones on a violet background



Salvia officinalis

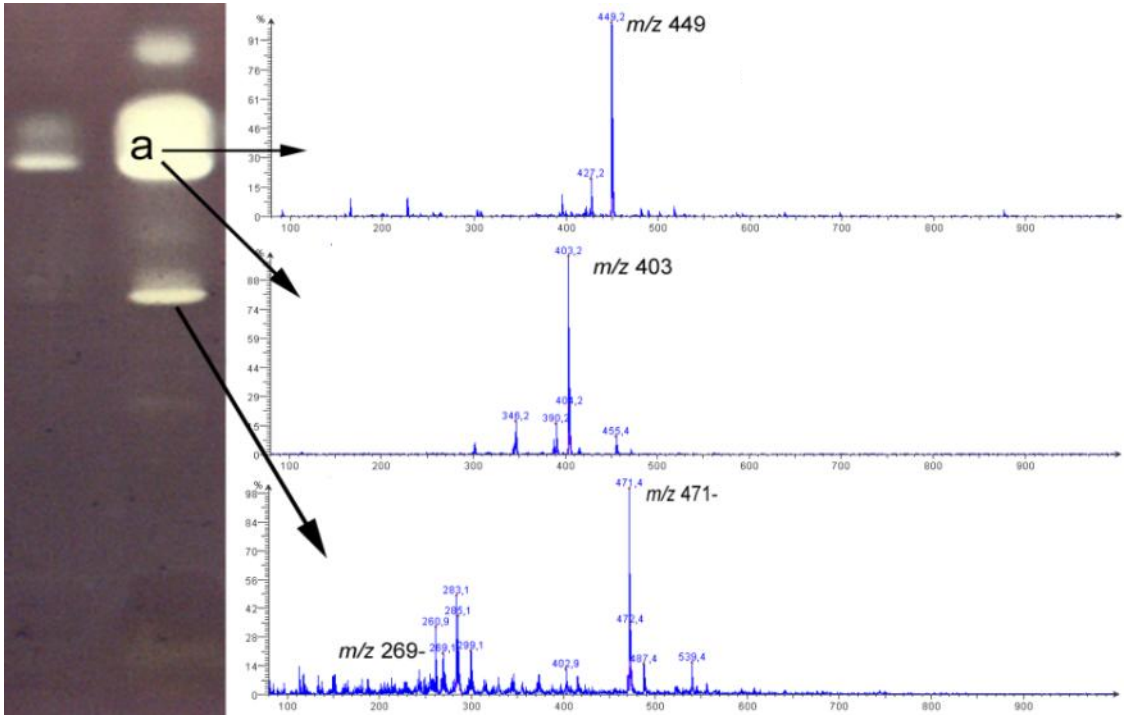


UV366 nm AChE α-/β-glucosidase *B. subtilis*

M. Jamshidi *et al.*, in preparation

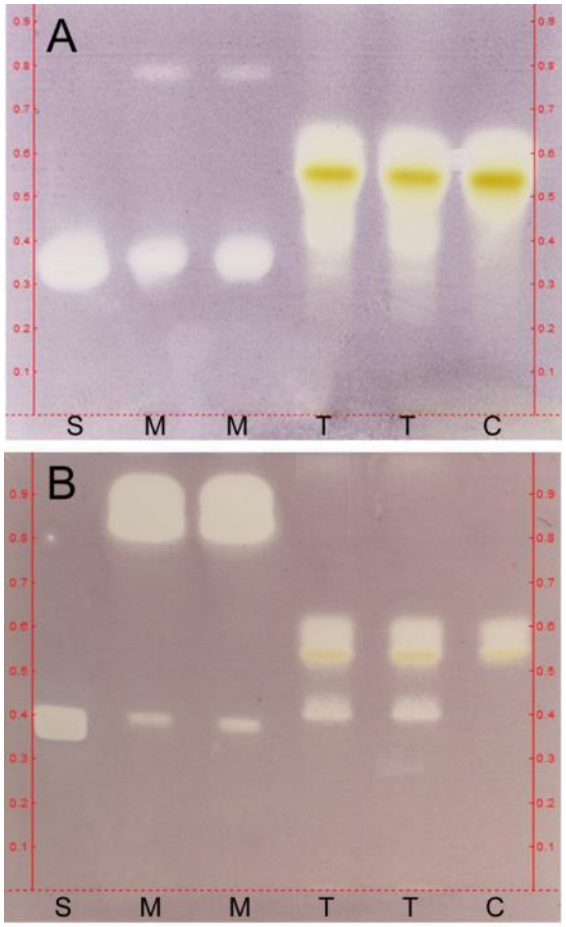
Detection of antibiotics

→ Immersion in *Bacillus subtilis* bacteria suspension and incubation
→ Immersion in tetrazolium salt as substrate for visualization
→ White zones on a pink background



M. Jamshidi *et al.*, in preparation

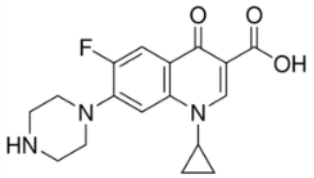
Current *vs*. optimized detection of antibiotics



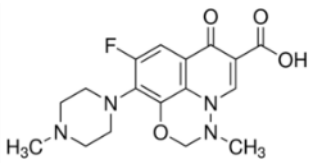
Detection of antibiotics

Antibiotics in milk down to the µg/kg range

Ciprofloxacin

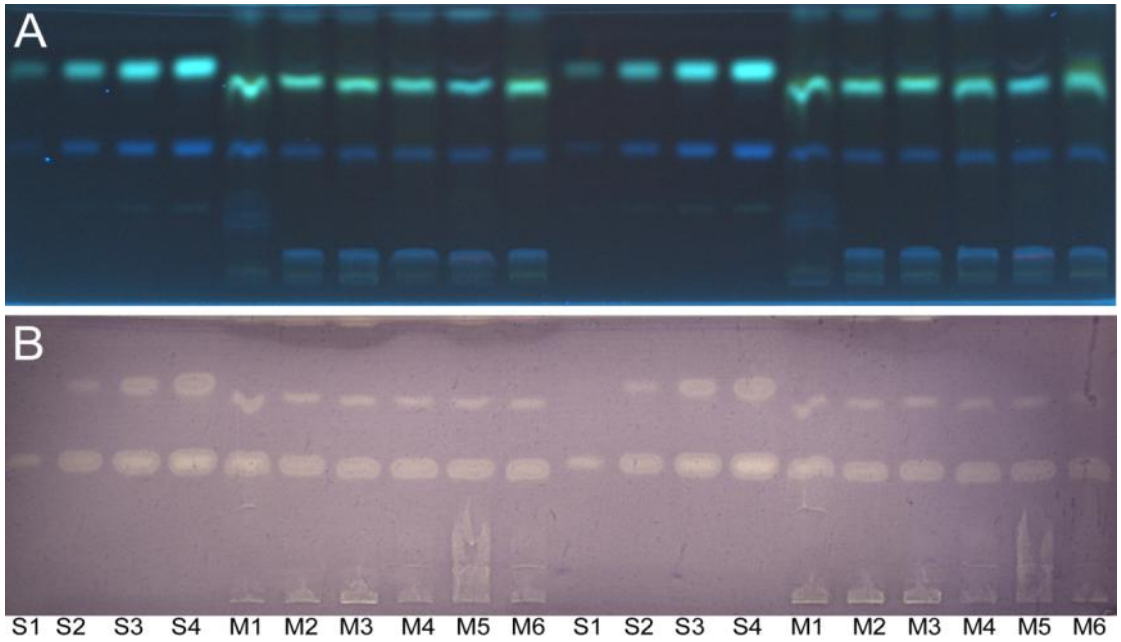


Marbofloxacin



UV 366 nm

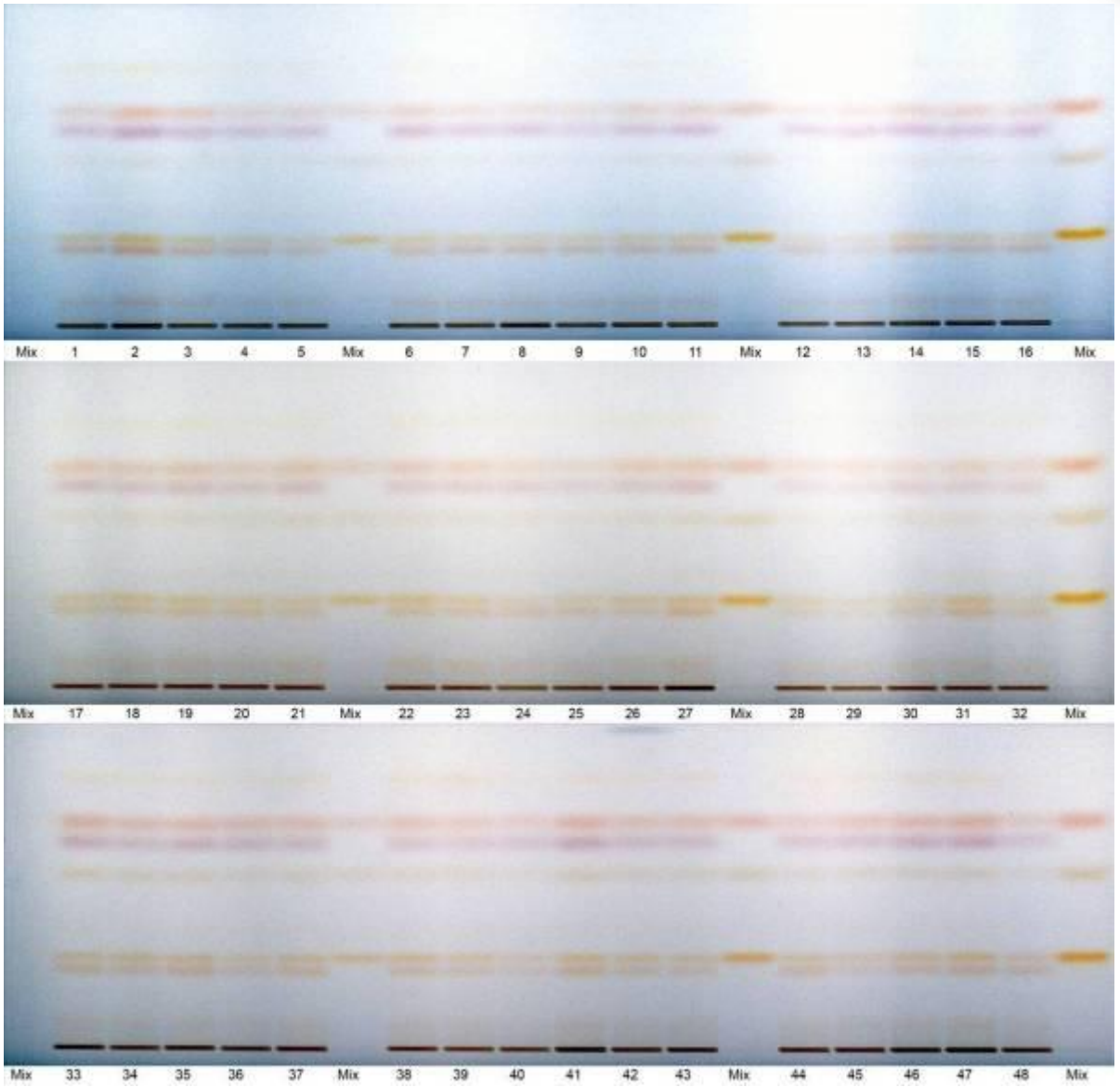
Bacillus subtilis



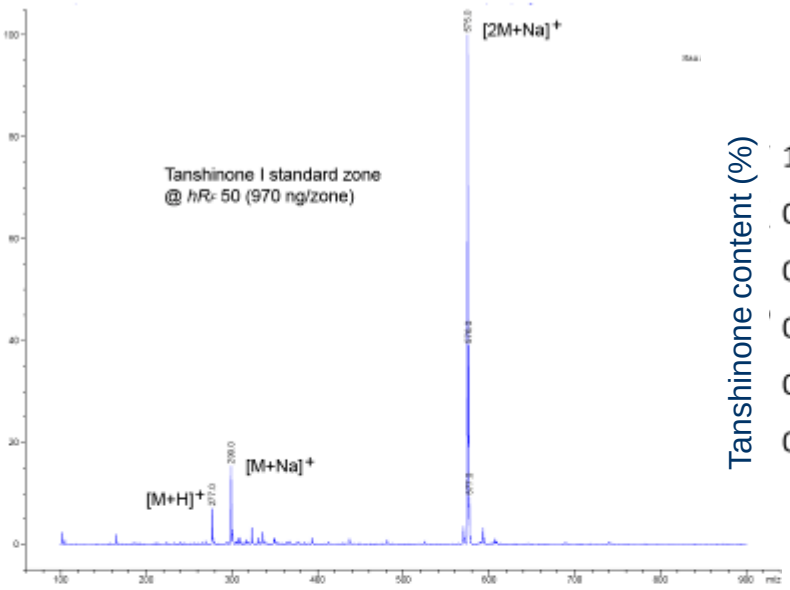
A. Cardak *et al.*, in preparation

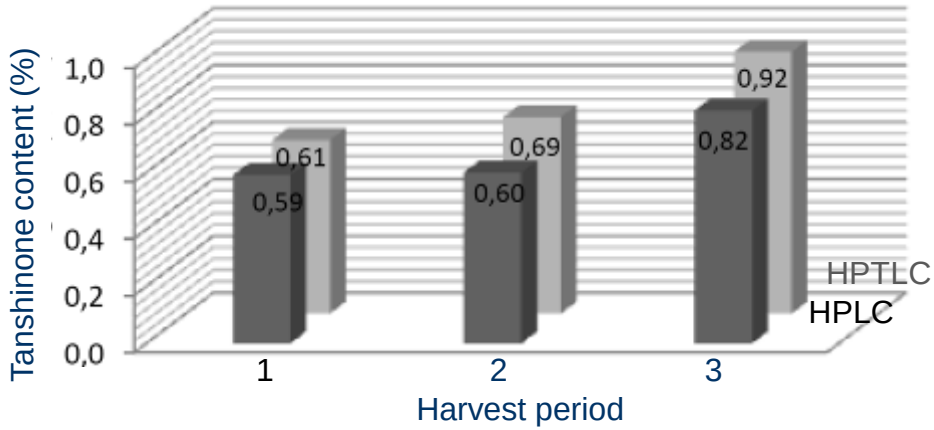
Quantitation of tanshinons in Chinese salvia

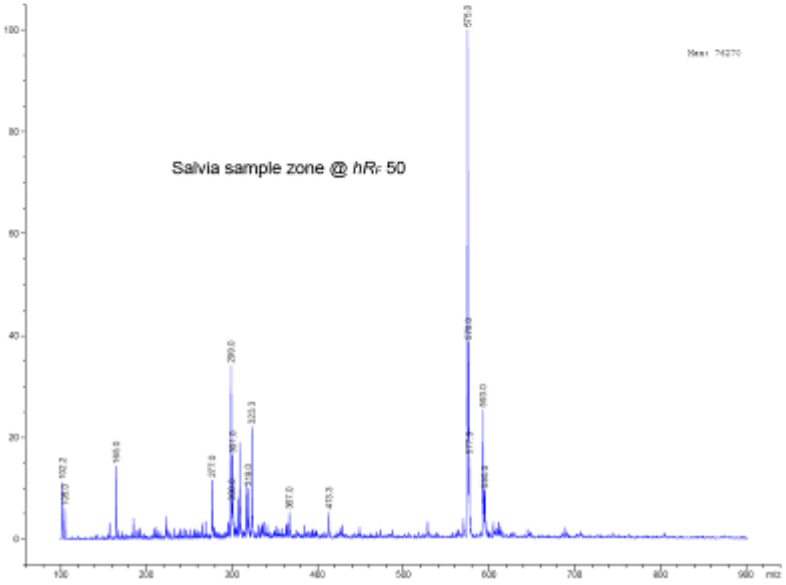
G. Morlock Food Science JLU Gießen

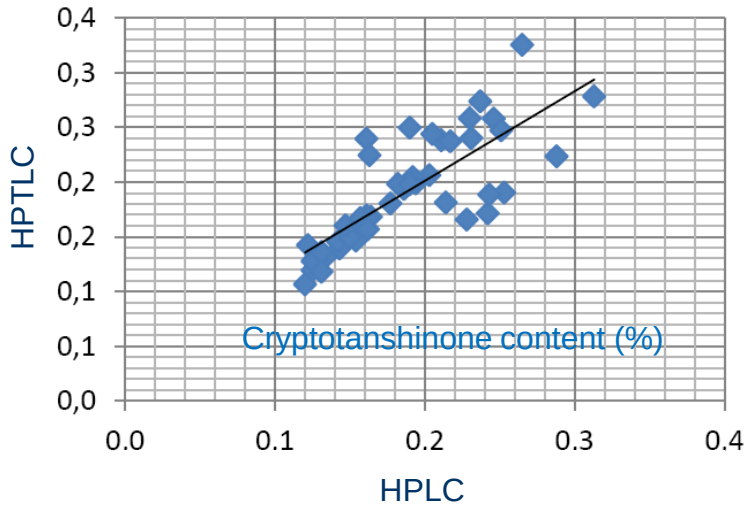


Confirmation by MS and method comparison

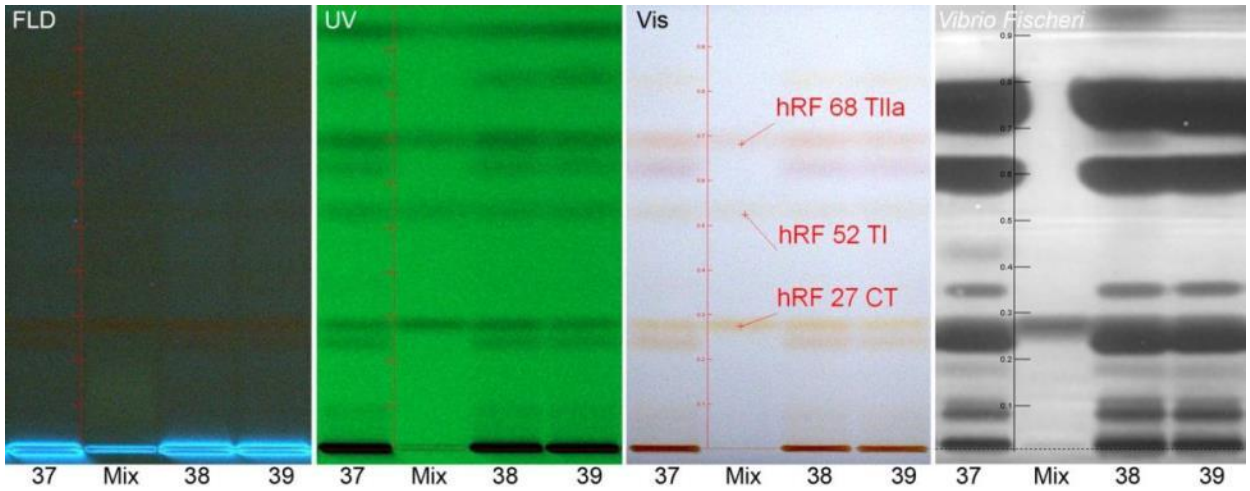






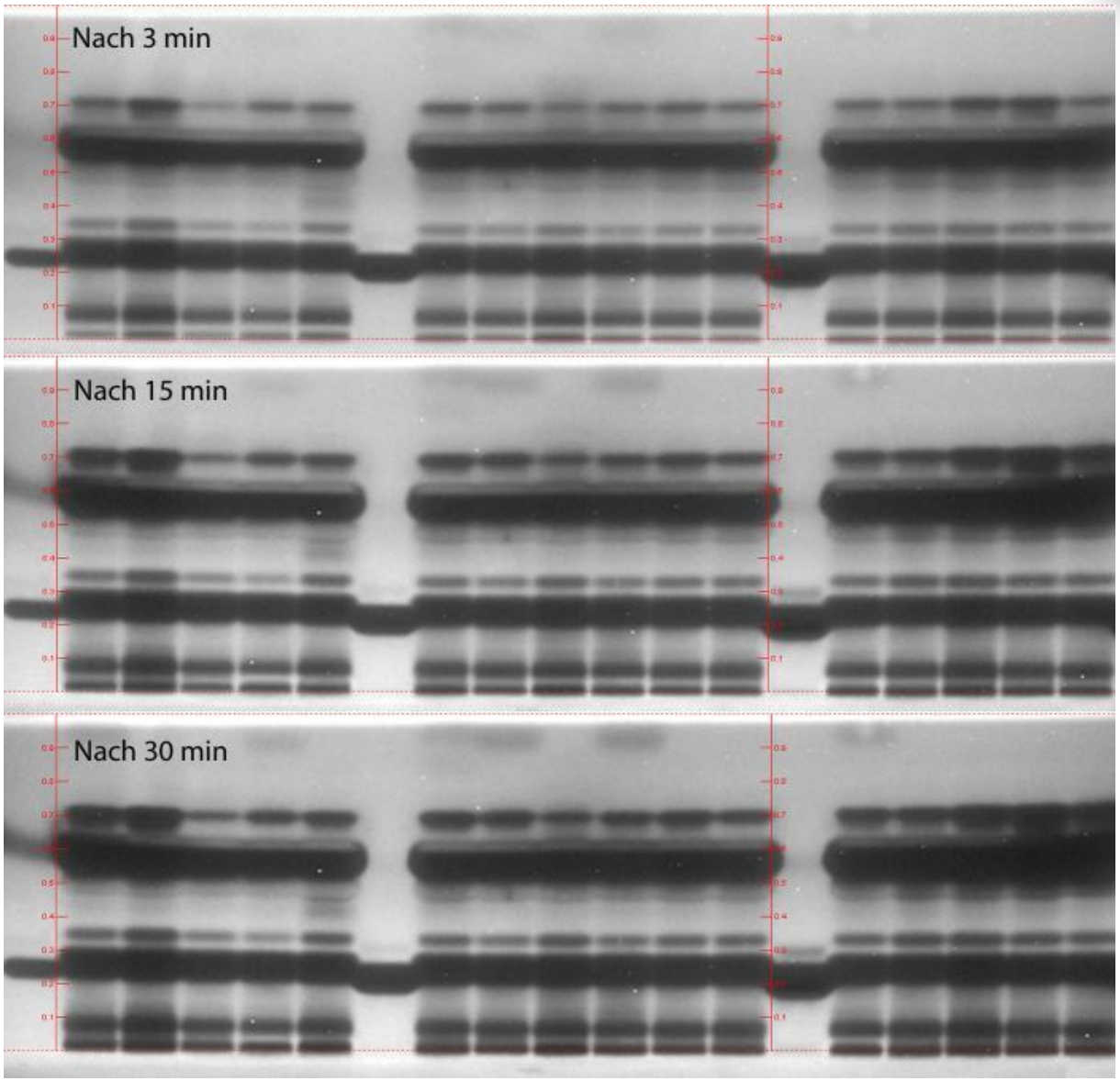


Detection of bioactive compounds



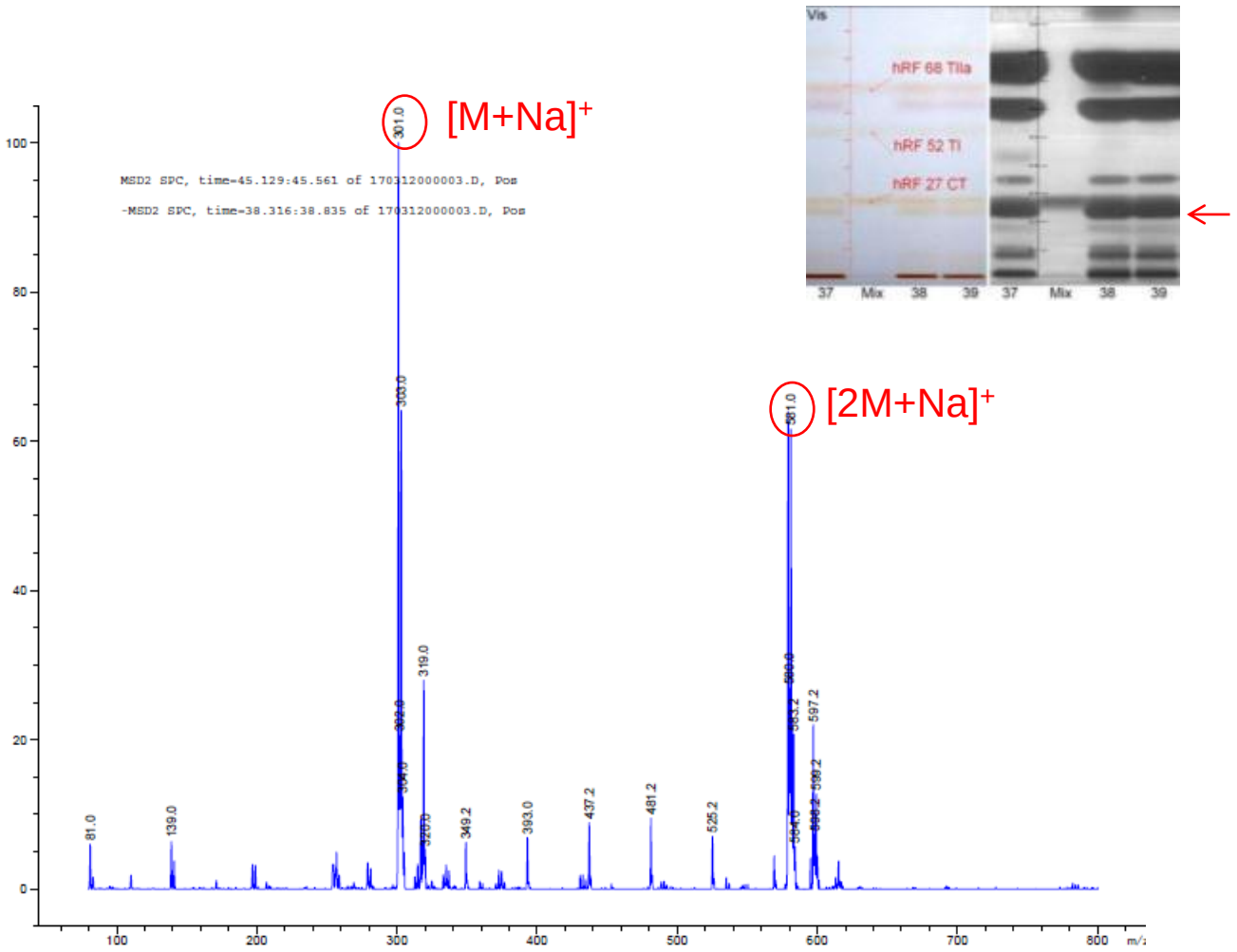
G. Morlock *et al.*, in preparation

Detection of bioactive compounds



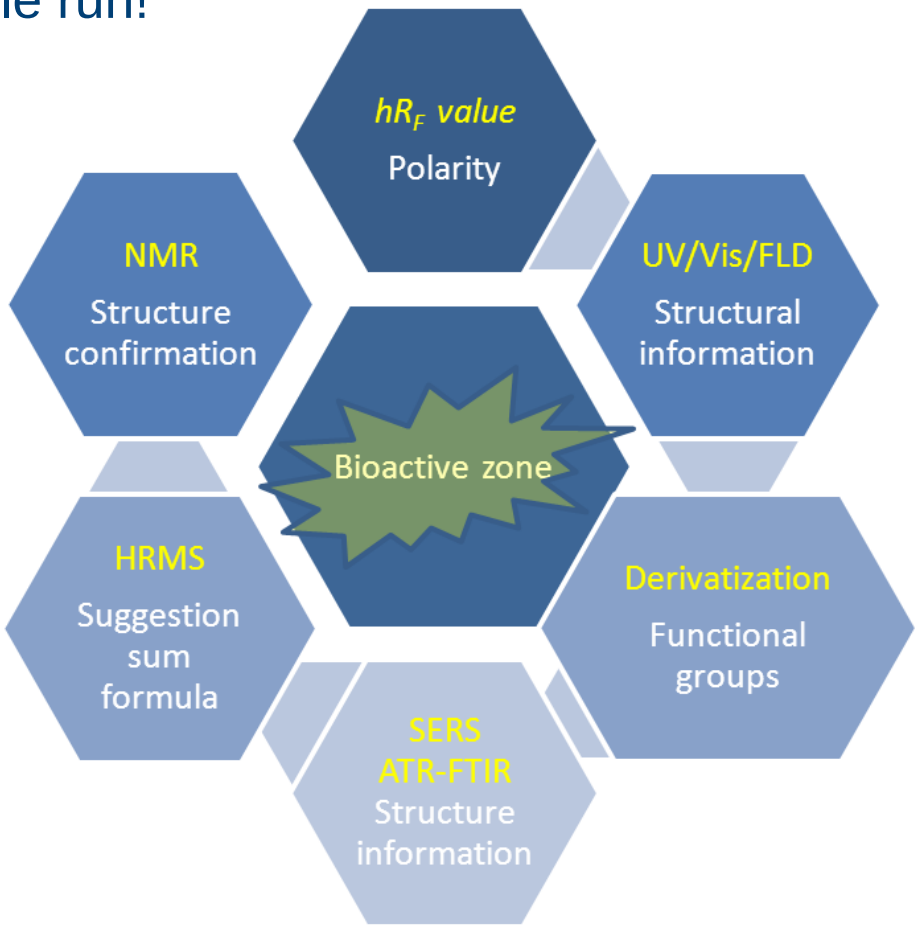
Detection of bioactive compounds

Mass spectra recorded after detection with bioassay → salt adducts are pronounced!



Goal: From bioactive zone to sum formula

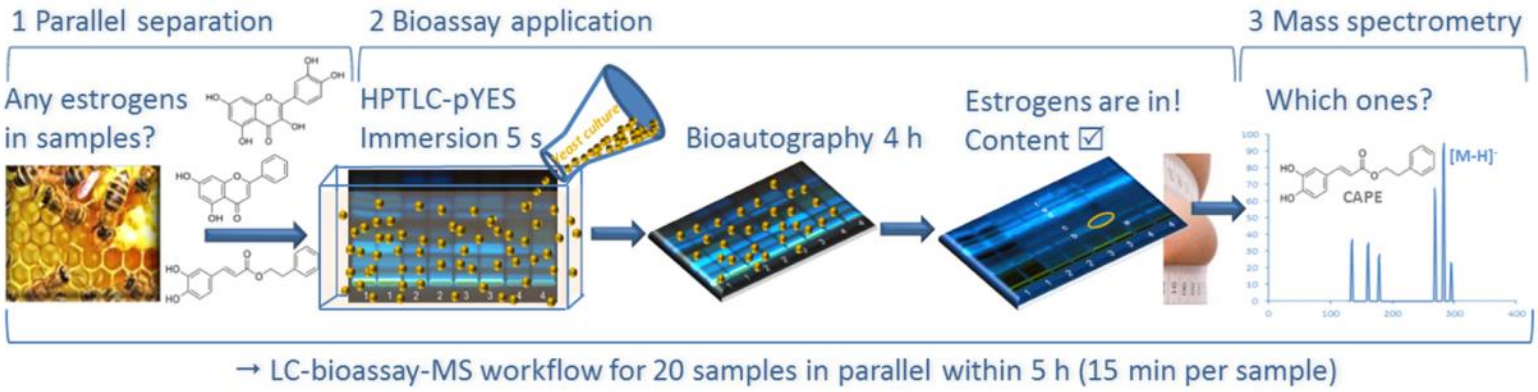
…in a single run!



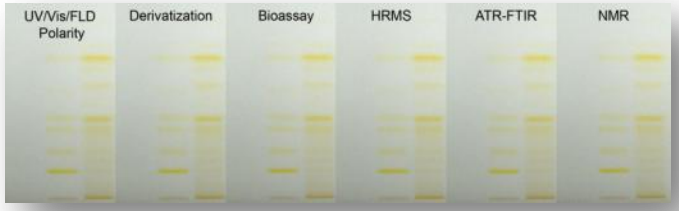
G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

Hyphenated technique

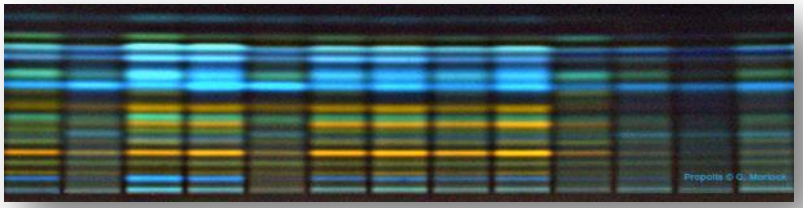
HPTLC-UV/Vis/FLD-bioassay-HRMS/ATR-FTIR/NMR



…in a single run

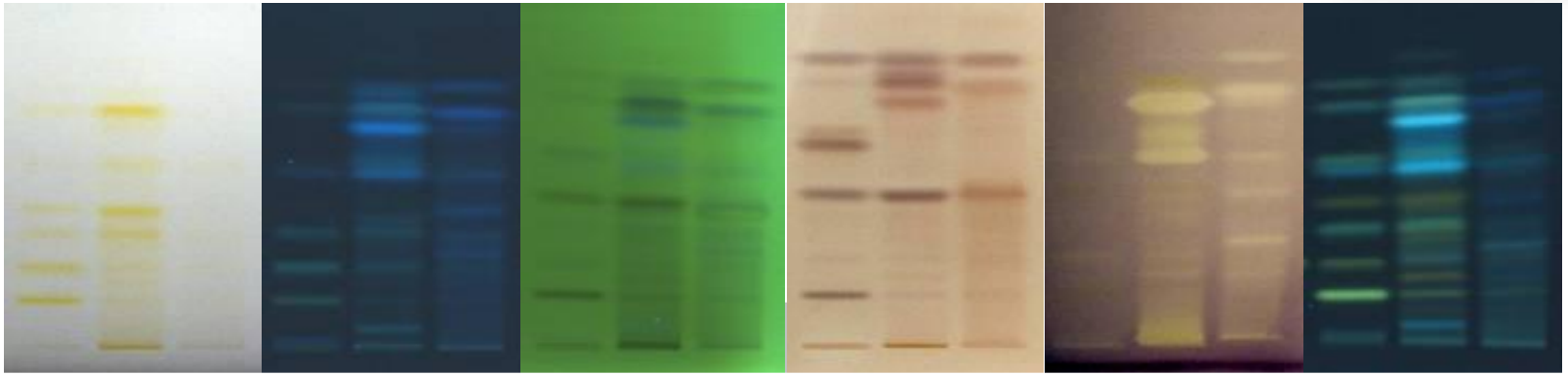


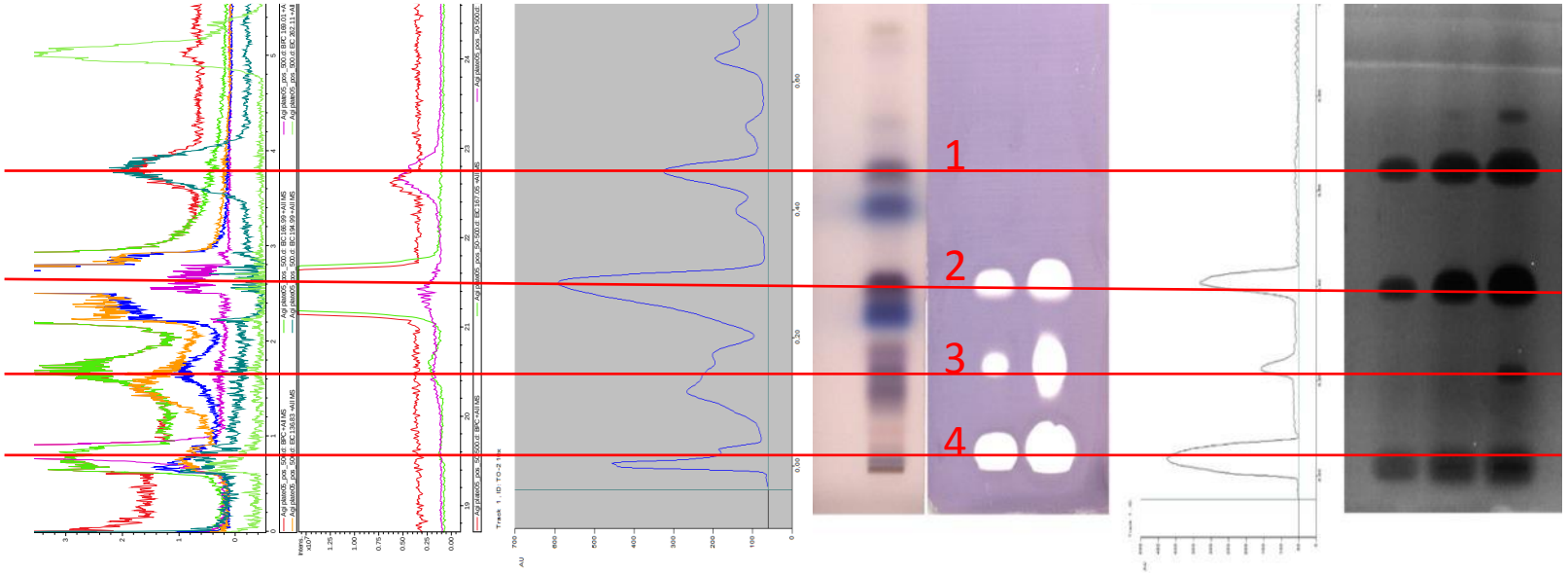
….or for screening



G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

Multi-detection

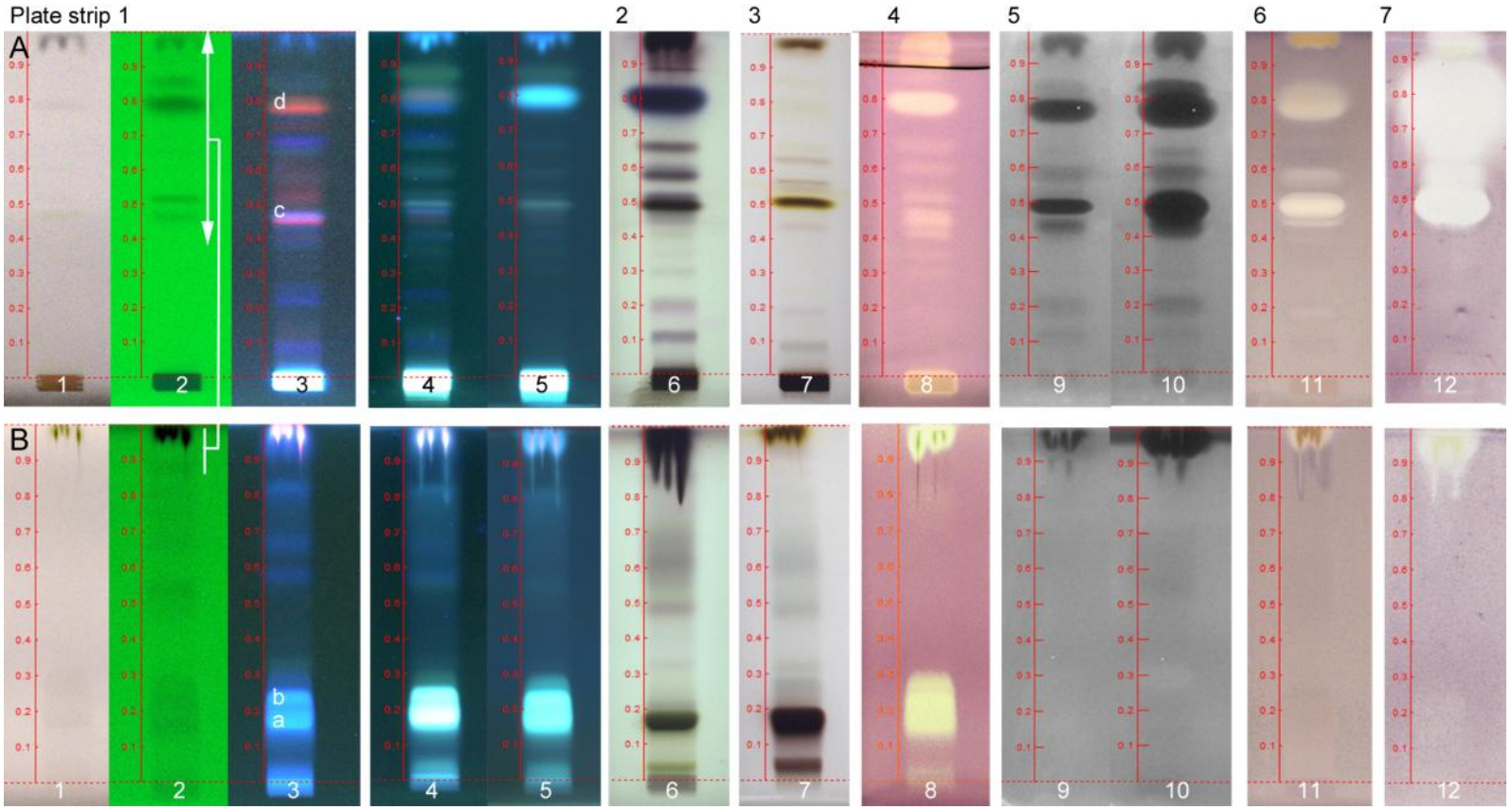




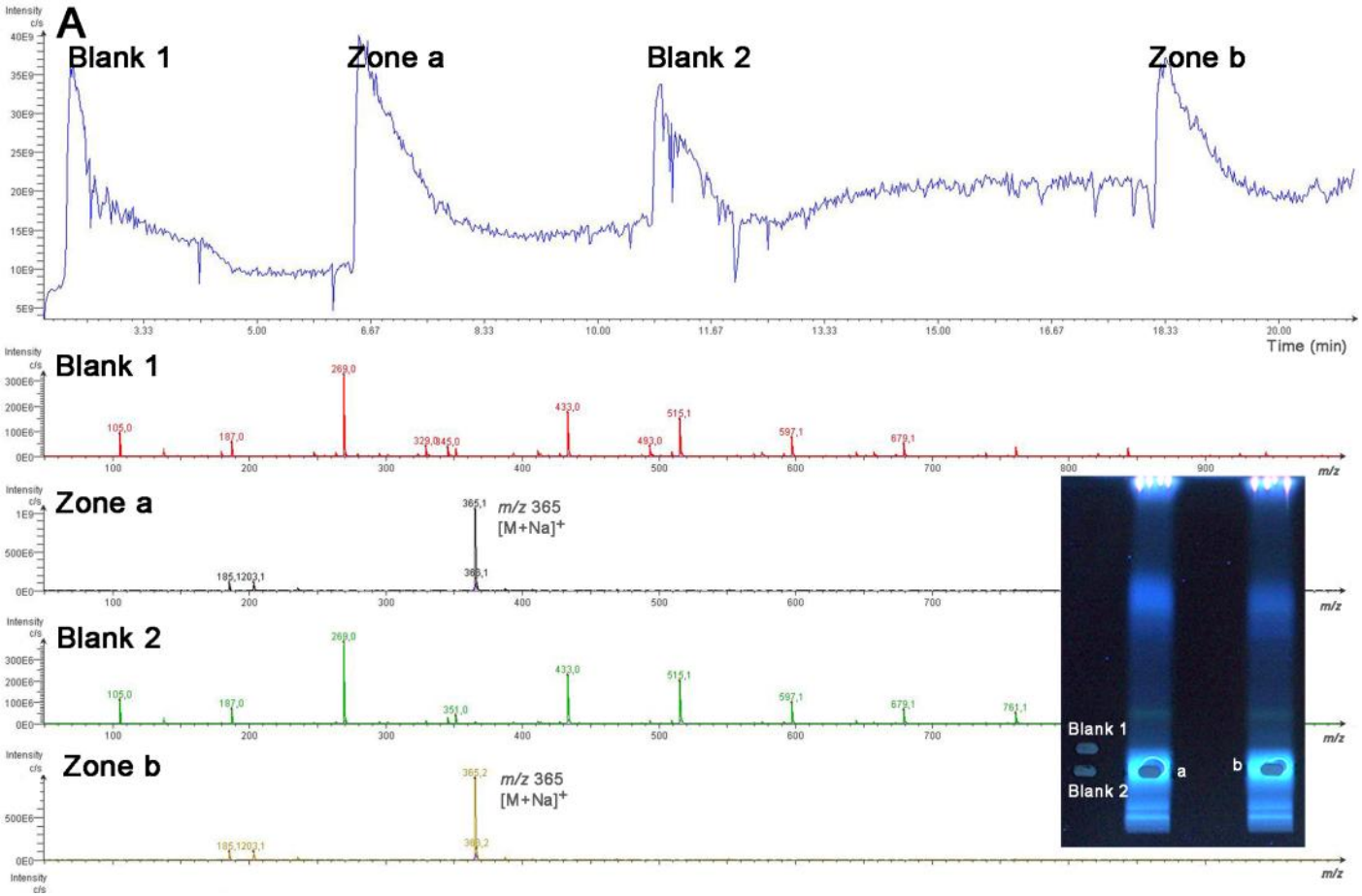
G. Morlock *et al.*, in preparation
A. Moricz *et al.*, in preparation

G. Morlock Food Science JLU Gießen

EDA of *Pimpinella saxifraga* L. root extracts



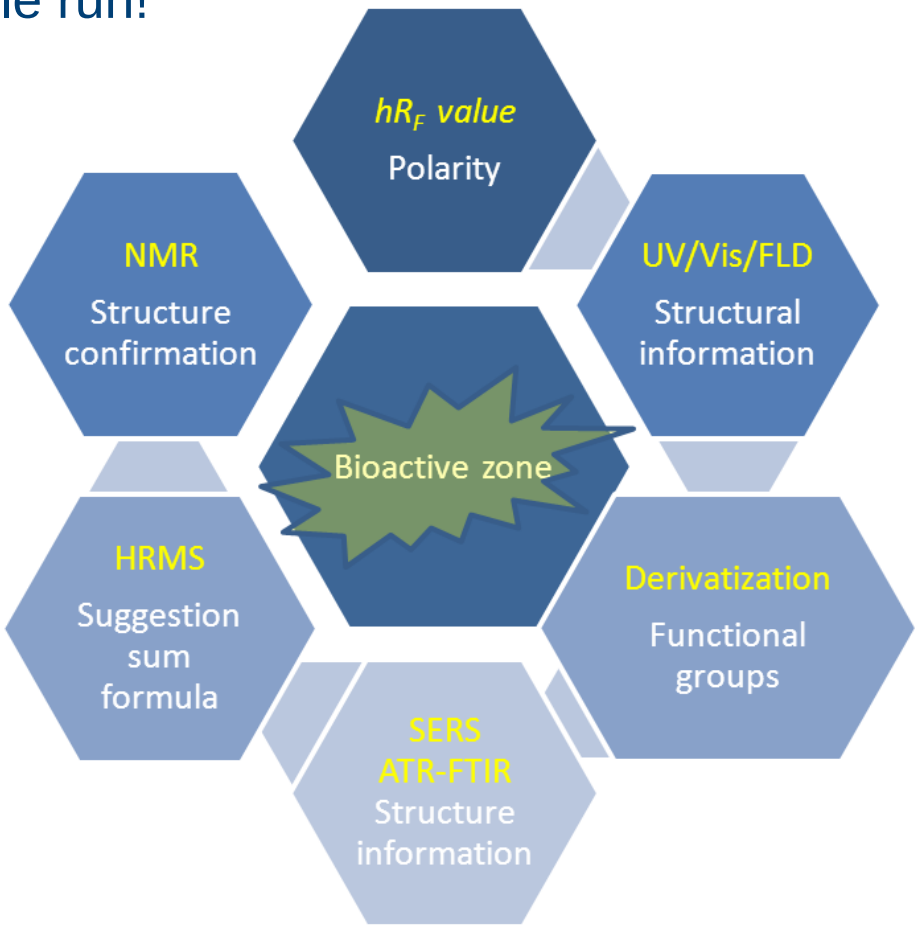
EDA of *Pimpinella saxifraga* L. root extracts



T. Lapin, G. Morlock, in preparation

Goal: From bioactive zone to sum formula

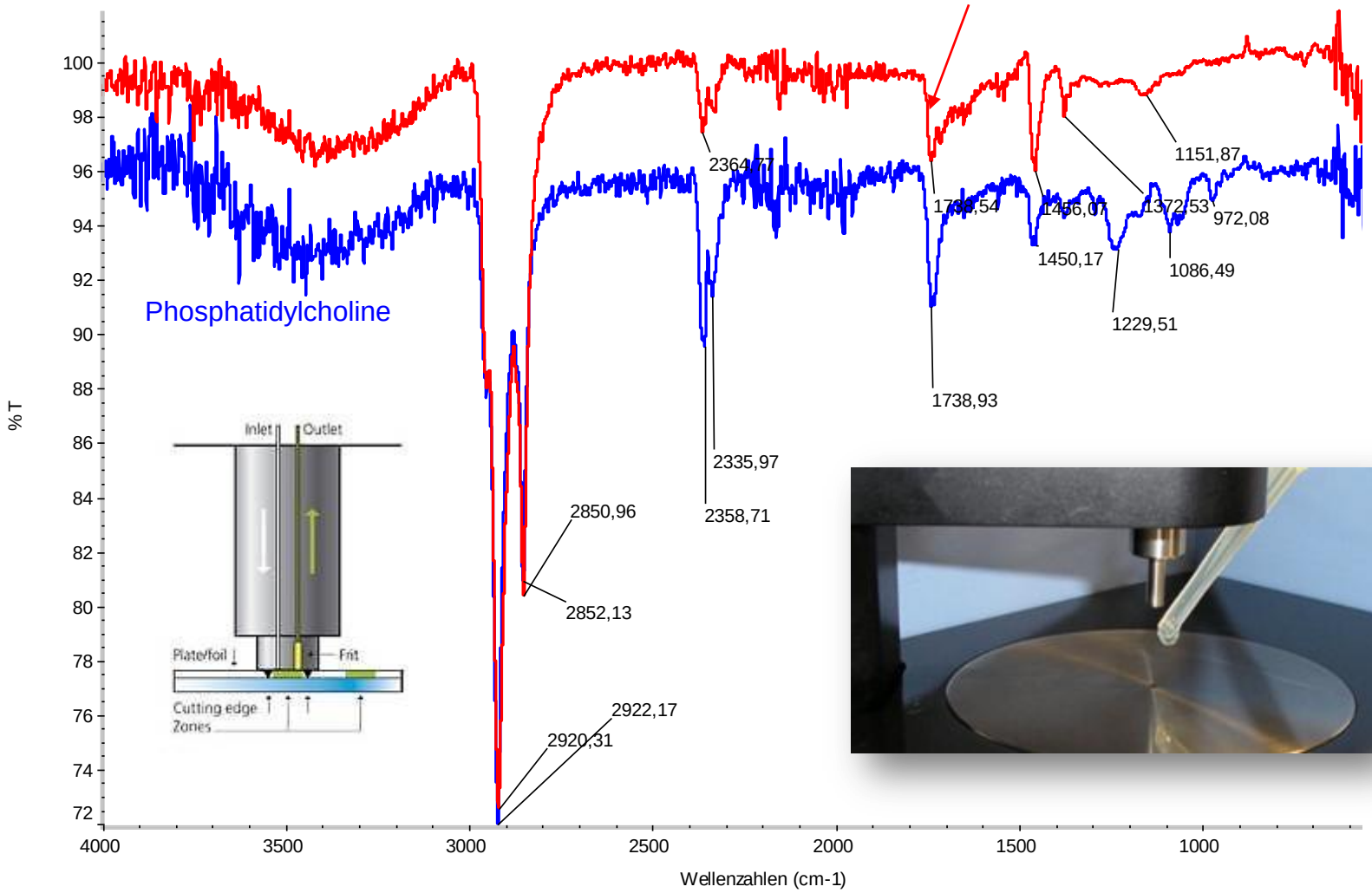
…in a single run!



G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

HPTLC-ATR FTIR

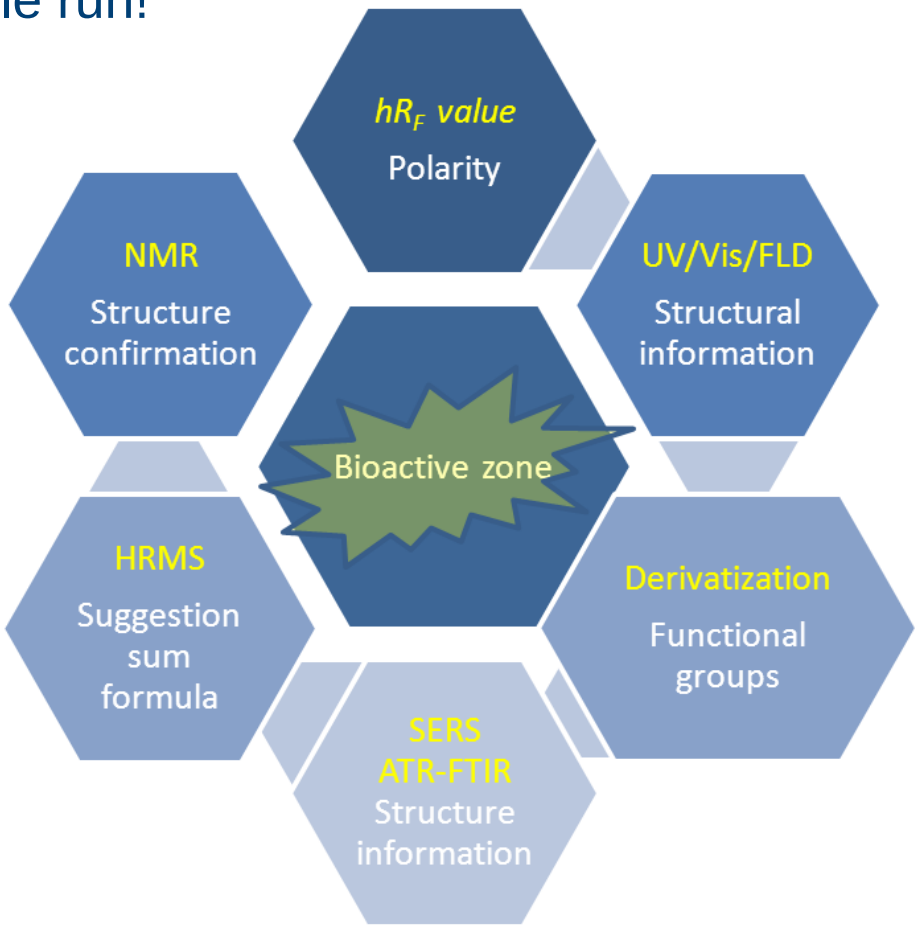
Anti-inflammatoric compound isolated from *Lactobacillus fermentum*



E. Dytkiewitz, G. Morlock *J AOAC Int* 91 (2008) 1237-1244
G. Morlock *et al.,* in preparation

Goal: From bioactive zone to sum formula

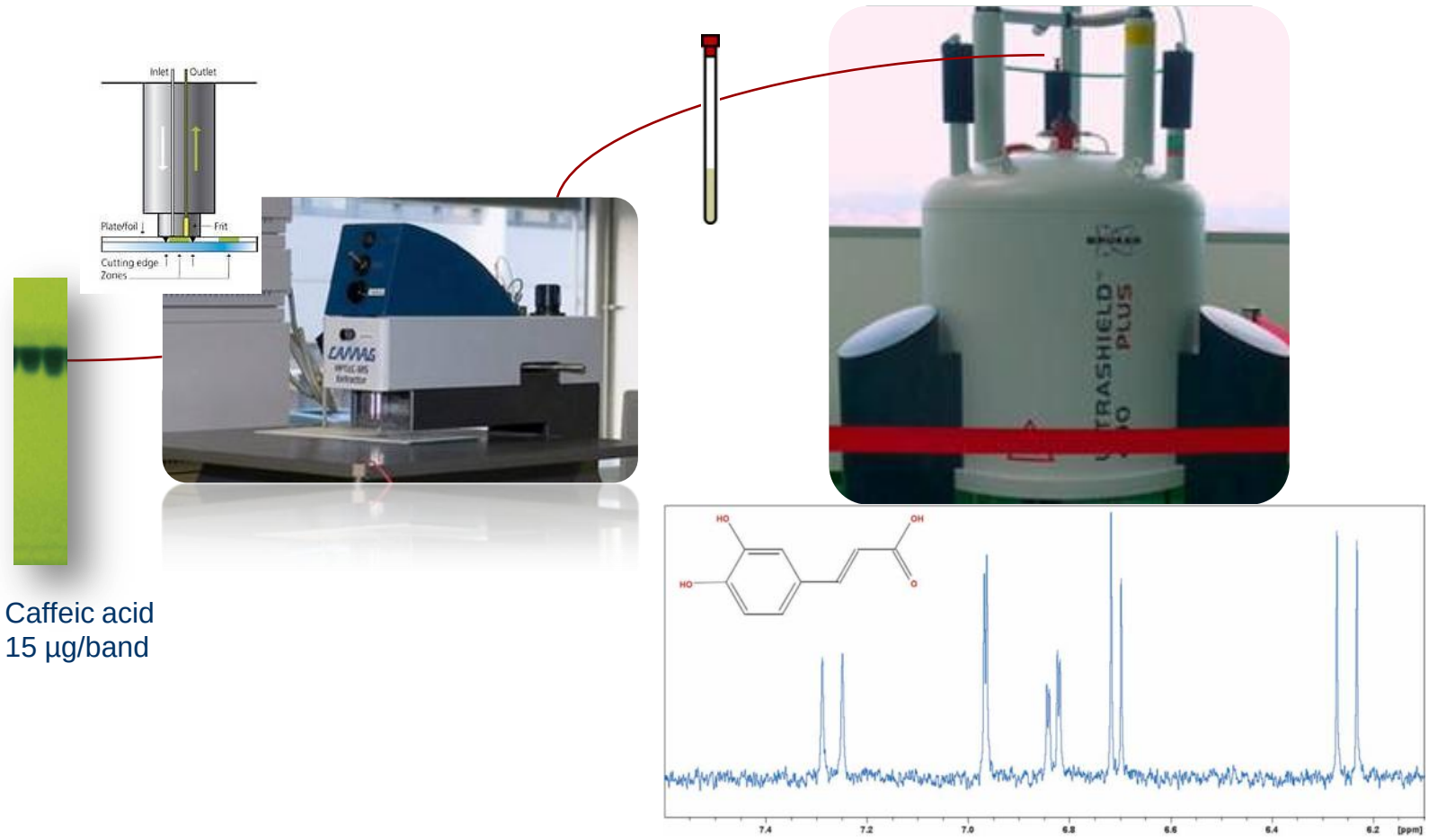
…in a single run!



G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

HPTLC-NMR

→ Hyphenation of HPTLC with ^{1}H-NMR via TLC-MS Interface

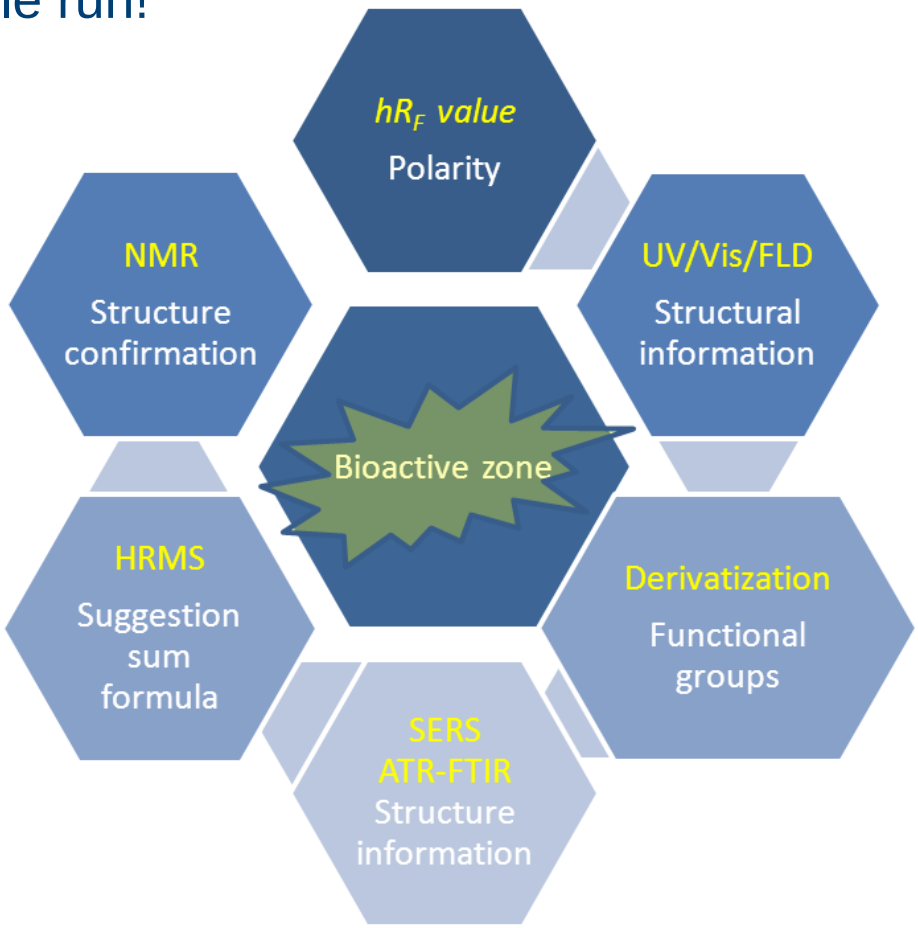


Caffeic acid
15 µg/band

G. Morlock, Seminar HPTLC-MS, Fachhochschule Nordwestschweiz, Basel, 23.06.2010
→ → → Gössi, U. Scherer, G. Schlotterbeck, *Chimia* 66 (2012) 347

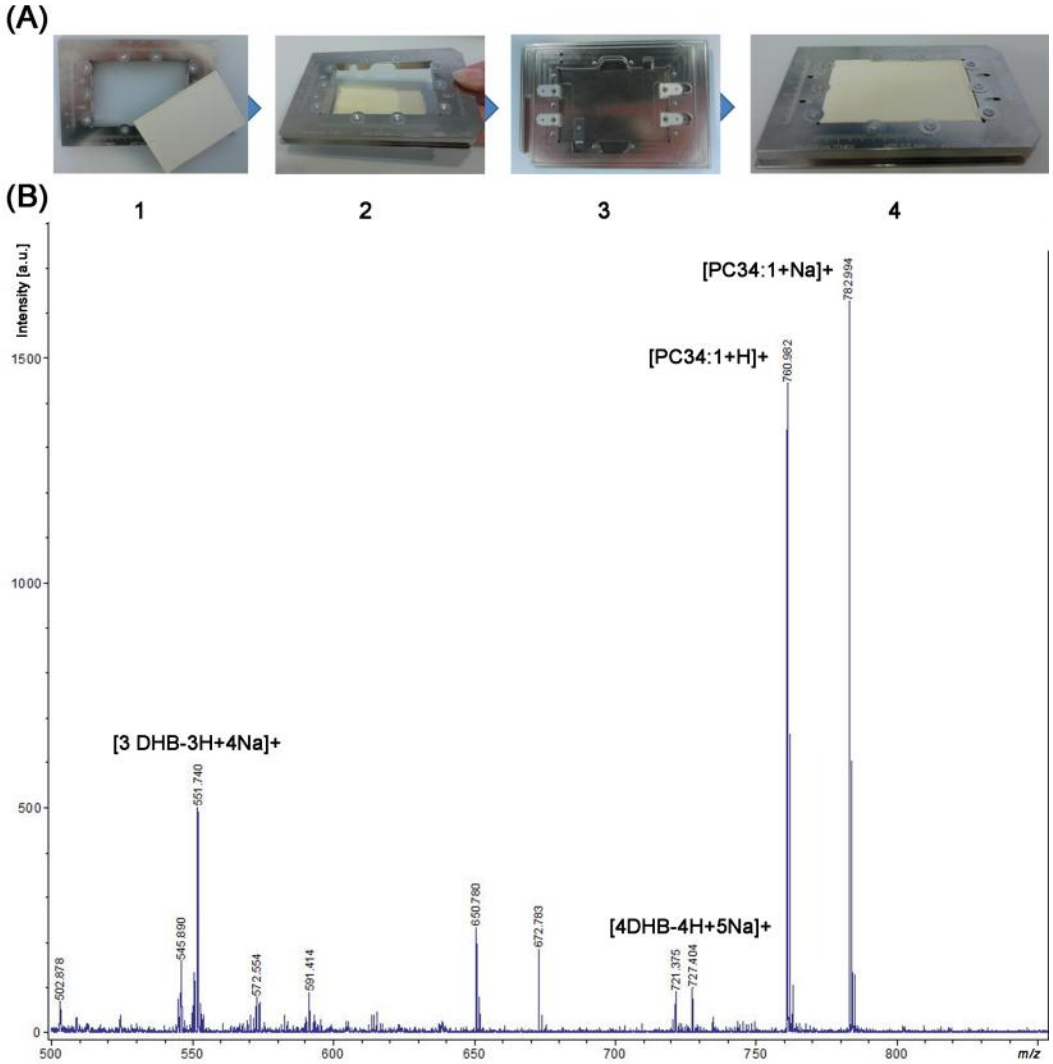
Goal: From bioactive zone to sum formula

…in a single run!



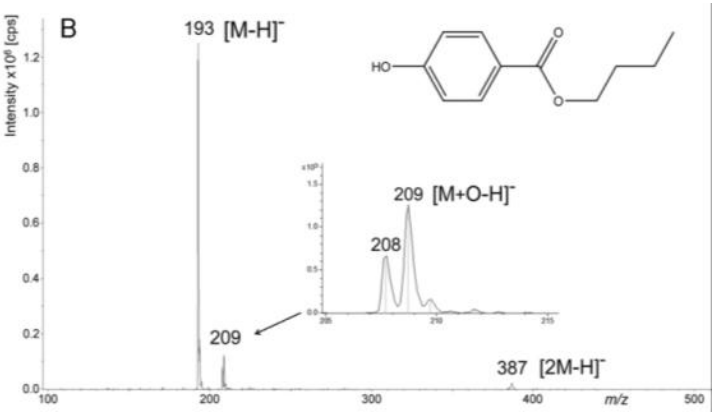
G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

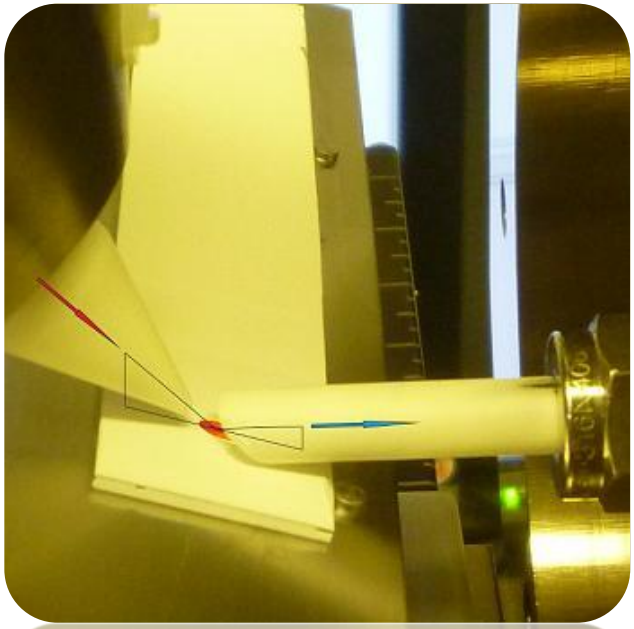
HPTLC-MALDI-TOF MS of phospholipids

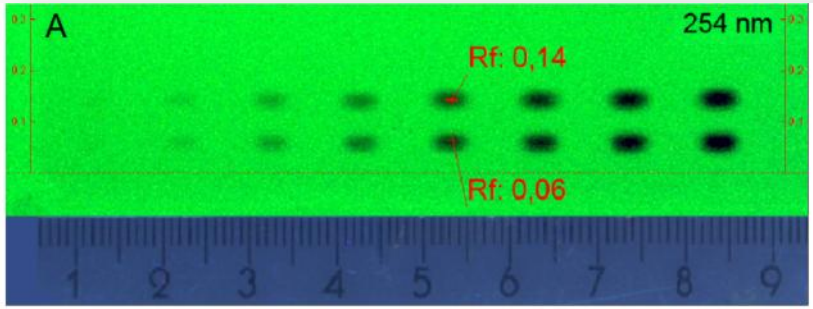


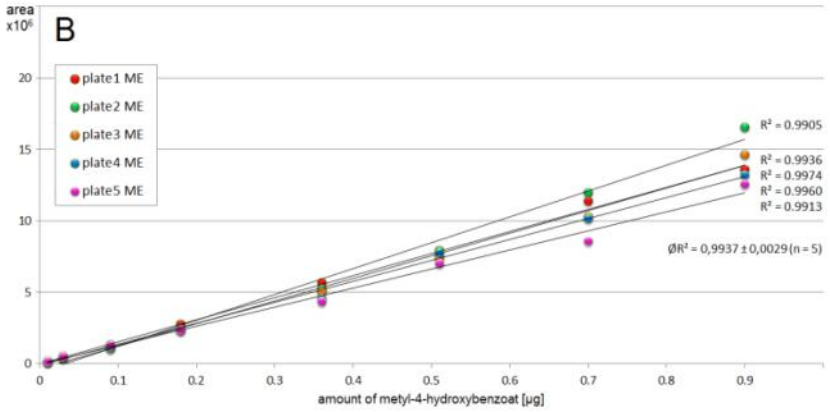
S. Krüger, L. Bürmann, G. Morlock, *J Agric Food Chem,* in print

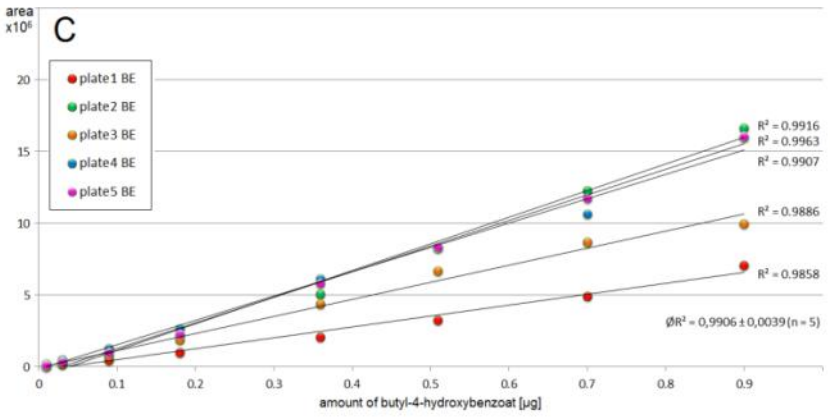
HPTLC-DART-SVPA-MS of preservatives





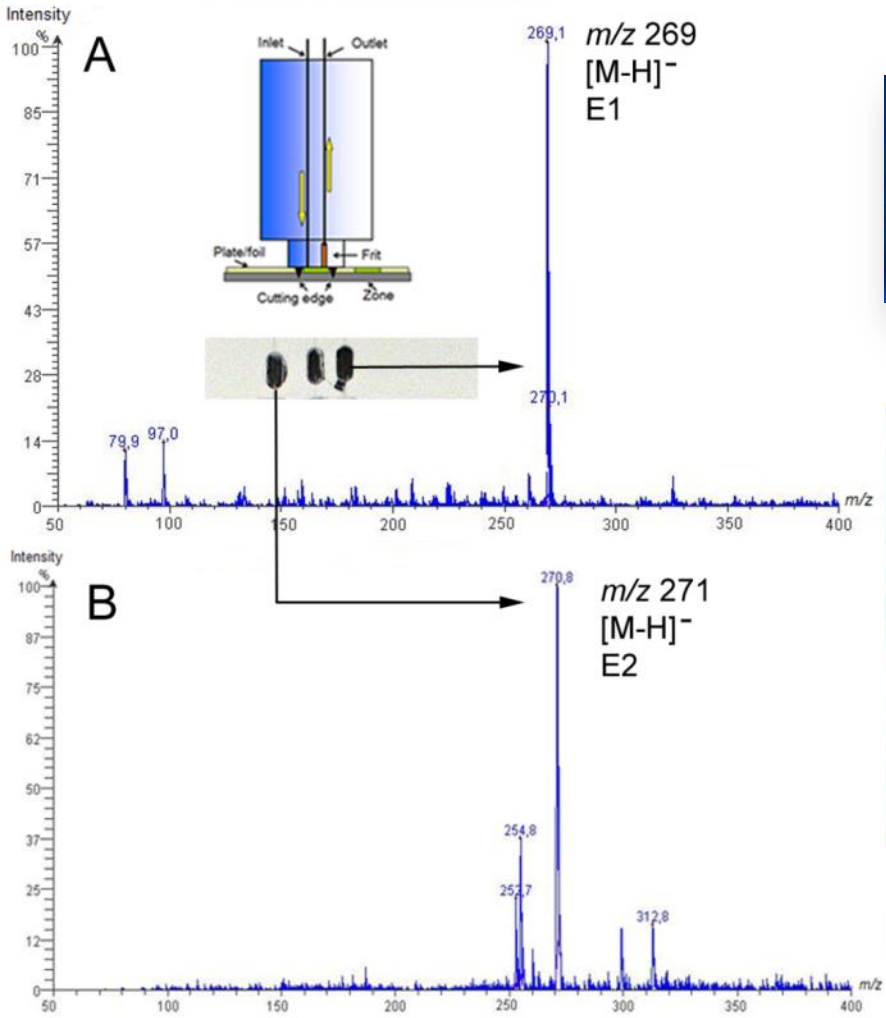


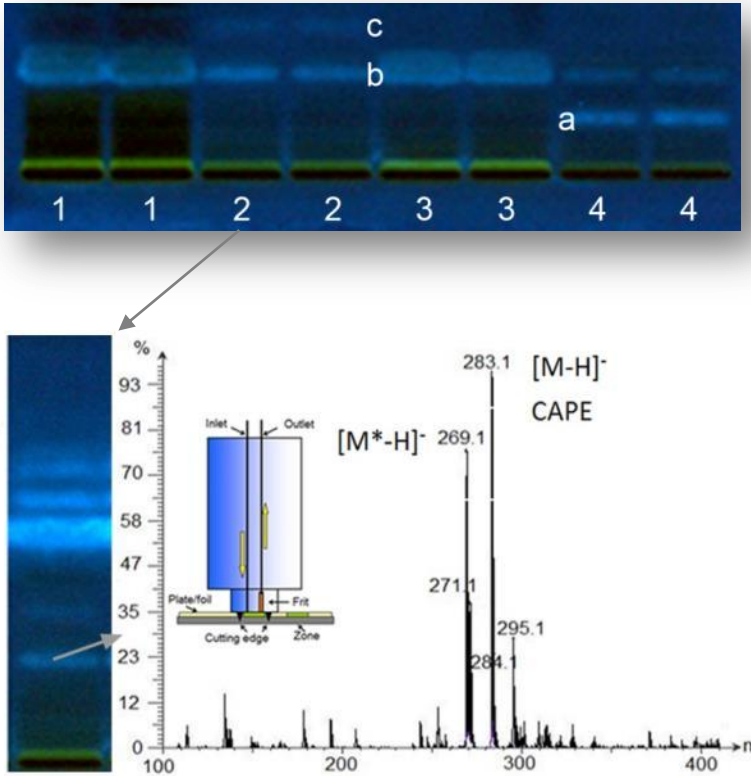




T. Häbe, G. Morlock, Rapid Commun Mass Spectrom 29 (2015) 474–484

Confirmation by MS

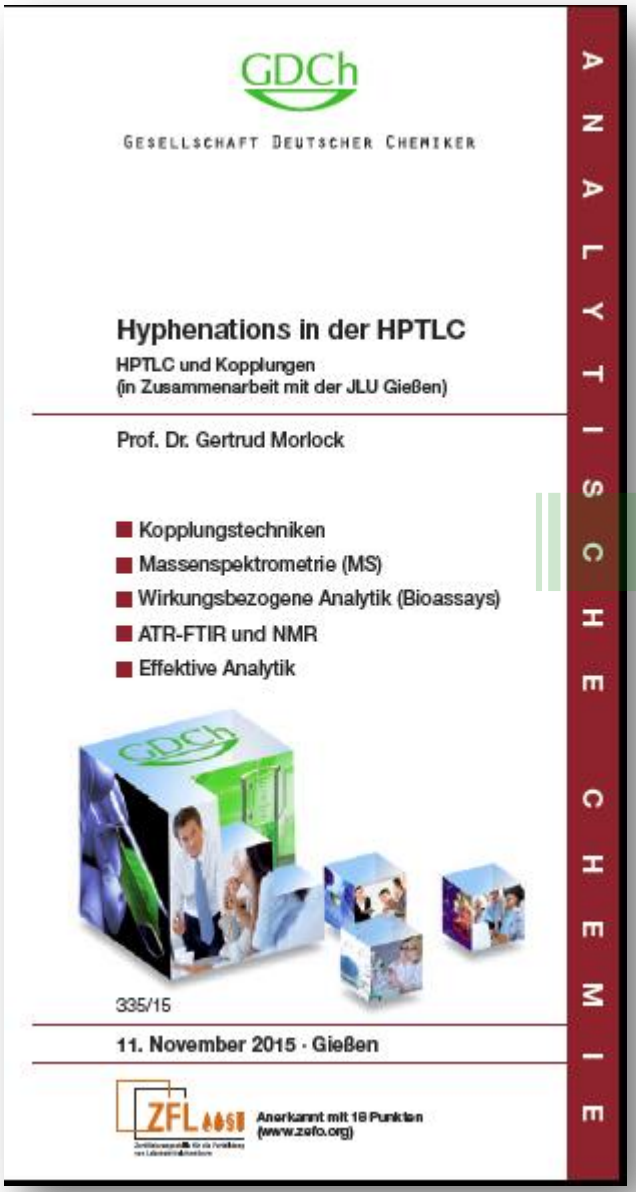




I. Klingelhöfer, G. Morlock, *J. Chromatogr. A* 1360 (2014) 288-295

G. Morlock, I. Klingelhöfer, *Anal. Chem*. 86 (2014) 8289–8295

GDCh course 335/15



PROGRAMM

Mittwoch, 11. November 2015

9.00 Begrüßung und Einführung in die HPTLC (Morlock)

9.45 HPTLC erfahren – Experimente (Häbe, Klingelhöfer)

10.45 Kaffeepause

11.00 Hyphenations in der Planar-Chromatographie – Teil 1 (Morlock, Schwack)

11.45 Gruppe 1: Experiment DC-HPLC/DAD-ESI MS (Oellig, Schwack)
Gruppe 2: Experiment HPTLC-UV/Vis/FLD-MALDI-TOF MS/MS (Lochnit, Krüger)

12.30 Gruppe 1: Experiment HPTLC-UV/Vis/FLD-MALDI-TOF MS/MS (Lochnit, Krüger)
Gruppe 2: Experiment DC-HPLC/DAD-ESI MS (Oellig, Schwack)

13.15 Mittagspause

13.45 Hyphenations in der Planar-Chromatographie – Teil 2 (Morlock)

14.00 Gruppe 1: Experiment HPTLC-UV/Vis/FLD-ATR FTIR (Klingelhöfer, Gerbig)
Gruppe 2: Experiment HPTLC-UV/Vis/FLD-Bioassay-ESI MS (Krüger, Kirchert)

14.45 Gruppe 1: Experiment HPTLC-UV/Vis/FLD-Bioassay-ESI MS (Krüger, Kirchert)
Gruppe 2: Experiment HPTLC-UV/Vis/FLD-ATR FTIR (Klingelhöfer, Gerbig)

15.30 Kaffeepause

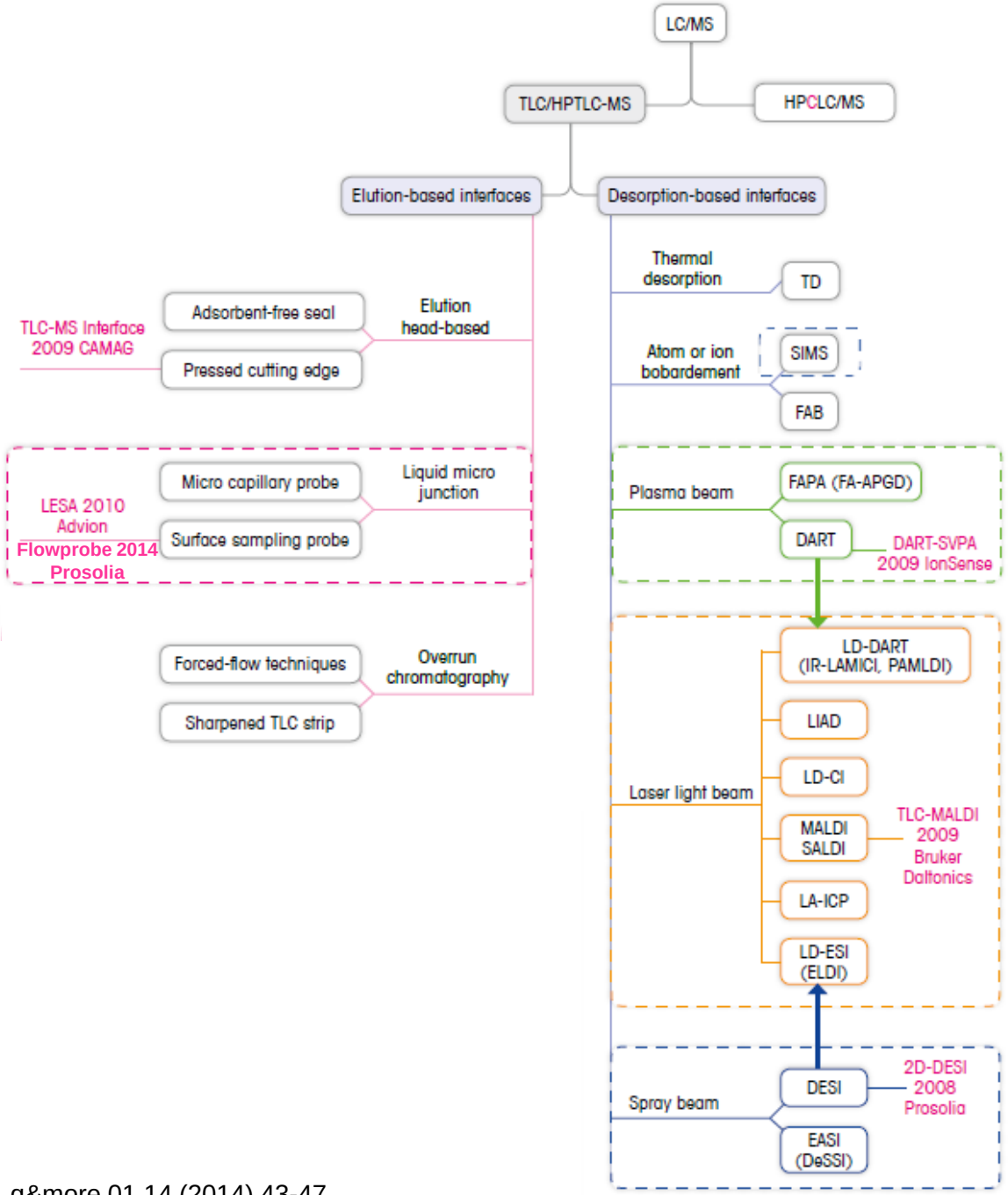
15.45 Hyphenations in der Planar-Chromatographie – Teil 3 (Morlock)

16.00 Gruppe 1: Experiment HPTLC-UV/Vis/FLD-DART-MS (Häbe, Krüger)
Gruppe 2: Experiment HPTLC-UV/Vis/FLD-DESI-MS (Kirchert, Stiefel)

16.15 Gruppe 1: Experiment HPTLC-UV/Vis/FLD-DESI-MS (Kirchert, Stiefel)
Gruppe 2: Experiment HPTLC-UV/Vis/FLD-DART-MS (Häbe, Krüger)

16.30 Diskussion (Morlock)

17.00 Voraussichtliches Ende der Veranstaltung

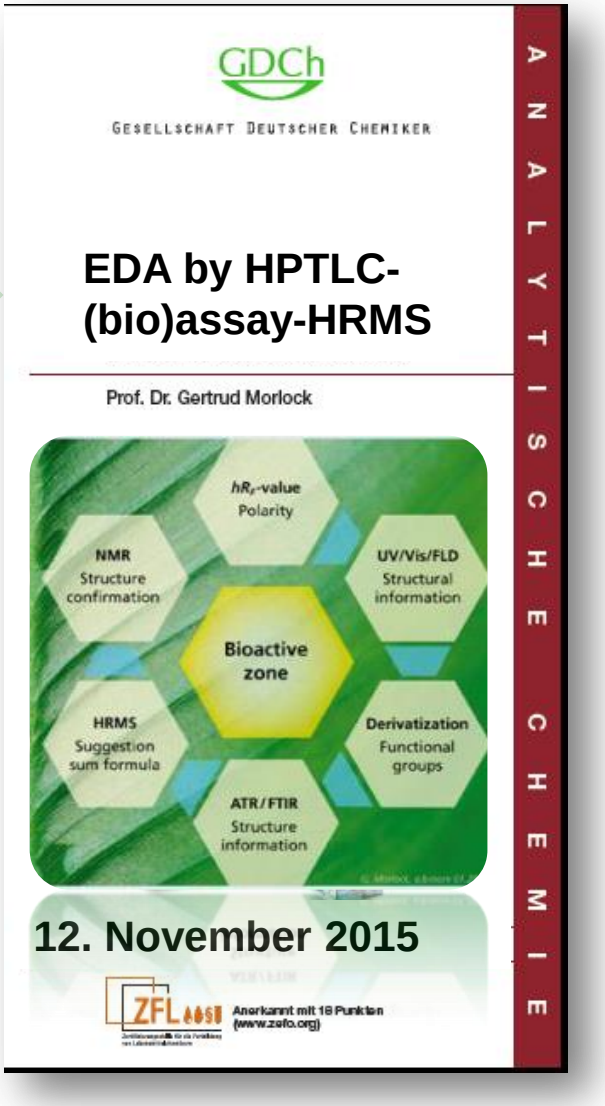


G.E. Morlock, q&more 01.14 (2014) 43-47

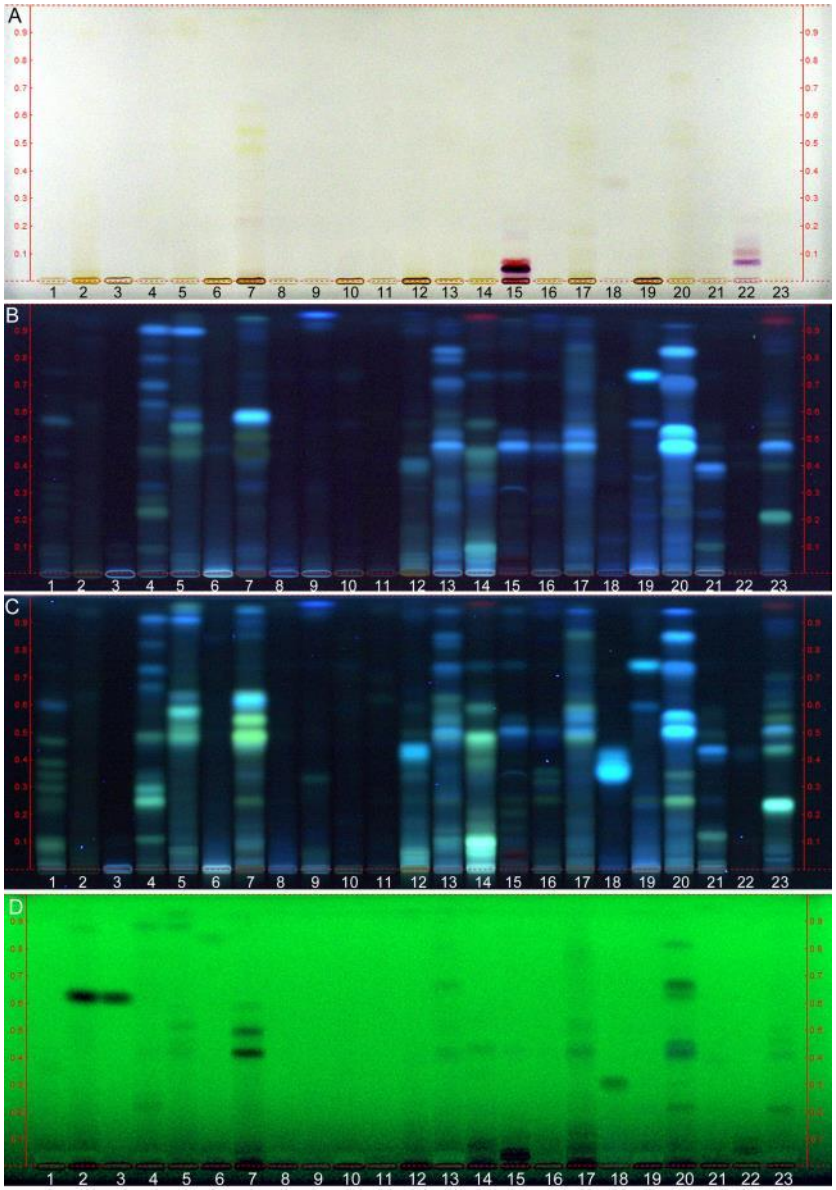
Bioactive compounds of interest in botanicals used as spices or food supplements?

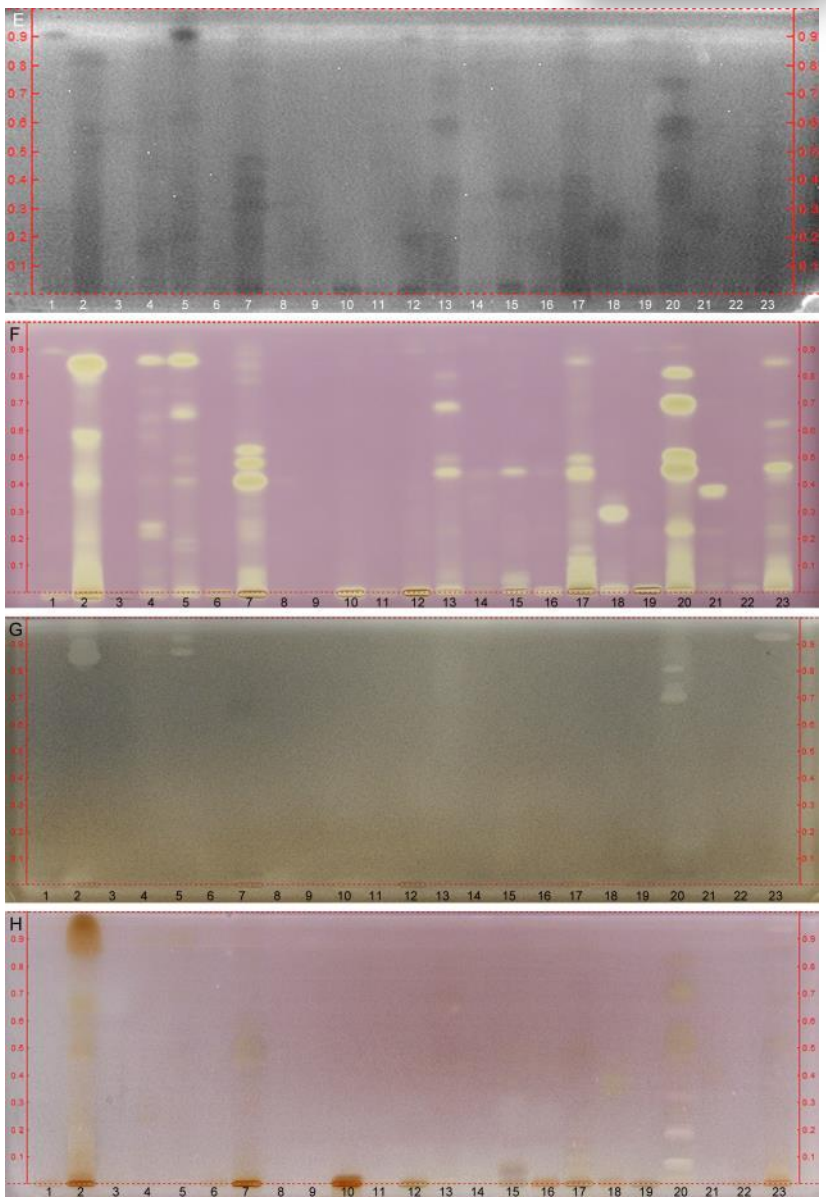
GDCh course 338/15

The day after

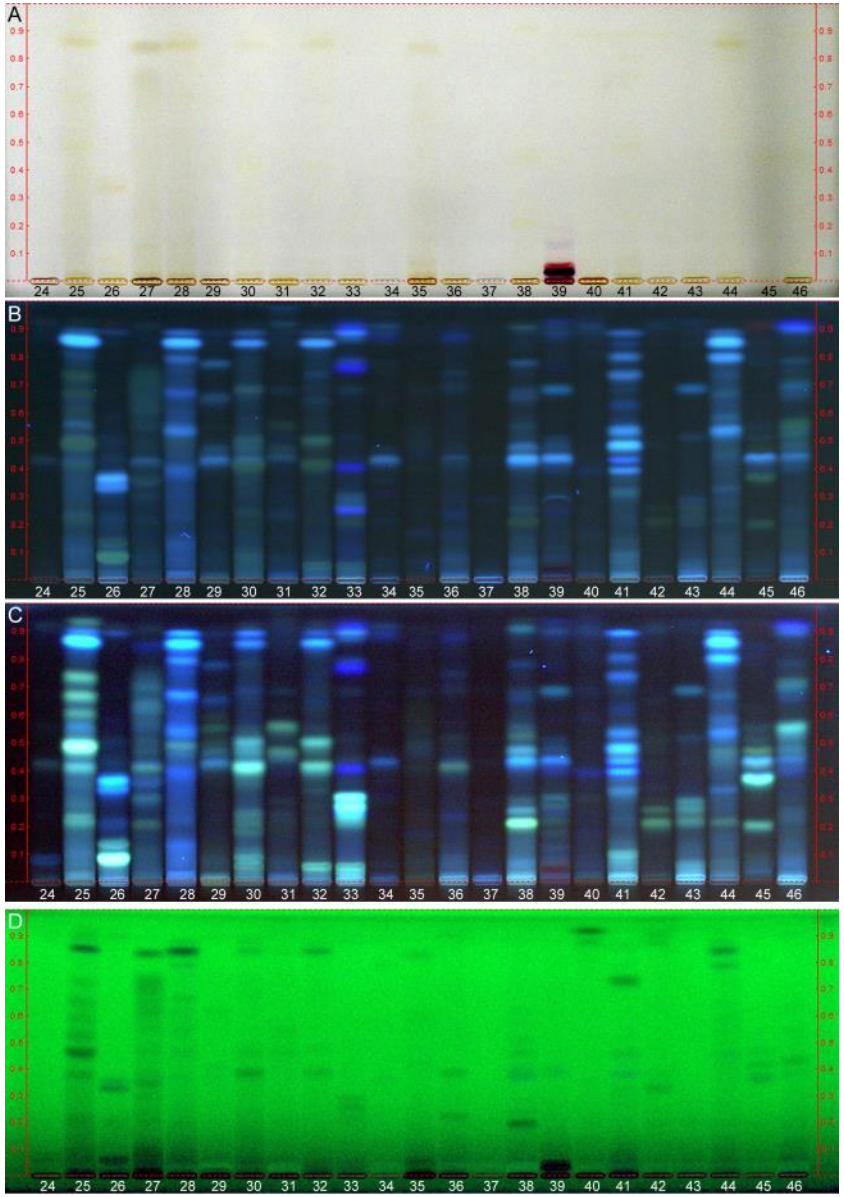


Effect-directed analysis (EDA) of 68 botanicals





EDA of 68 botanicals (#24-46)



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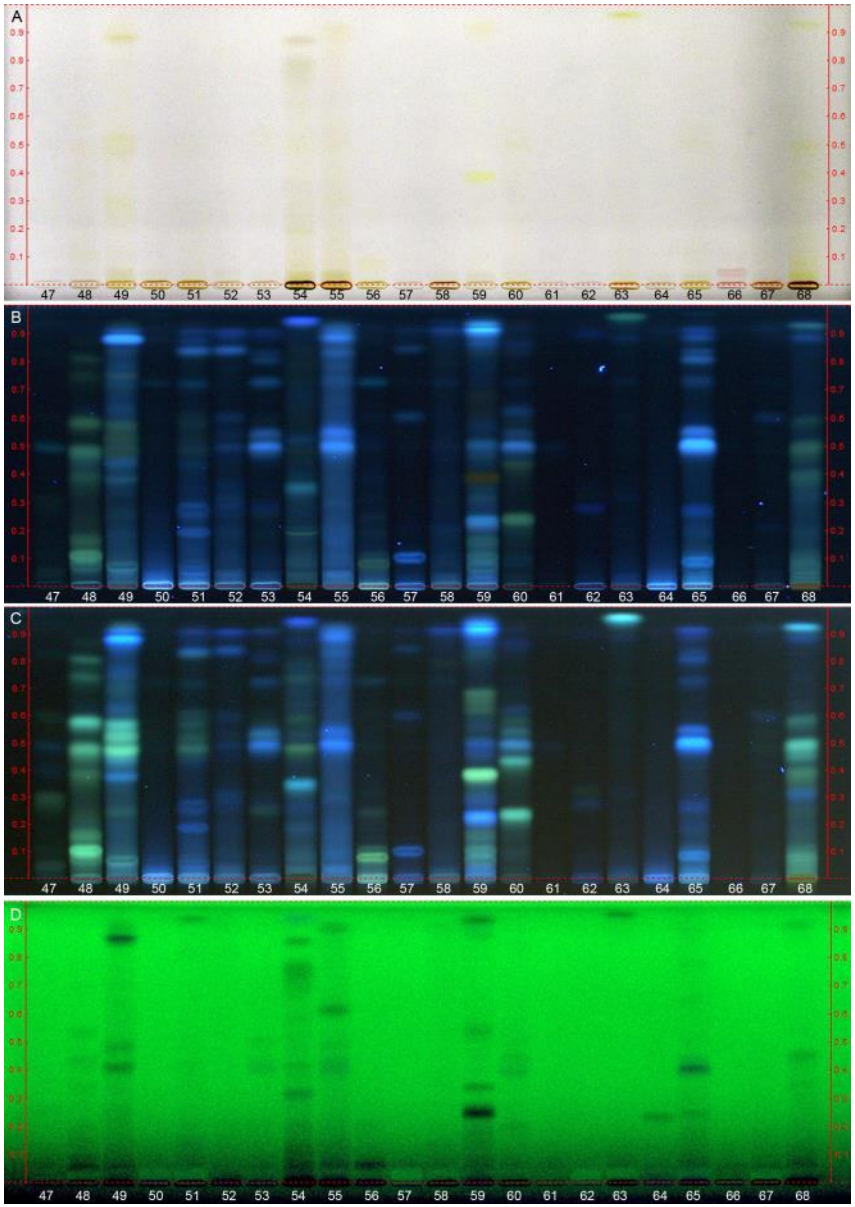
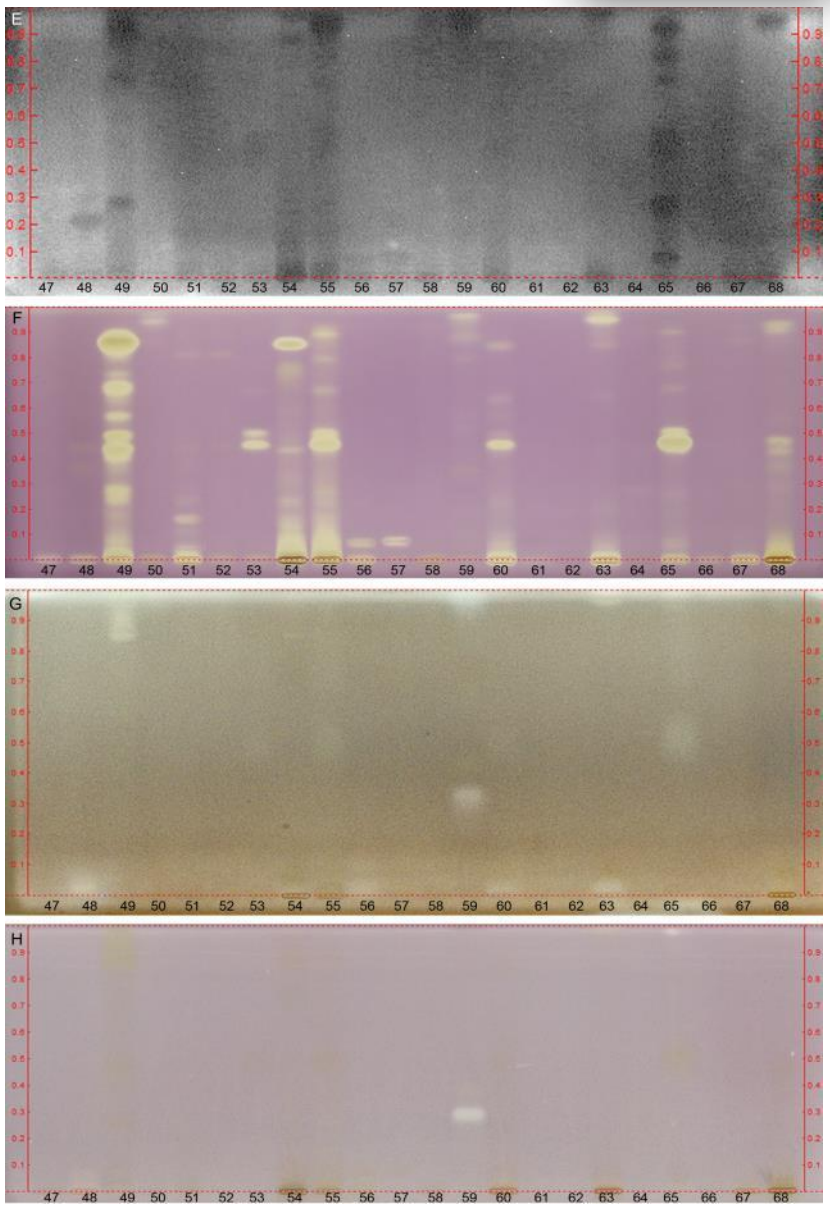
EDA of 68 botanicals (#47-68)





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JLU

EDA of 68 botanicals

1. Ginkgo	24. Heidelbeere	47. Sanddorn
2. Guarana	25. Rosmarin	48. Bockshornklee
3. Kola	26. Lemon verbena	49. Thyme
4. Pfefferminze	27. Eukalyptus	50. Ingwer
5. Oregano	28. Melisse	51. Andorn
6. Zichorie	29. Kümmel	52. Brennnessel
7. Weinblätter	30. Salbei	53. Artischocken
8. Kardamom	31. Hopfen	54. Nelken
9. Zitronenschale	32. Majoran	55. Gerösteter Matetee
10. Hagebutte	33. Orangenschale	56. Jasmin
11. Wacholderbeere	34. Liebstöckelwurzel	57. Sonnenhut
12. Honigbusch	35. Traubenkerne	58. Koriander
13. Schafgarbe	36. Fenchel	59. Licorice
14. Passionsblume	37. Ginseng	60. Weißdornextrakt
15. Hibiskus	38. Holunderblüten	61. Apfelschalen
16. Gelber Früchtetee	39. Roter Früchtetee	62. Sellerieknolle
17. Brombeerblätter	40. Zimtrinde	63. Galgant
18. Acerola	41. Schachtelhalm	64. Knoblauch
19. Holunderbeere	42. Sternanis	65. Taigawurzel
20. Grüner Matetee	43. Orangenblüten	66. Himbeersaftkonzentrat
21. Spitzwegerich	44. Basilikum	67. Traubenschalen
22. Schwarzes Johannis-beersaftkonzentrat	45. Weißdornblätter Charge 2	68. Rooibos
23. Weißdornblätter #1	46. Kamille	

Characterization of bioactive compounds

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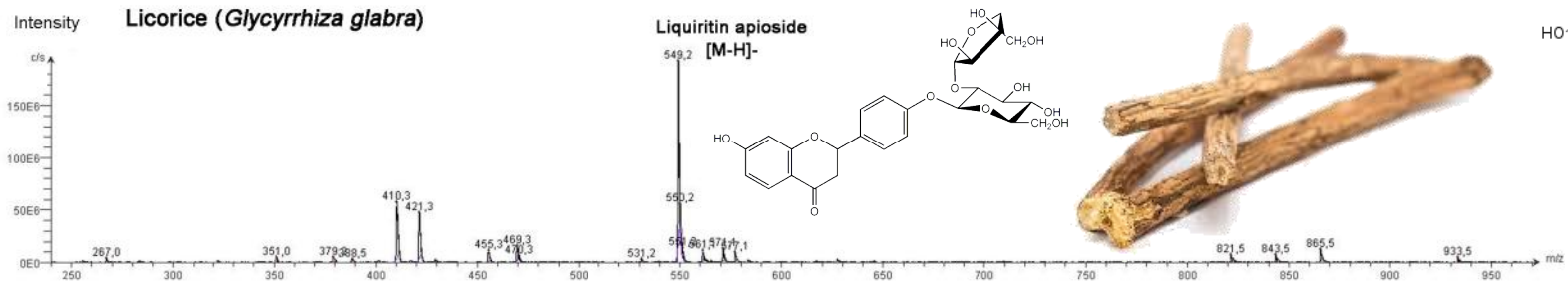
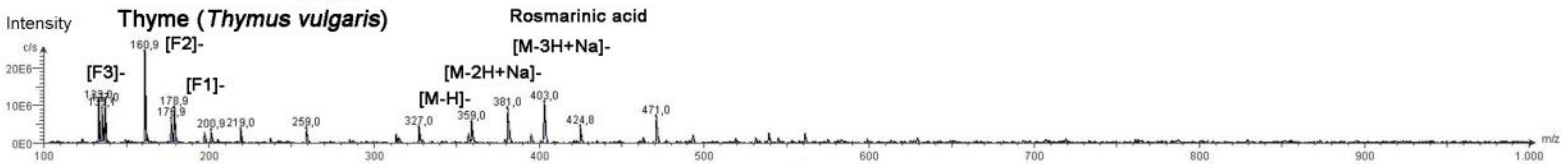
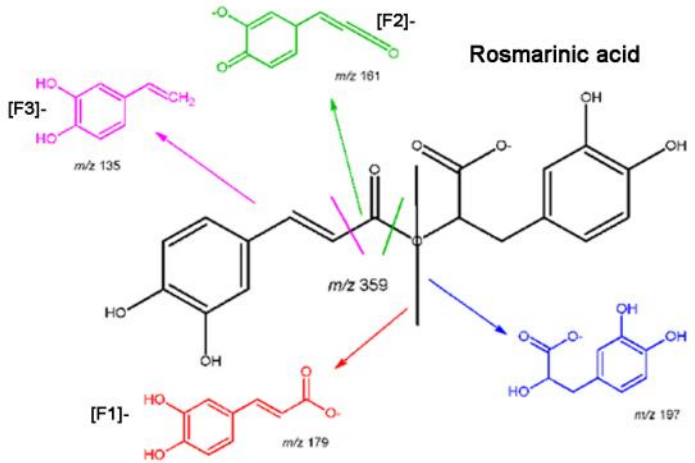


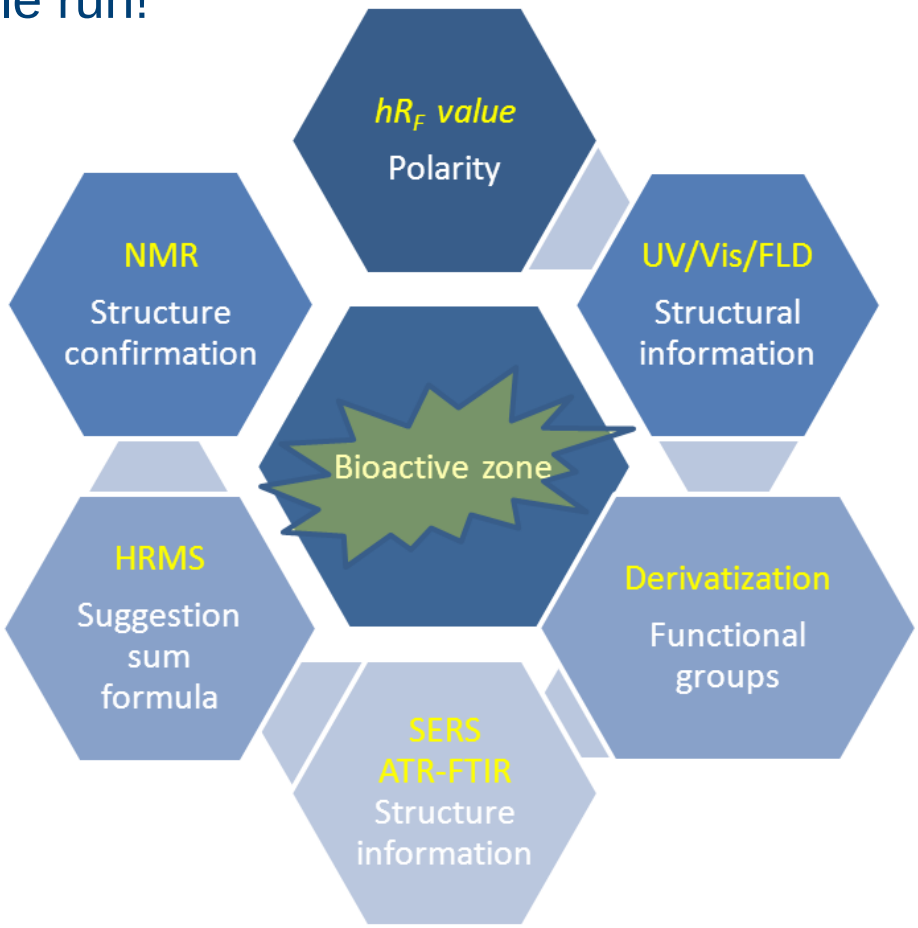




S. Krüger, R. Fornasari, I. Scainelli,
L. Hüsken, G. Morlock, in preparation

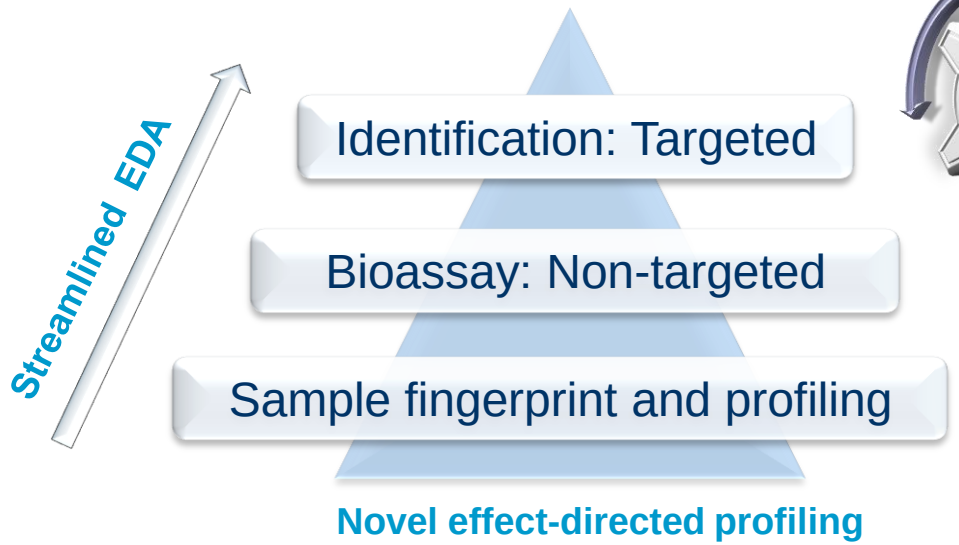
Goal: From bioactive zone to sum formula

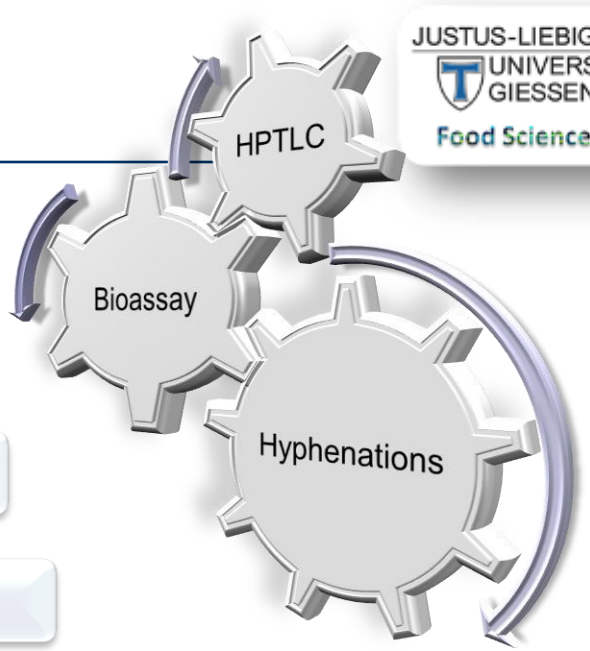
…in a single run!



G. Morlock, in *Instrumental Methods for the Analysis of Bioactive Molecules*, B. S. Patil, G. K. Jayaprakasha, F. Pellati, Eds., ACS Books Publishing, 2014

Conclusion





Comprehensive information

- Analysing samples as raw as possible
- Identifying single compounds generating the effect
- Reduced matrix interference
- Improved capability of detection
- Support decisions in natural product search and health issues

Thank you!

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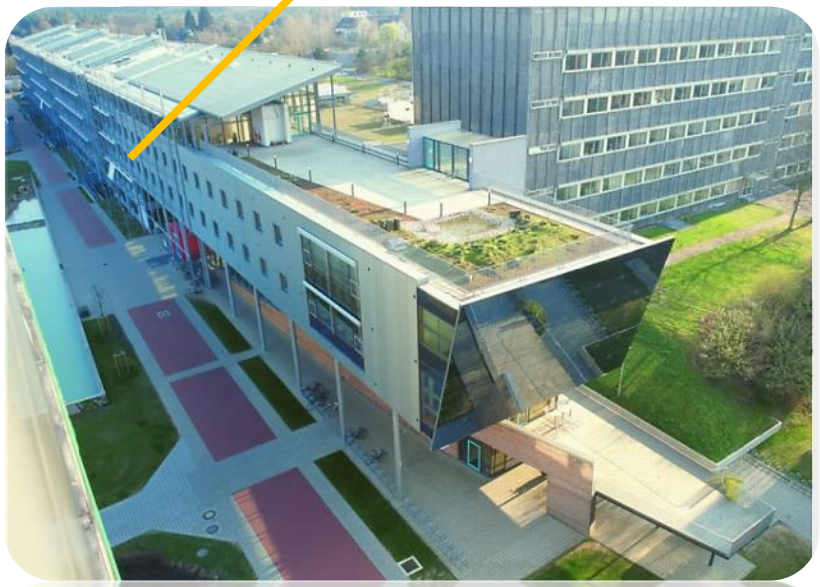
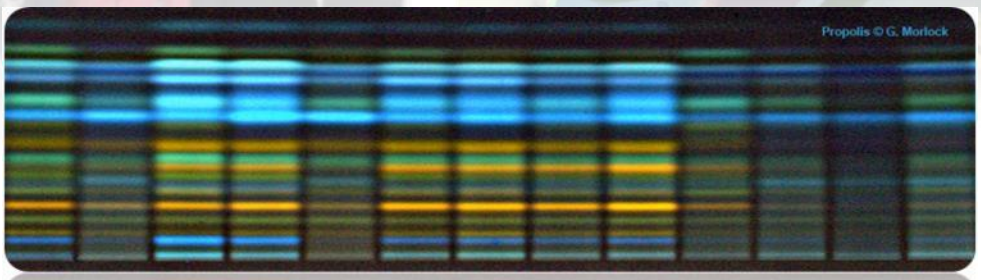
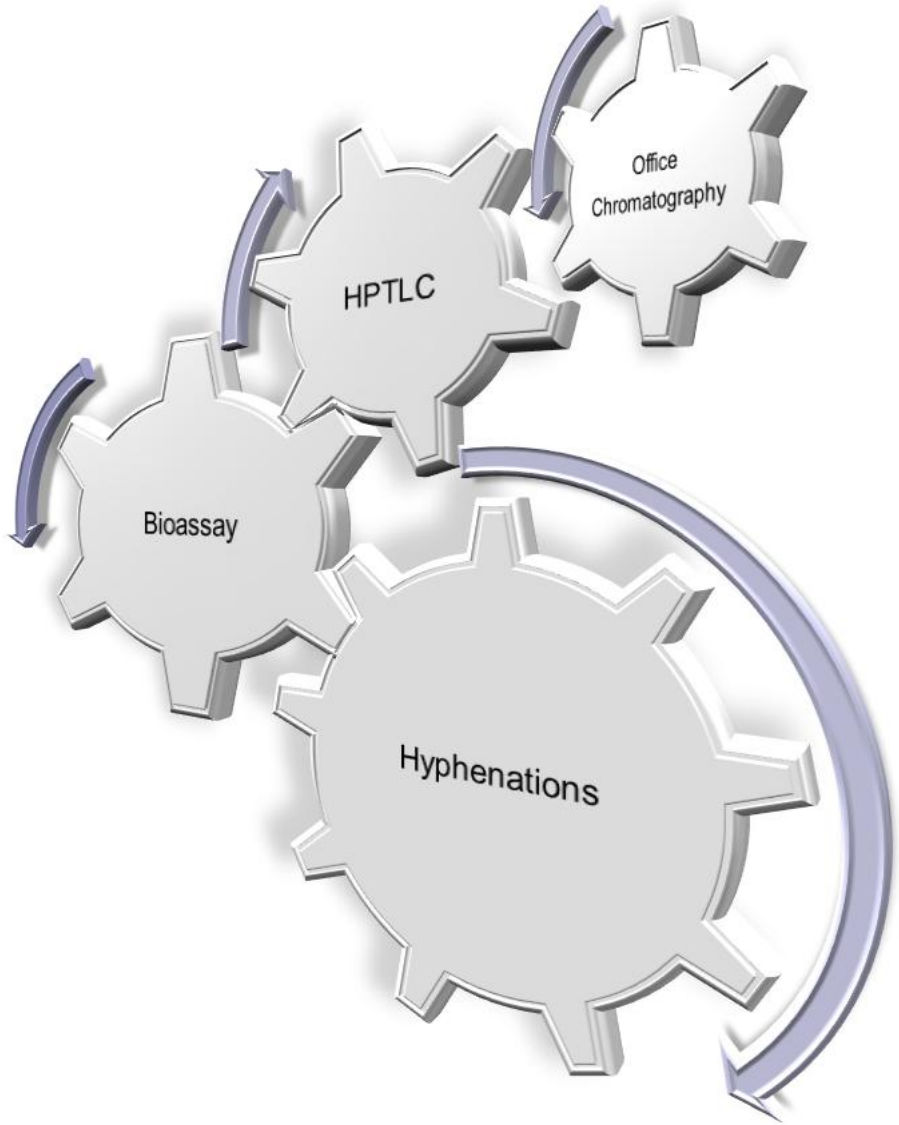
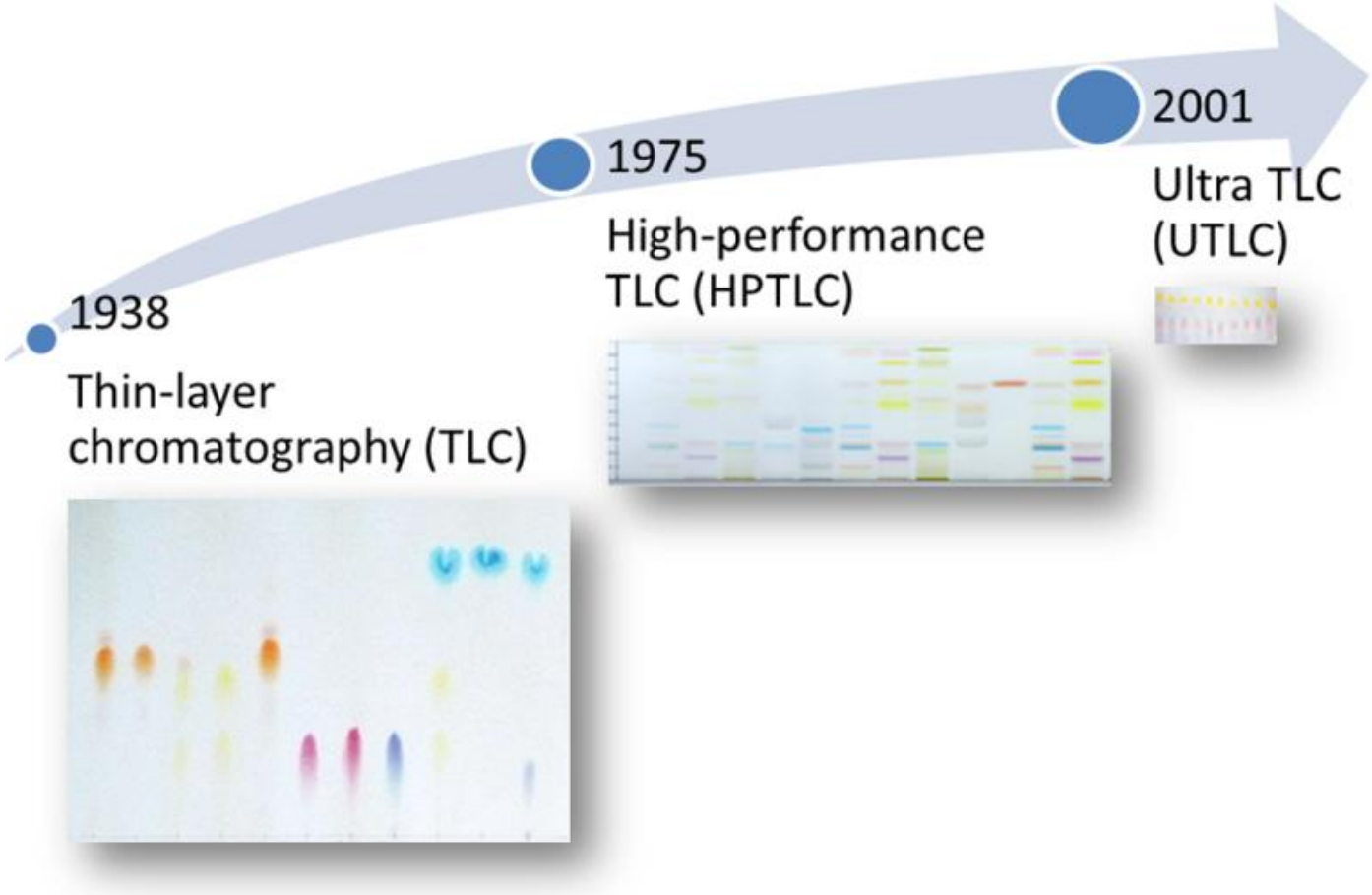




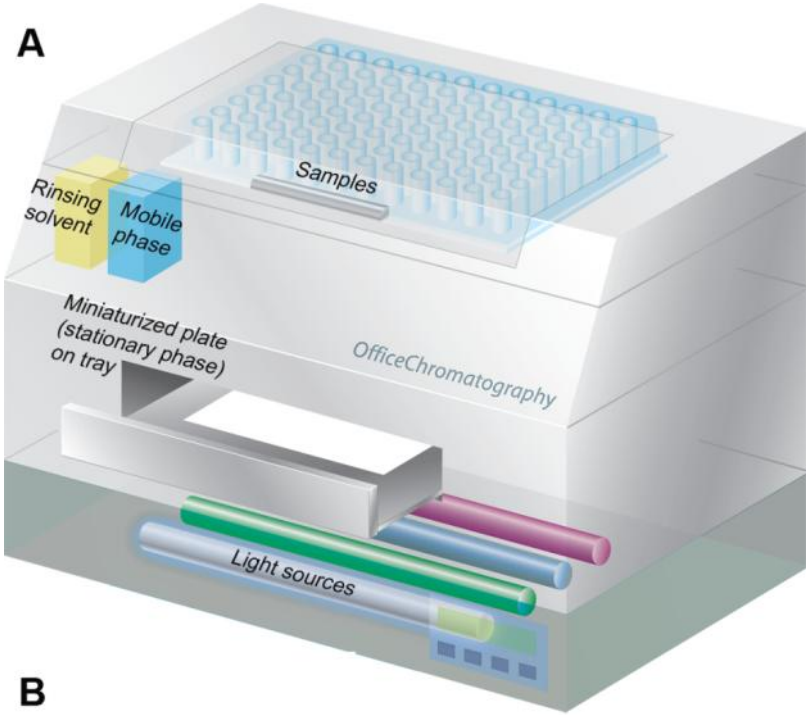
Outlook



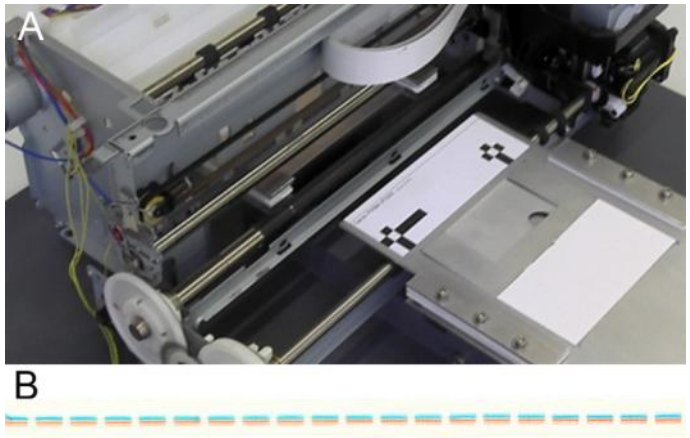
Miniaturization

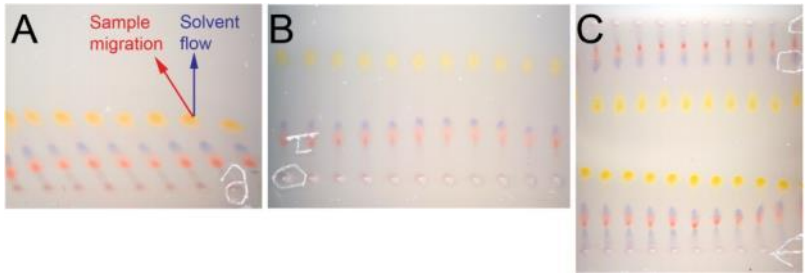


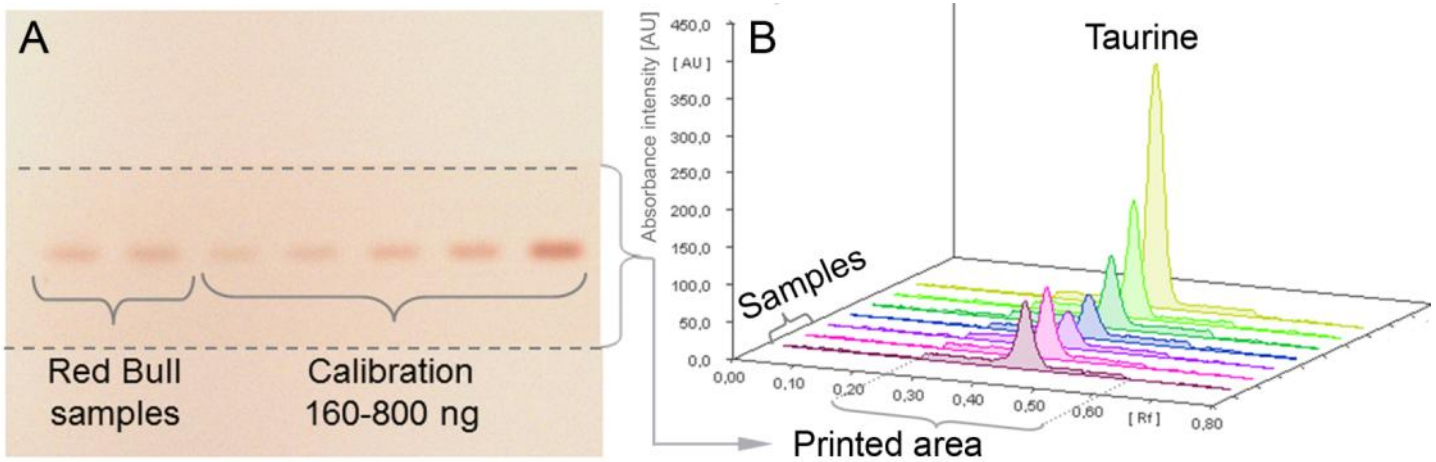
Office chromatography



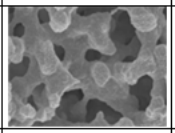
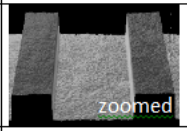
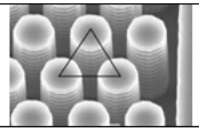
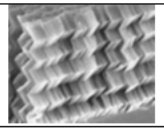
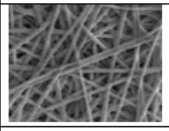
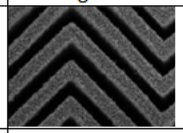
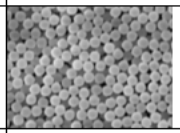
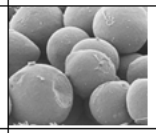
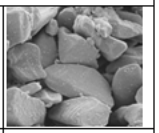
UTLC & PMT


A
B


A
Sample migration
Solvent flow
B
C

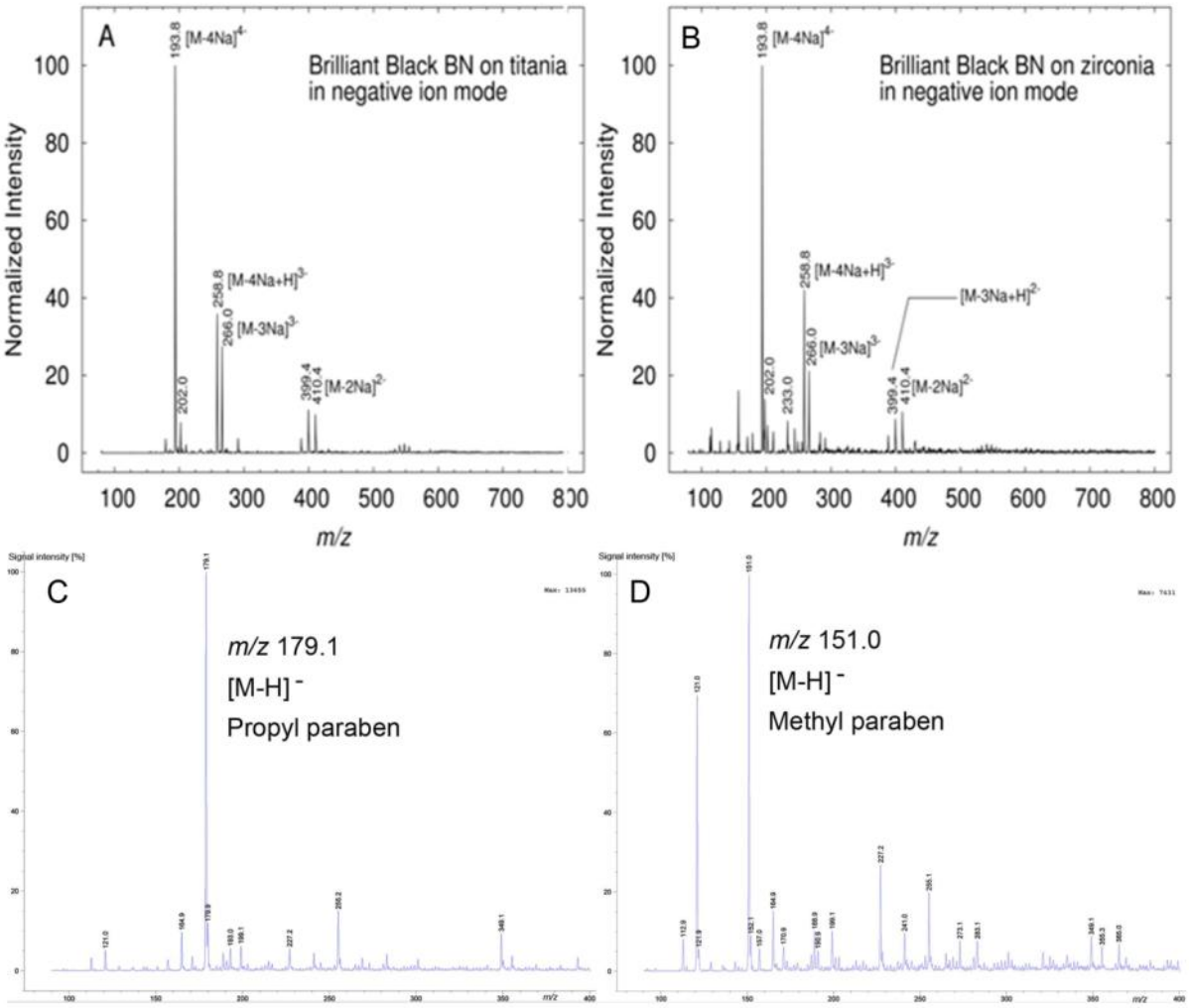

A
Red Bull samples
Calibration 160-800 ng
Absorbance intensity [AU]
450,0
[AU]
350,0
300,0
250,0
200,0
150,0
100,0
50,0
0,0
B
Taurine
Samples
0,00
0,10
0,20
0,30
0,40
0,50
0,60
[Rf]
0,80
Printed area

Novel layers

	UTLC							HPTLC
First report	**2001 [5]**	**2001 [47]**	**2007 [49]**	**2008 [28]**	**2009 [56]**	**2011 [60]**	**2011 [63]**	**1975 [3]**
Layer type	Monolithic layer	Monolayer on channel bottom	Ordered (non)po-rous pillar arrays	Nanostructured layer	Electrospun mat	Carbon-nano-tube-templated microfabrication (CNT-M)	Submicrometer particulate layer with cross linked polymer brushes	Particulate layer
Technique of fabrication	Polymerization on glass plate (sol-gel pro-cess) and opt. photografting	DRIE of Si-wafer surface and coating	Mid/Deep-UV lithography, DRIE of Si-wafer surface and coating	GLAD of inorganic oxides on glass plates	Electrospinning of (composite) polymer solutions on aluminium foil	Coating CNTs with silica by (pseudo) atomic layer deposition plus second coating	Slurry overlay on Si-wafer and brush coating by polymerization	Slurry overlay on various carriers (glass plate, aluminium or polymer foil) and coatings
Layer icon								 
Layer struc-ture	Monolithic texture with 1-2 µm macropores	Monolayered porous silicon bottom of a nanochannel (0.7 µm wide, 0.3 µm deep)	Monolayer coa-ting or monolithic silica shell of cylindrical pillars (Ø 4 µm, 10 µm high, spaced 0.3 - 1.7 µm) in a 70 µm nanochannel	Column array of verticals, posts, helices, zig-zags or blades (spaced 2 - 50 nm) with (an)isotropic structure	Spun mat of nanofibers (Ø 200-400 nm, cm to m long) forming cylindric channels	Silica coated (20-60 nm) herring-bone hedge array (3-4 µm wide, spaced 4-7 µm) forming channels (50-100 µm long)	Non-porous particles coated with a polymer brush layer	Spherical / Irregular particles of ∅ 5 - 7 µm
Layer thick-nesses (µm)	10 - 50	Monolayer 0.05 – 0.3	Monolayer or 0.5 µm porous shell	1.3 - 7	15 - 25	50	15	50 - 200
Adsorbent types	Silica gel, poly-(4-methyl-styrene-co-chloromethyl-styrene-co-divinylbenzene)	C8, C18	C8; C18 [50]	Silica, zirconia alumina, titania, C18	Glassy carbon, polyvinyl alcohol, polyacrylonitrile	Silica, amino	Polyacrylamide; poly(GMA-co-DEGDMA-NH_2) [64]	Silica, amino, cyano, diol, C2, C8, C18, cellulose, *etc.*
Layer geome-try (mm)	60 × 36 or 30 x 33	0.7 × 20	0.14 x 40; 10 x 30 [50]	25 × 25 or 100 × 20	30 × 60, indivi-dually sliceable	12 x 60	25 × 25	200 × 100, individually sliceable

G. Morlock, *J. Chromatogr. A* 1382 (2015) 87-96

Mass spectra



Bioassay printing