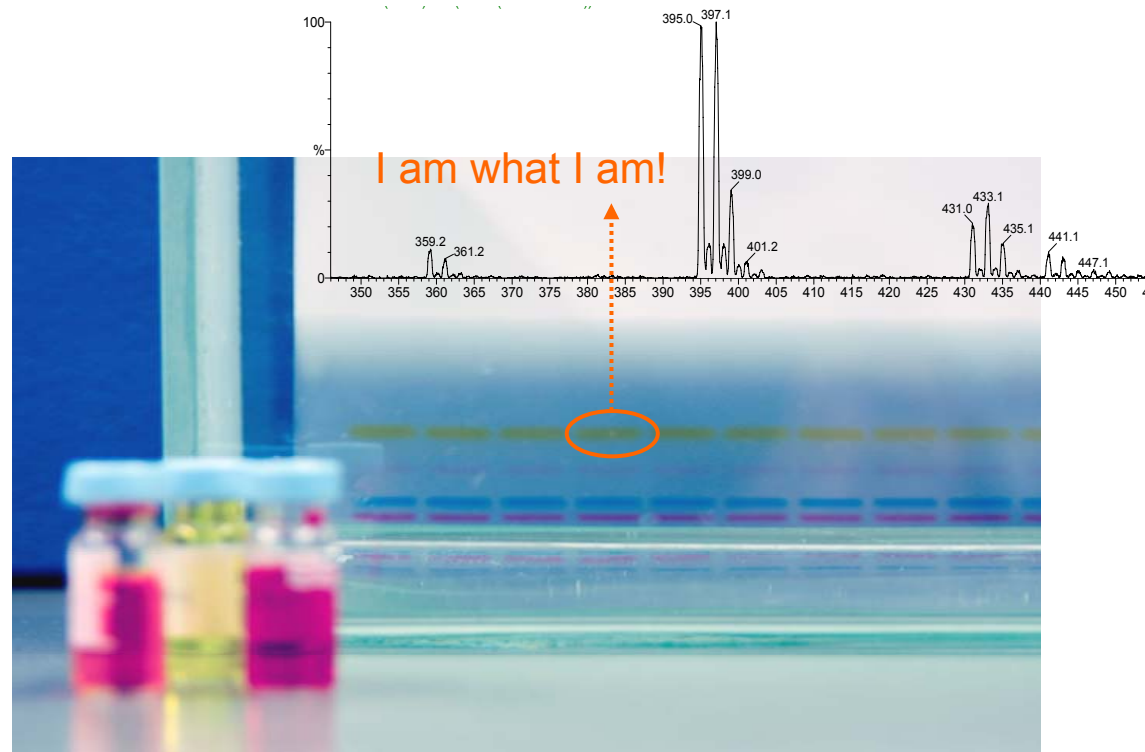




Automated coupling of planar chromatography with mass spectrometry

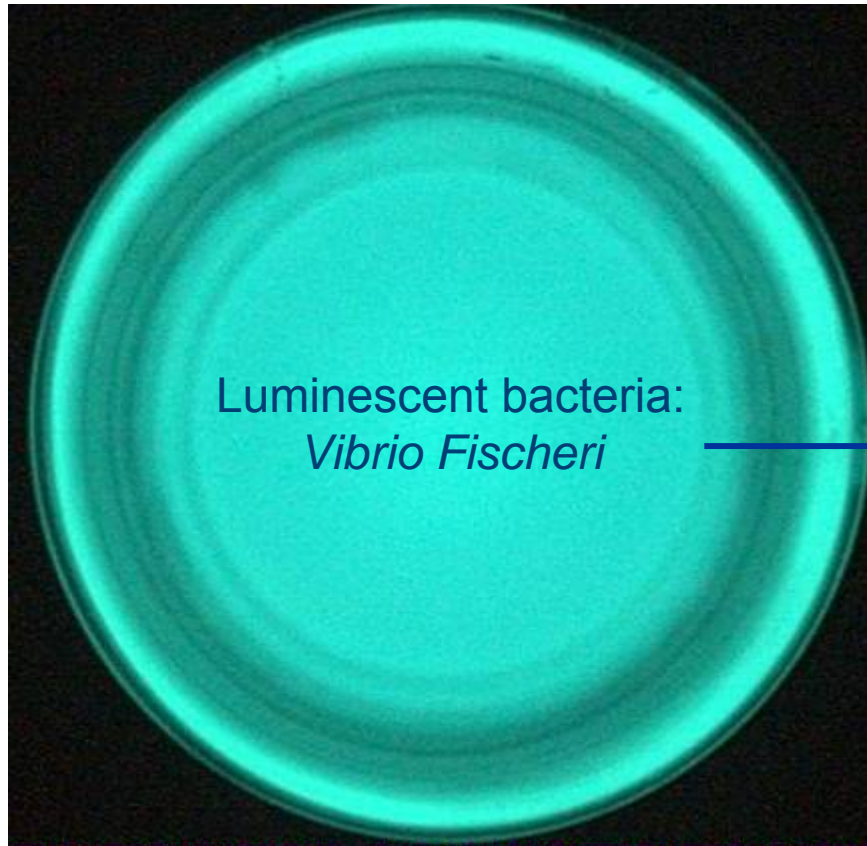


G. Morlock*, M. Aranda, L. Luftmann

*Institute of Food Chemistry, University of Hohenheim, Stuttgart, Germany

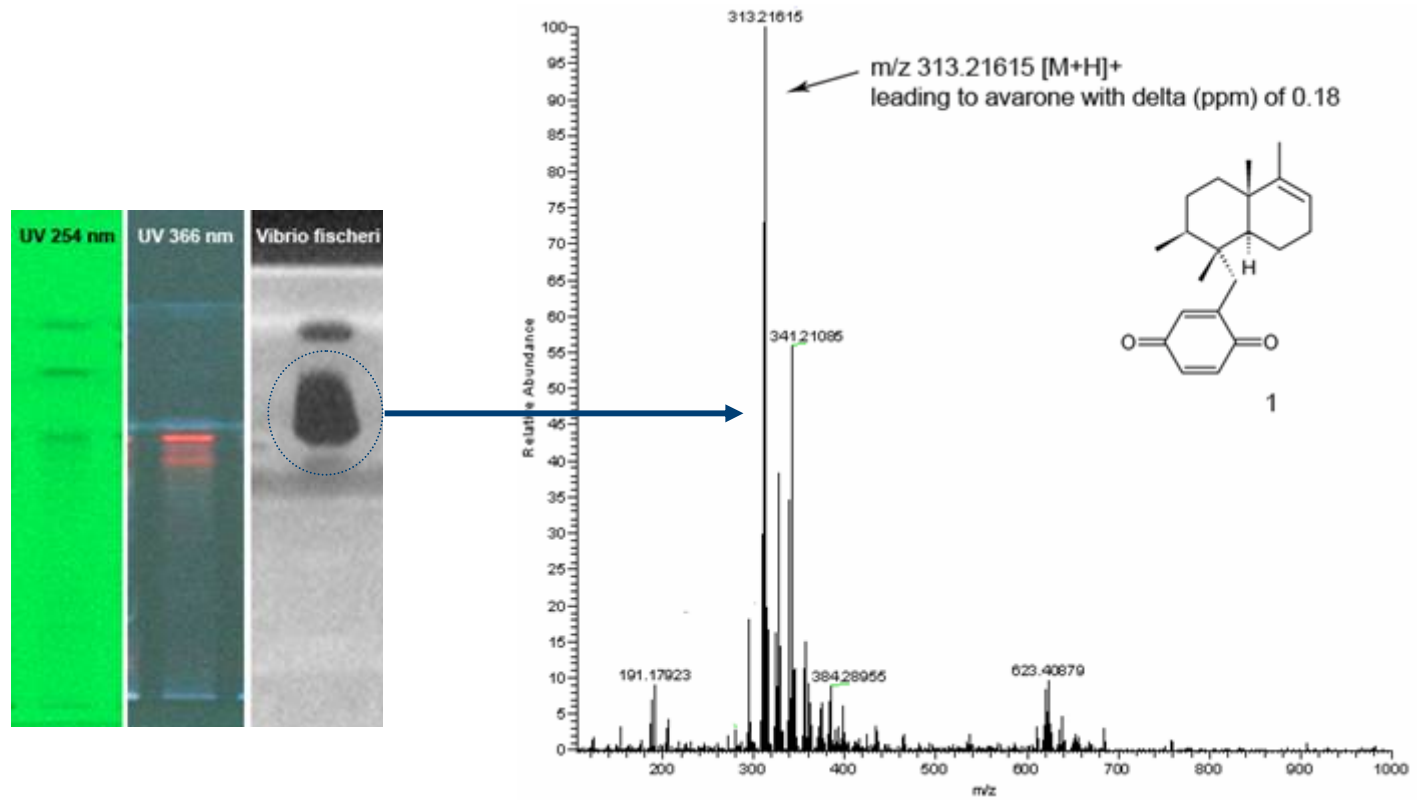


Detection of bio-active compounds





What is it?





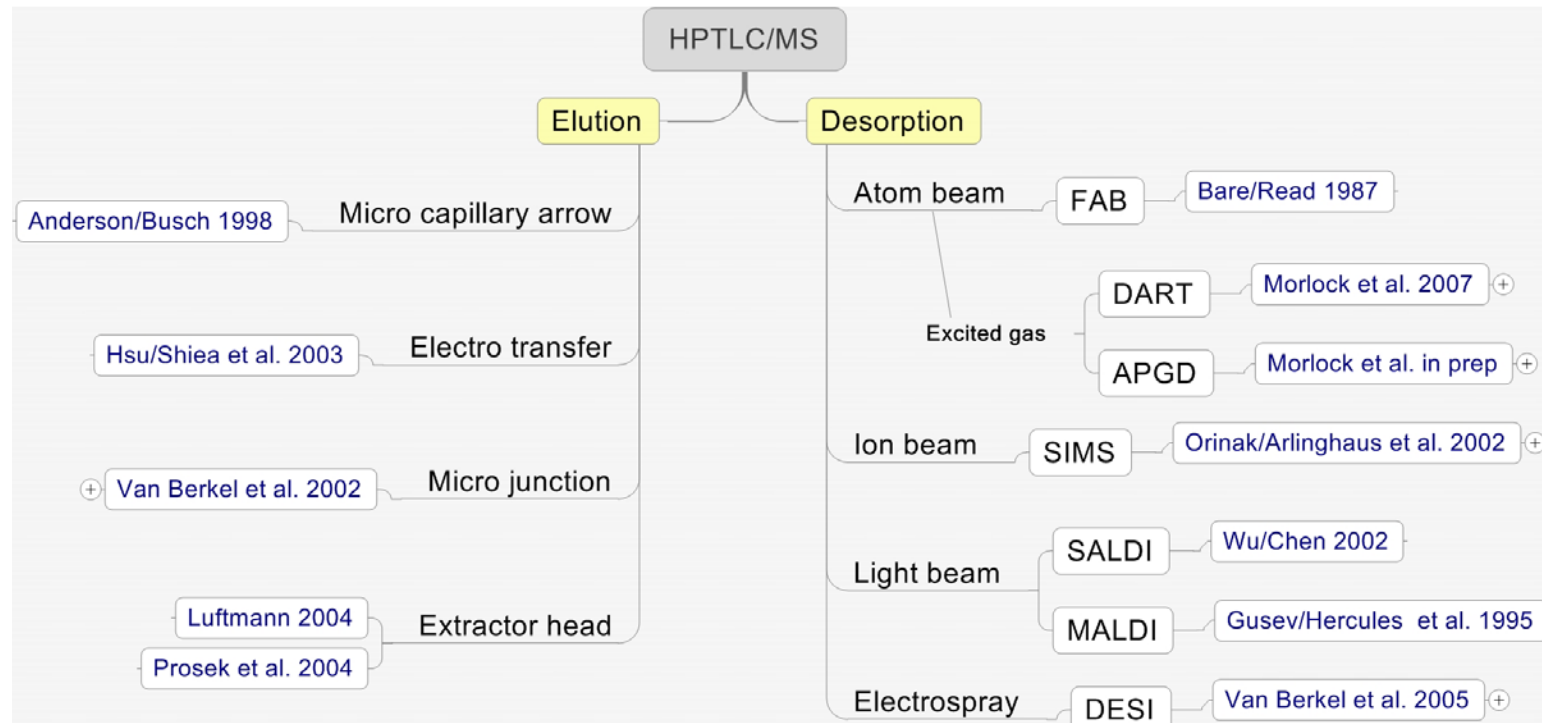
HPTLC → MS

Institute of Food Chemistry
University of Hohenheim, Stuttgart



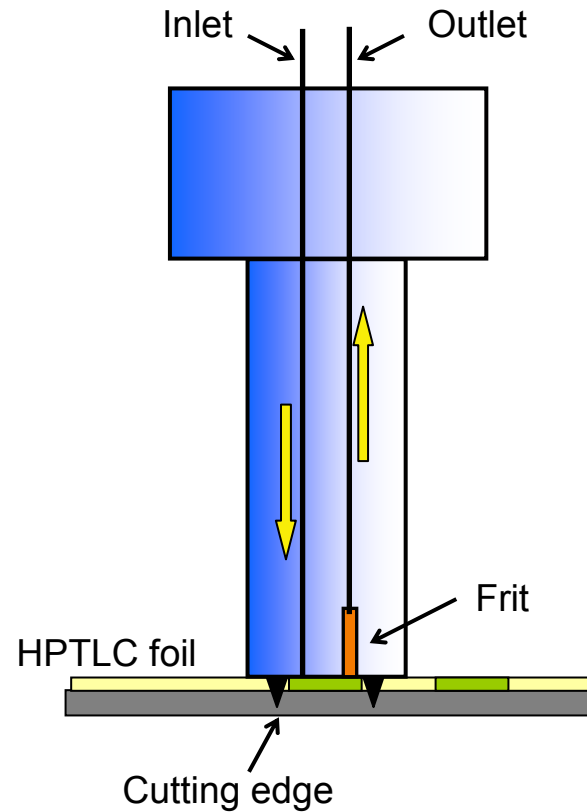


Approaches of HPTLC/MS coupling





Online extraction

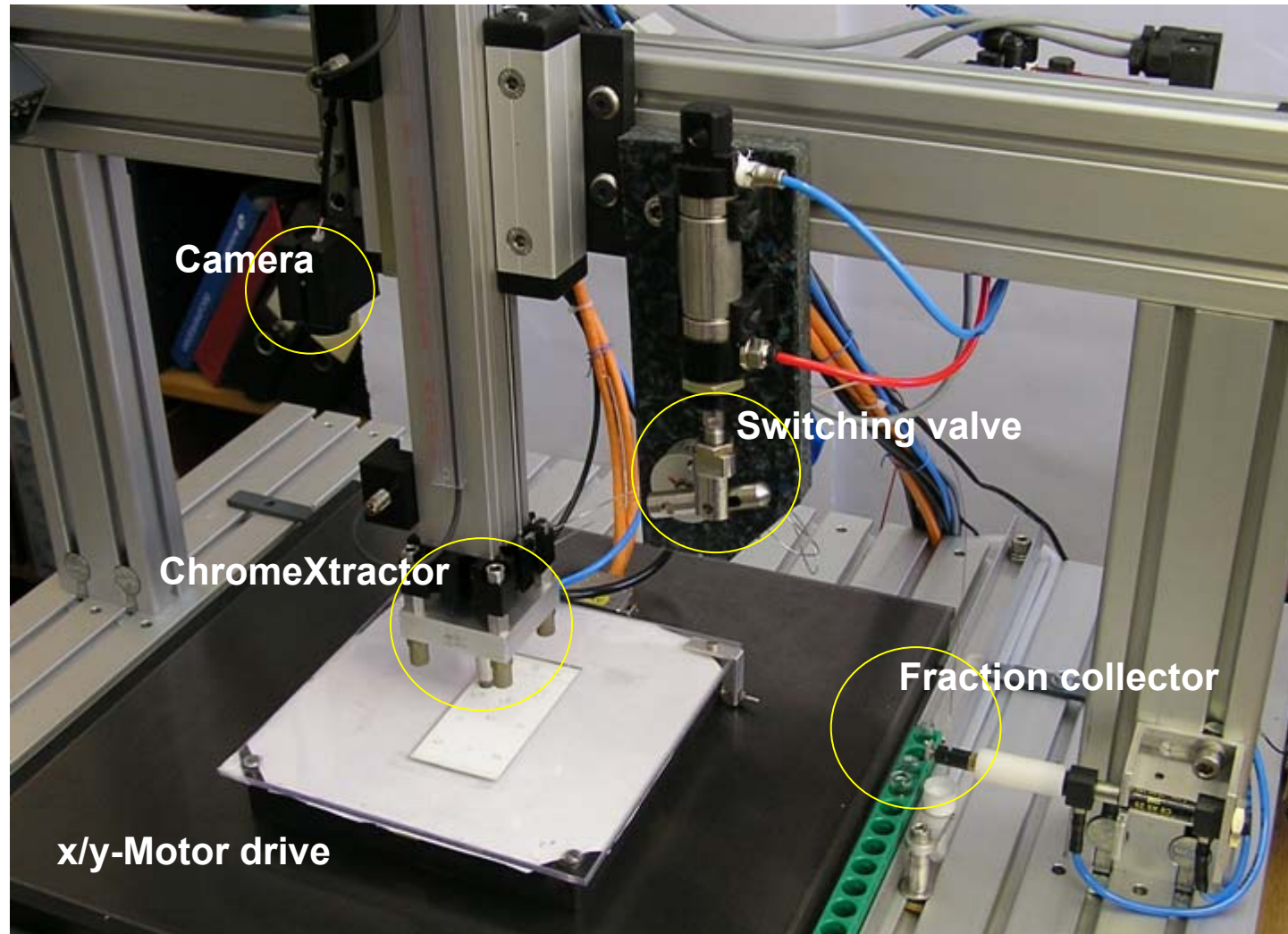


H. Luftmann, Anal Bioanal Chem 378 (2004) 964

A. Alpmann, G. Morlock, Anal Bioanal Chem 386 (2006) 1543

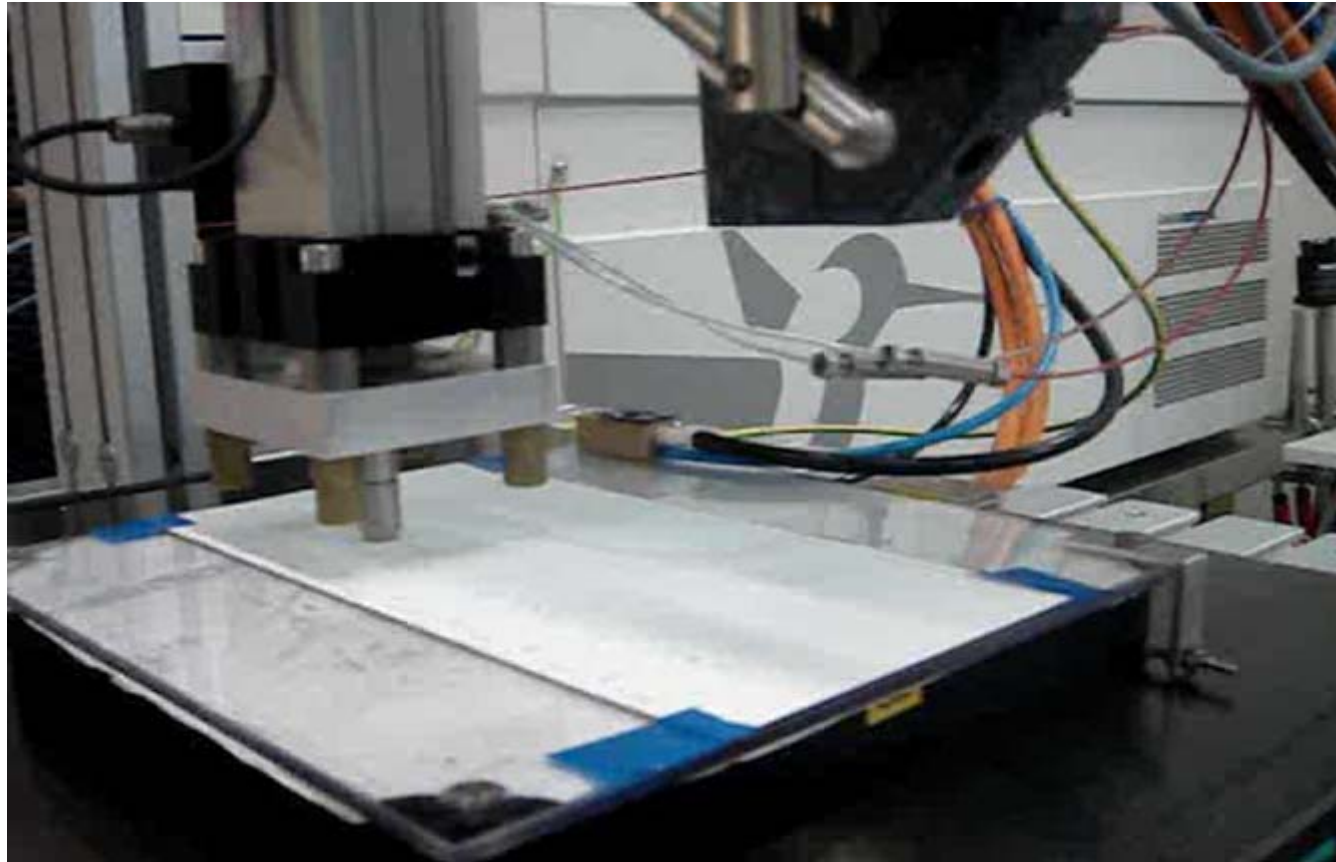


The hands-free interface called 'R3D3'



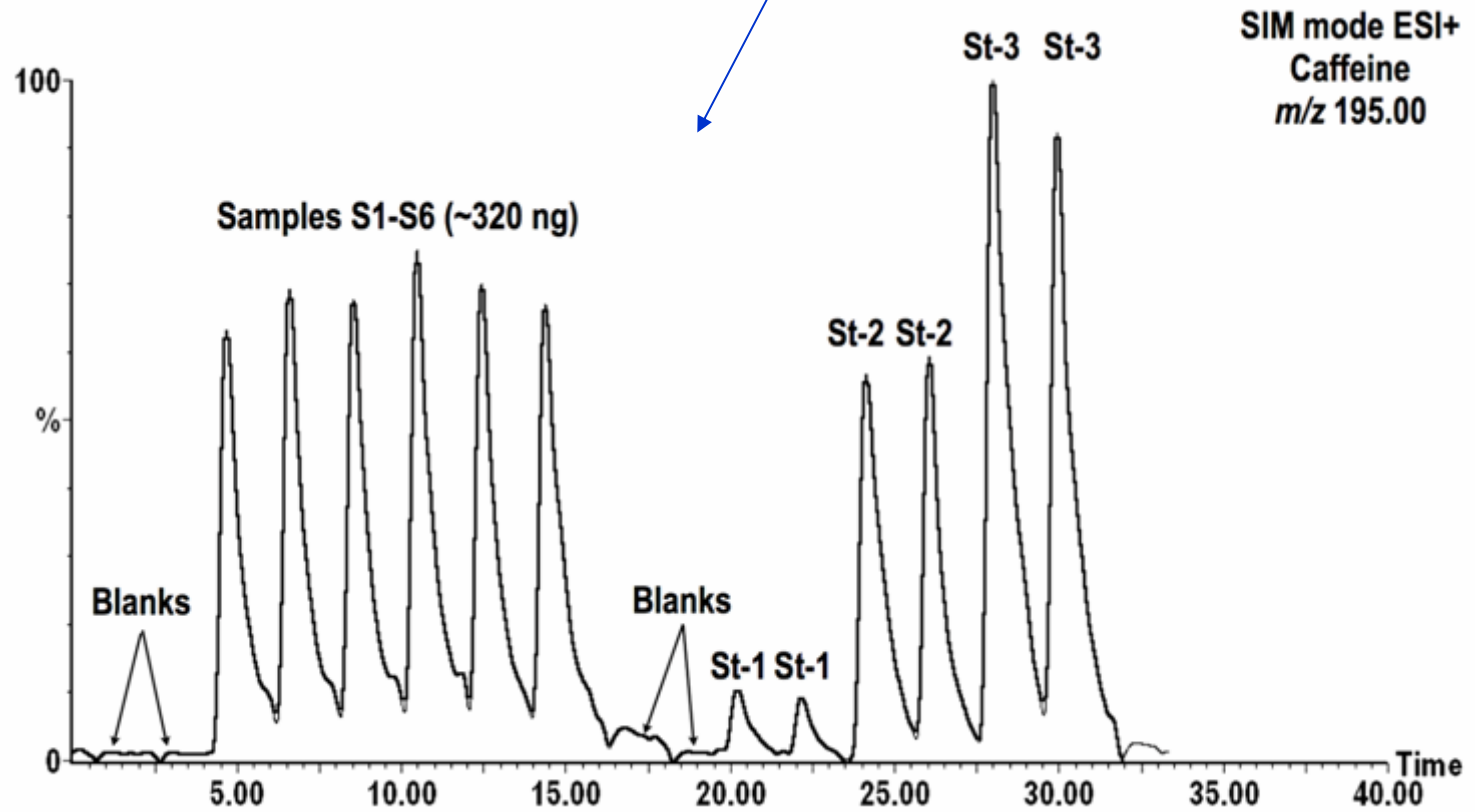
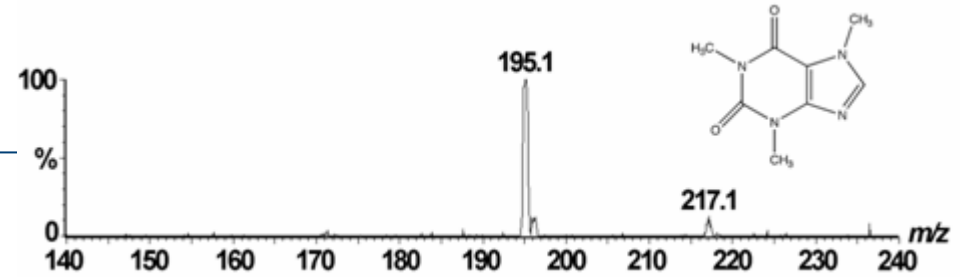


R3D3 working...





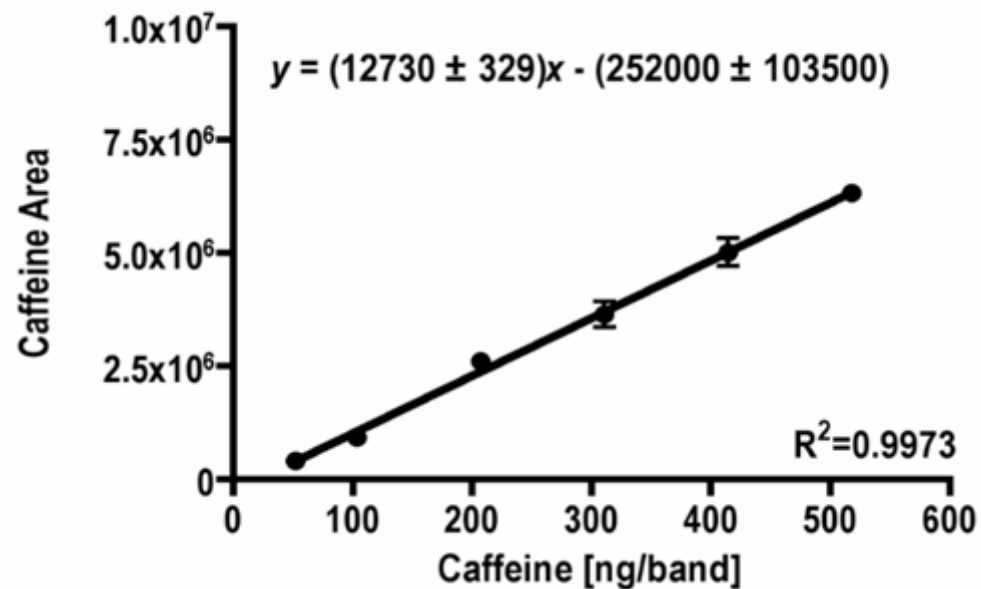
Elution profile





Data of validation without IS

- repeatability (%RSD) in matrix of 5.6 % ($n = 6$)
- linear response (R^2) of 0.9973



H. Luftmann, M. Aranda, G. Morlock, Rapid Commun Mass Spectrom 21 (2007) 3772



Analysis of samples containing caffeine

→ comparable findings to validated HPTLC/UV methods (F-test, t-test)

Sample	Pharmaceutical Mean \pm SD (mg/tablet)	Energy drink Mean \pm SD (mg/100 mL)
HPTLC/ESI-MS RSD (% , n = 6)	102.09 \pm 5.76 (5.6)	32.91 \pm 1.60 (4.9)
HPTLC/UV RSD (% , n = 5)	101.98 \pm 2.30 (2.3)	33.71 \pm 0.96 (2.8)
Label	100	32

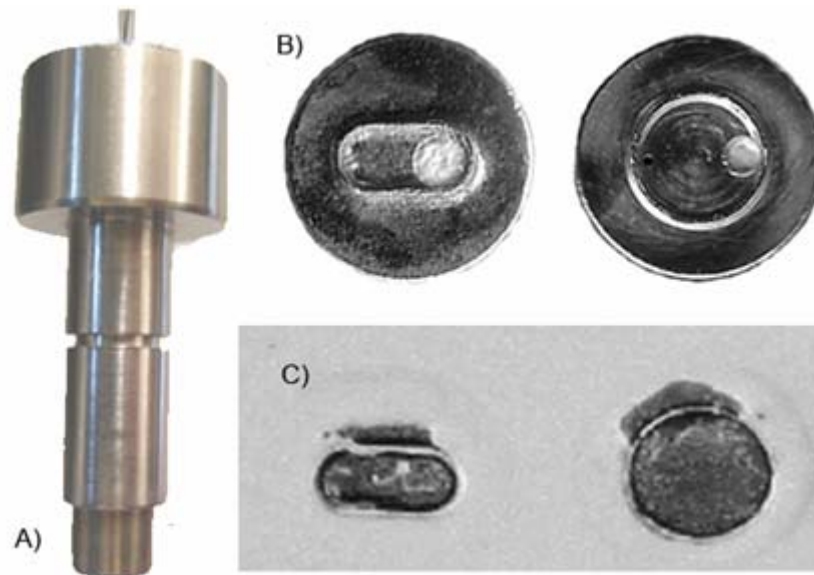


Comparison of differently shaped cutting edges

Cutting edge height

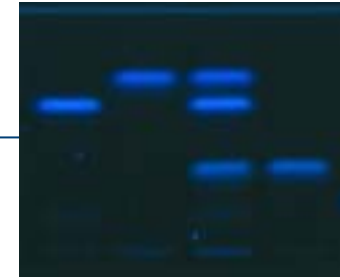
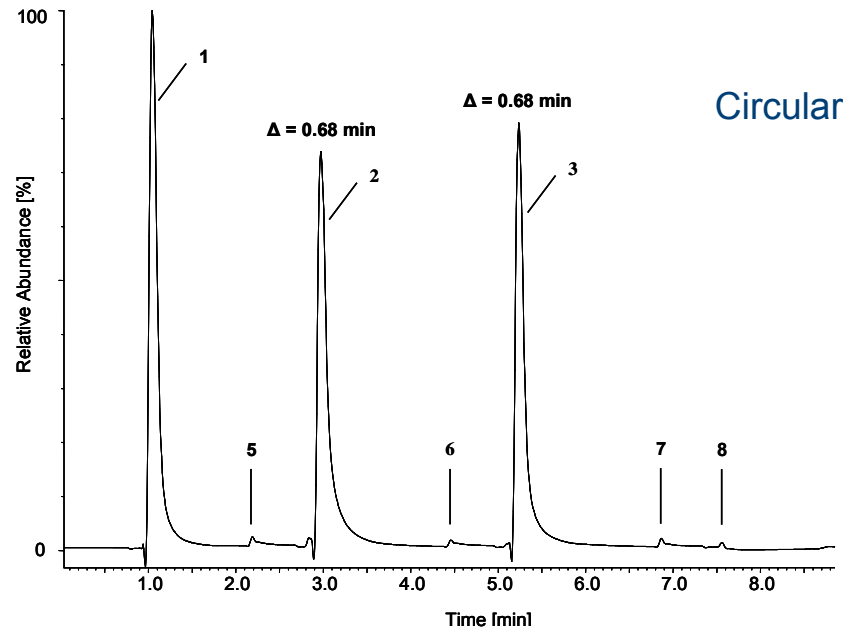
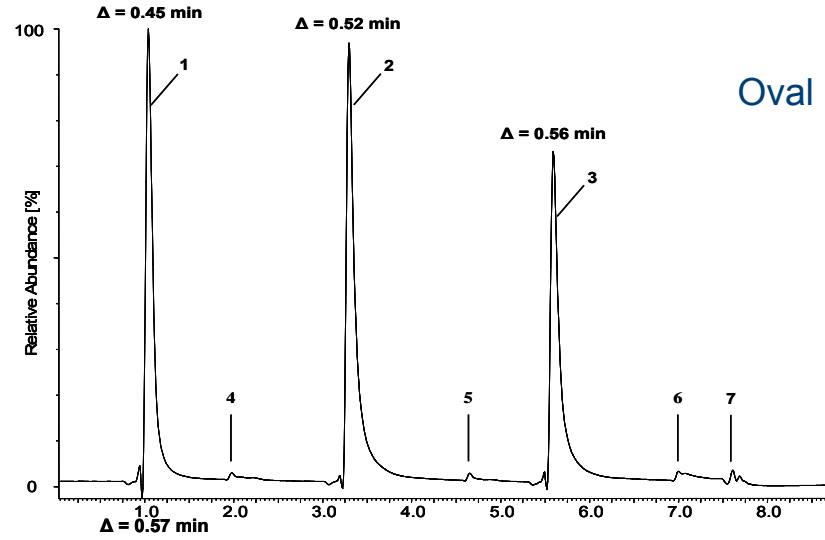
- 0.2 mm for standard HPTLC layers (plates or foils)
- 0.1 mm for extra-thin layers → U. Jautz et al. Anal Bioanal Chem 387 (2007) 1083
- 0.5 mm for preparative layers → E. Dytkiewitz et al. see poster

Cutting edge geometry

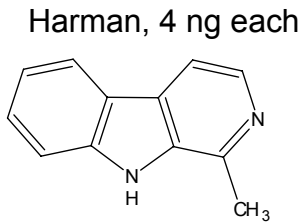




Elution profiles

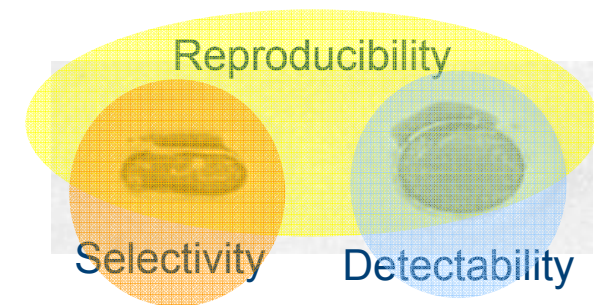
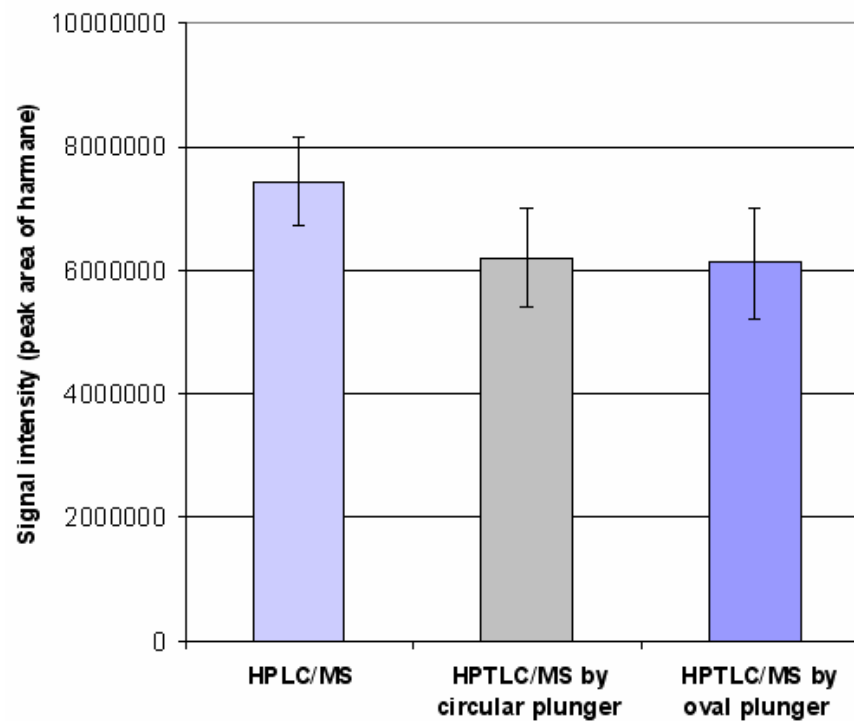


GluP1 AcC Mix A Harman





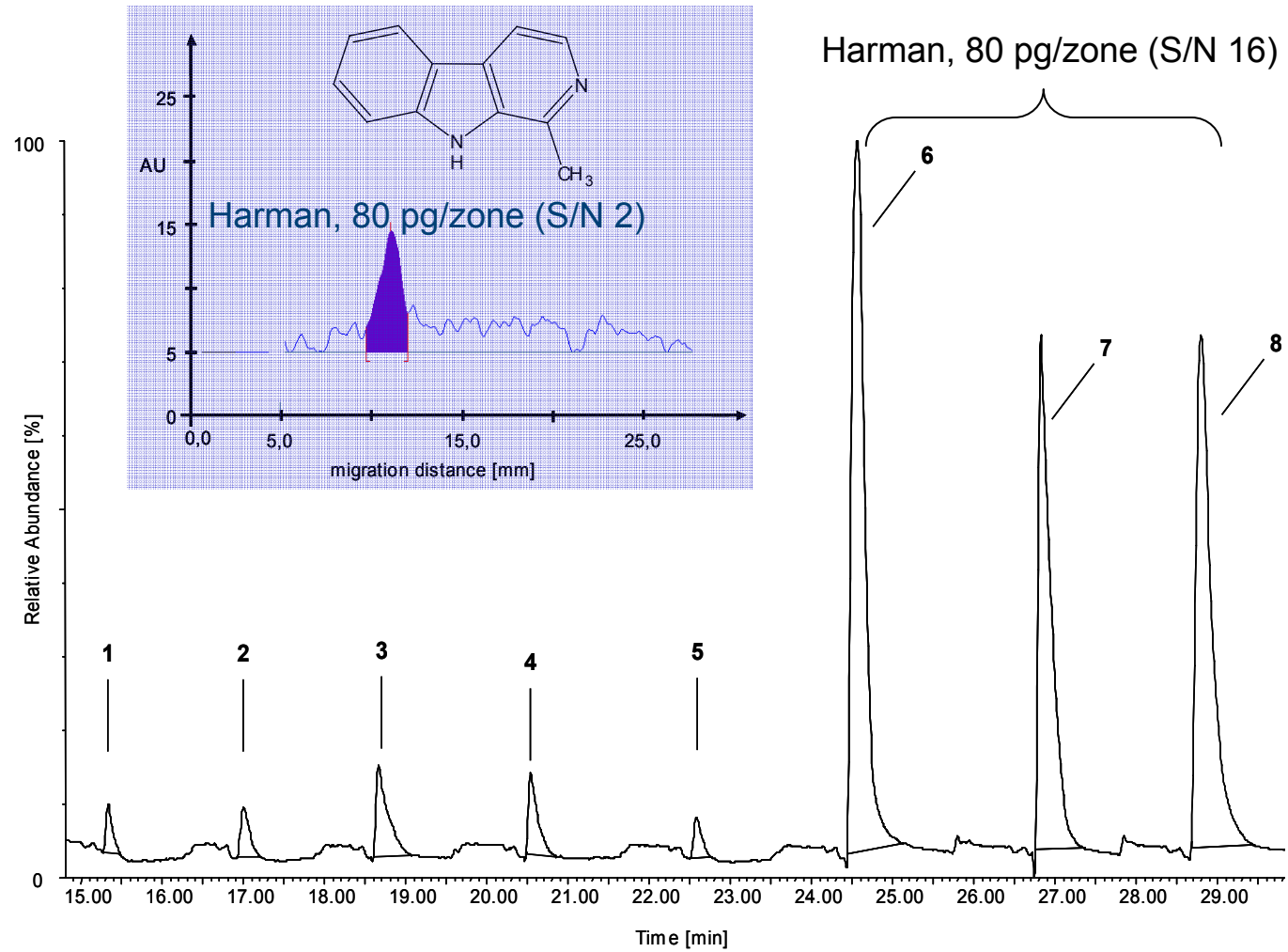
Elution profiles



U. Jautz, G. Morlock, J Planar Chromatogr, in submission



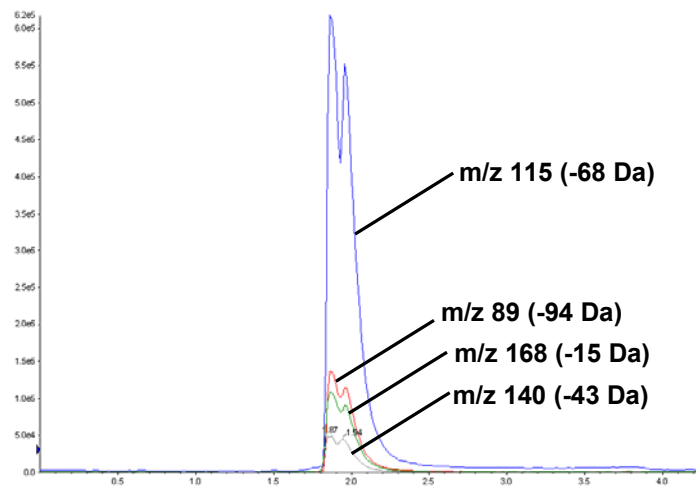
Detectability: FLD versus MSD



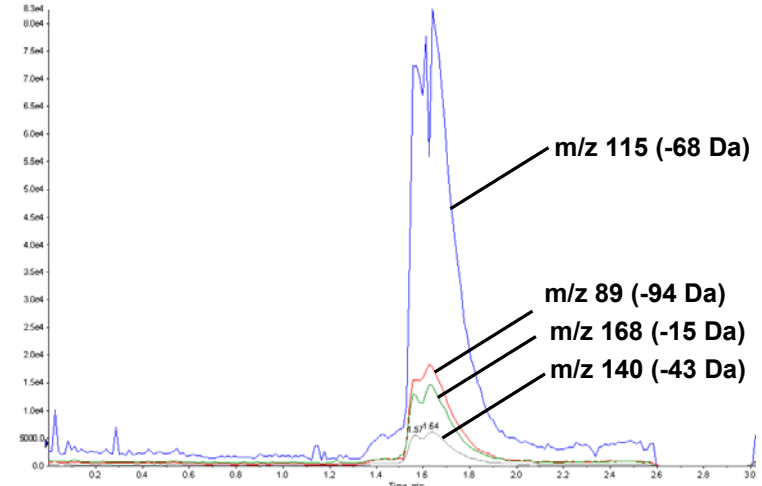


Detectability by HPTLC/ESI-MS-MS

- LOQ better than 20 pg/zone Harman (S/N 20)
- detectability comparable to HPLC/MS



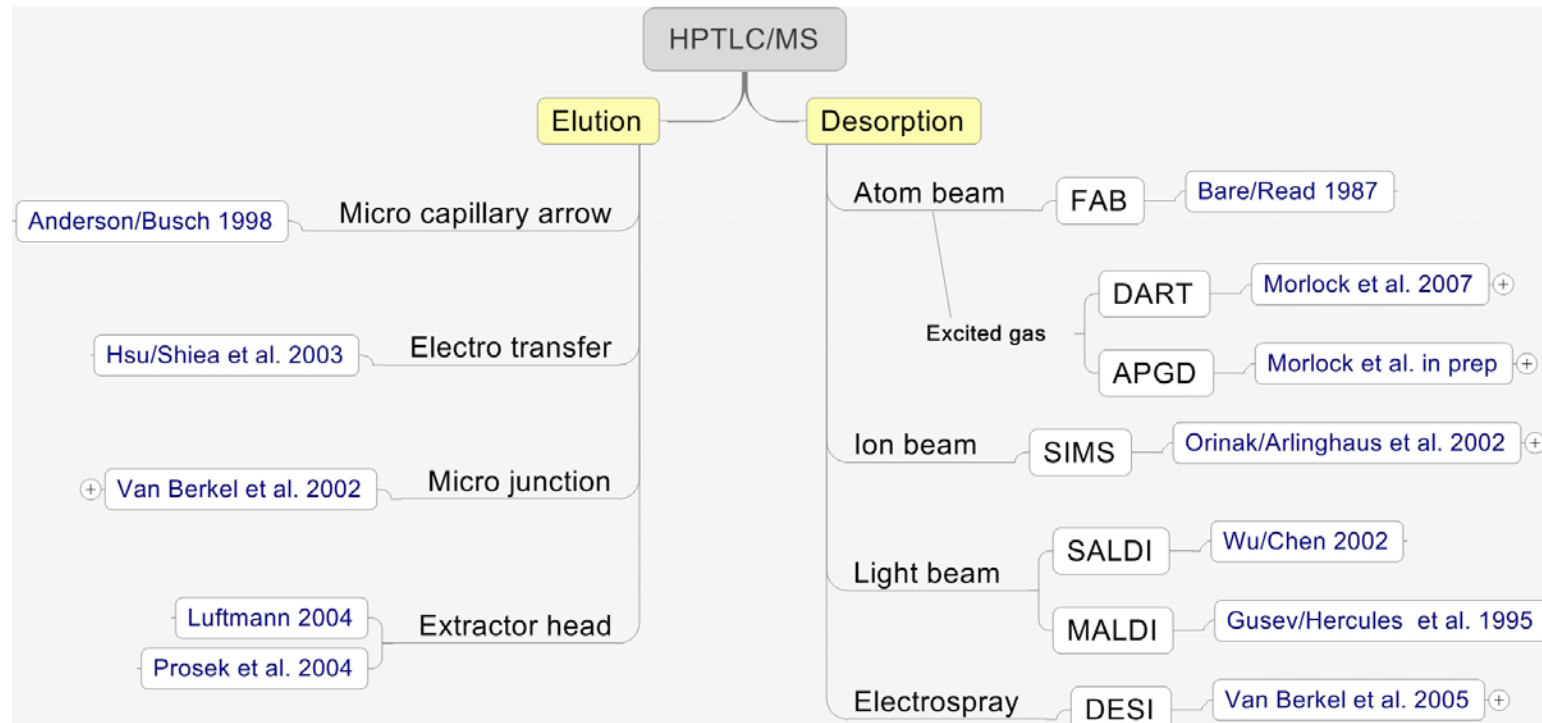
200 pg/zone Harman



20 pg/zone Harman

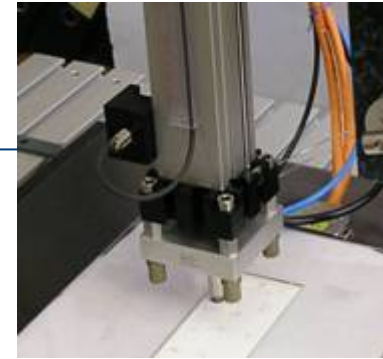


Approaches of HPTLC/MS coupling





Comparison to other interfaces



HPTLC/ESI-MS via R3D3

- ☺
 - ✓ common ionization principle
 - ✓ universally connectable to any LC-MS system given
 - ✓ without adjustments or mass spectrometer modifications
 - ✓ fully automated (hands-free)
 - ✓ no extra plate preparation
 - ✓ whole plate, all layers and carriers
 - ✓ detectability in the pg/zone-range
 - ✓ with good linear range and repeatability without internal standard
 - ✓ withstands validated methods
 - ✓ cost-effective interface for manual positioning available
- ☹
 - ✓ no scan function



Comparison of automated interfaces

Parameter	Precision %RSD	Linear Response r^2
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Quantification **without** internal standard

R3D3/ESI	$\leq 5.6 \%$	0.9973
DESI	$\leq 16.8 \%$	0.95 - 0.98

Quantification **with** internal standard

Micro-junction ESI	$\leq 4.4 \%$	0.9999
SALDI/APCI	7 %	0.9991
MALDI	$\leq 8.9 \%$	0.9969



Special thanks go to ...



**Ute
Jautz**



**Alex
Alpmann**



**Prof. Dr.
Aranda**



Dr. Luftmann



**Prof. Dr.
Schwack**

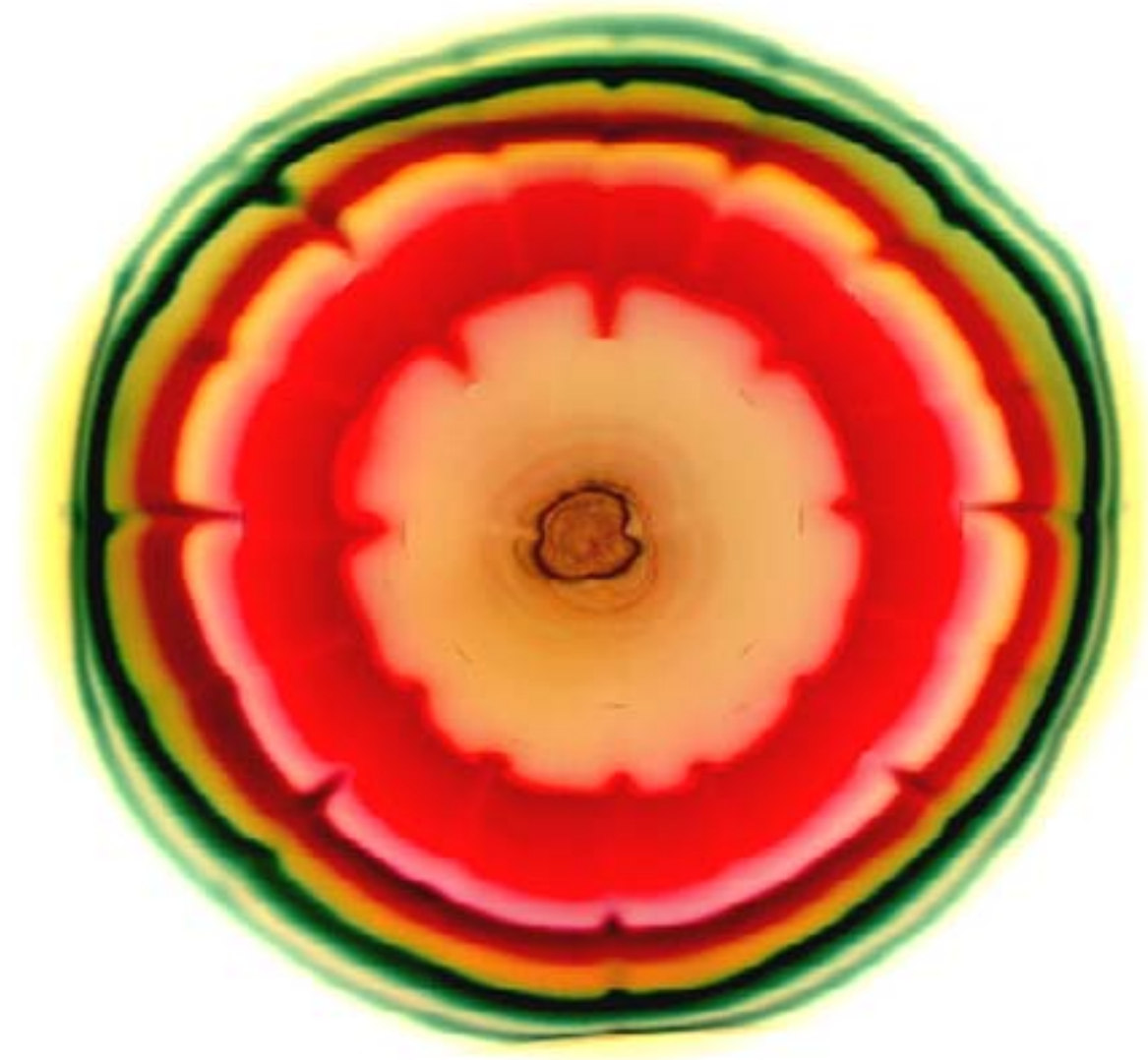
CAMAG, MuttENZ/CH

Merck, Darmstadt/D

Landesstiftung BW (Project No. P-LS-E2/25)



Institute of Food Chemistry
University of Hohenheim, Stuttgart



CHROMart by Drs. Karla und Herbert Halpaap



Why HPTLC?

