Quantitative HPTLC surface analysis by DART-MS scanning

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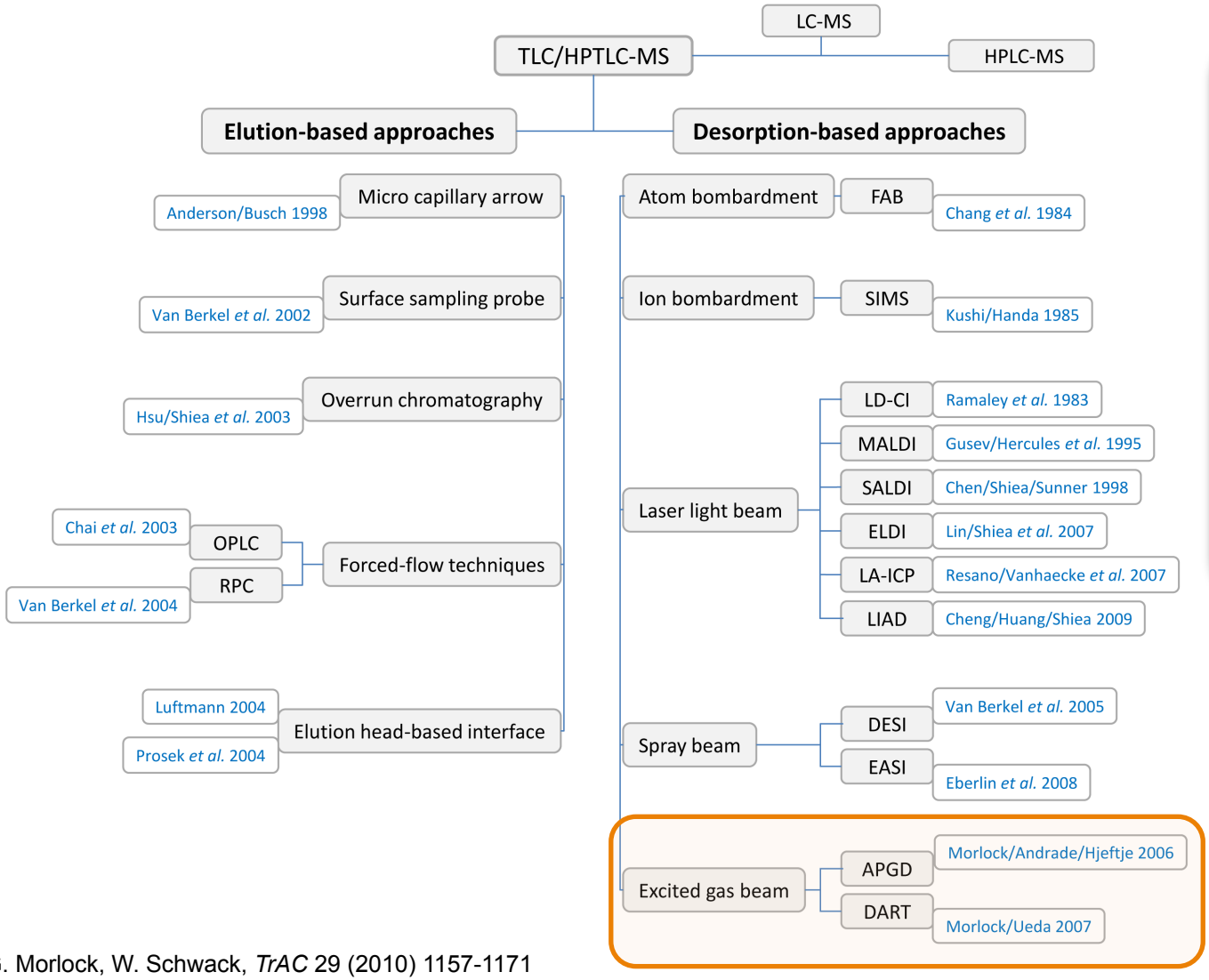
Hyphenations: HPTLC-DART-MS

TLC/HPTLC-MS



DART was first described by Robert B. Cody *et al.* in 2005.

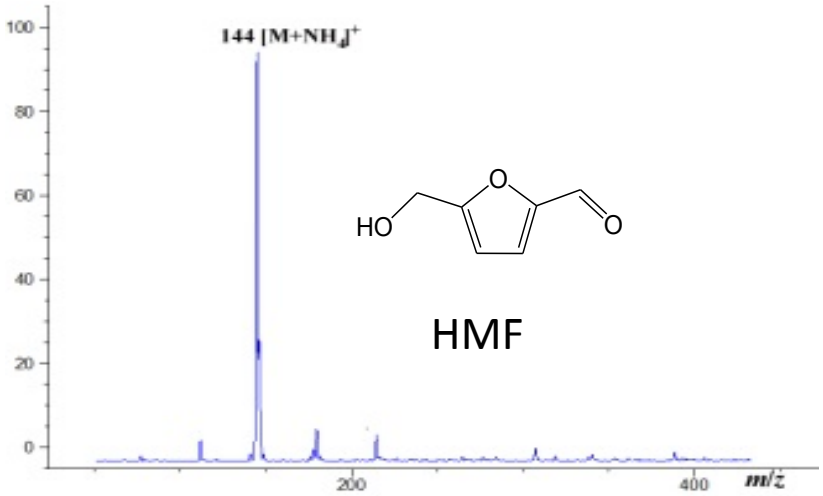
G. Morlock, W. Schwack, *TrAC* 29 (2010) 1157-1171

Hyphenations: HPTLC-DART-MS

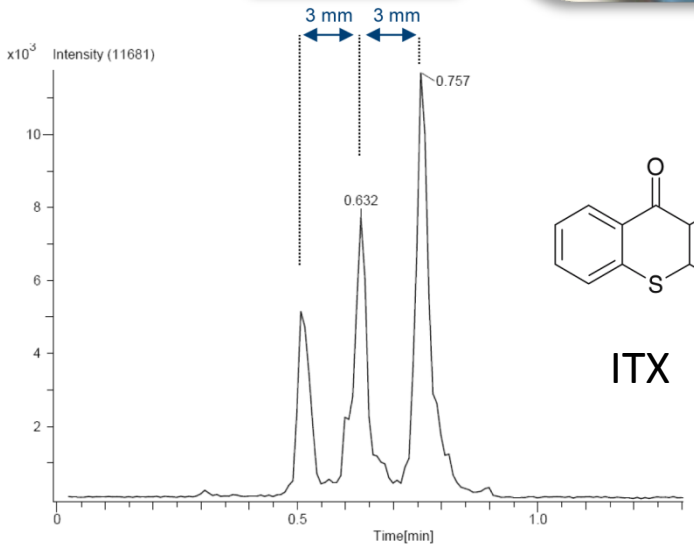
Desorption-based HPTLC-DART-MS

2006 – 2010

- Horizontal ion source angle
- Manual and linear plate introduction
- Low reproducibility
- Low spatial resolution



HMF



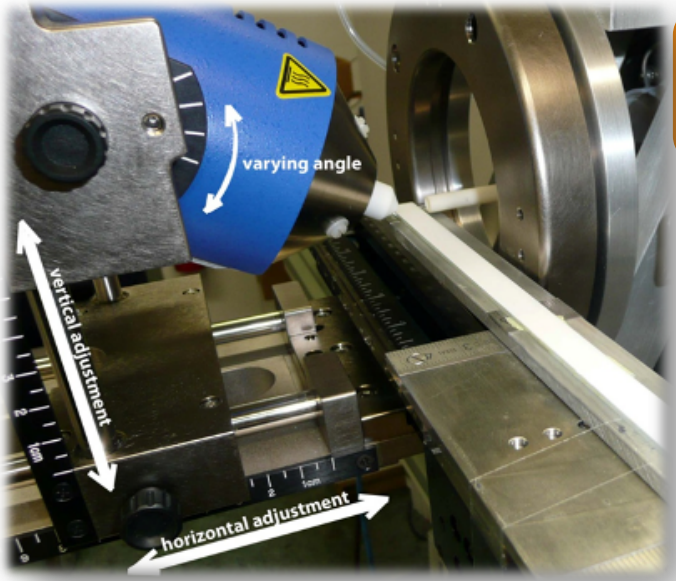
ITX

G. Morlock, W. Schwack, *Anal. Bioanal. Chem.* 385 (2006) 586-595

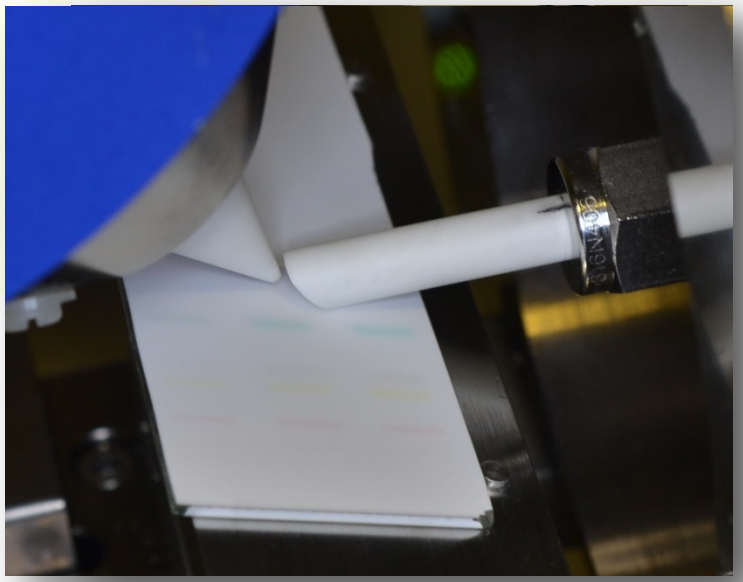
G. Morlock, Y. Ueda, *J. Chromatogr. A* 1143 (2007) 243-251

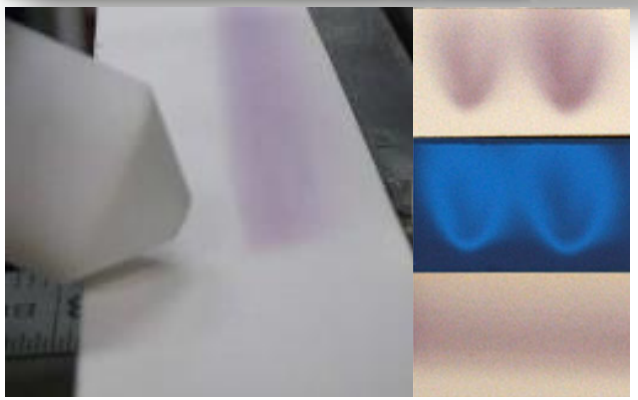
Hyphenations: HPTLC-DART-MS

SVP-A quantitative surface scanning



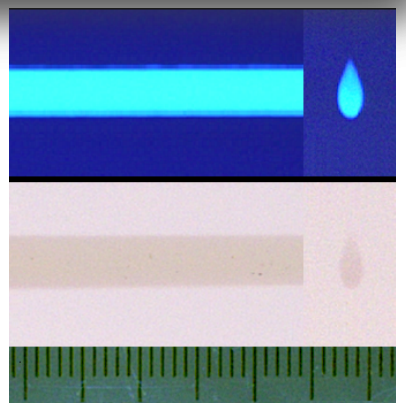
Introduction of DART SVPA in 2009 with controllable sample carrier.





2011 – 2014

- Adjustable ion source angle
- Linear SVPA to angled SVPA-3DSα
- Impact area stabilized as scan track
- High spatial resolution and ion catch

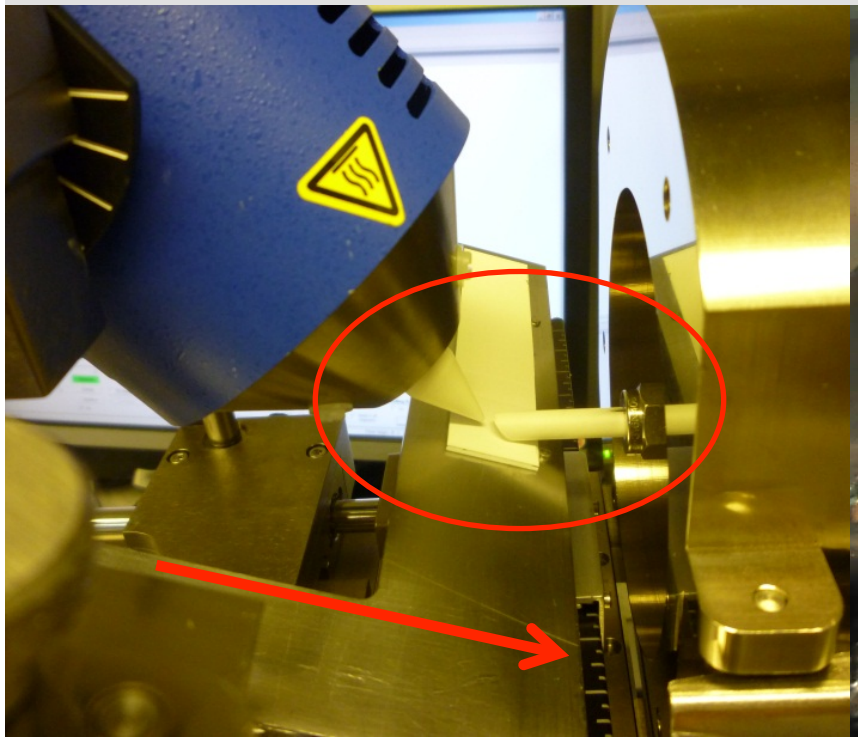


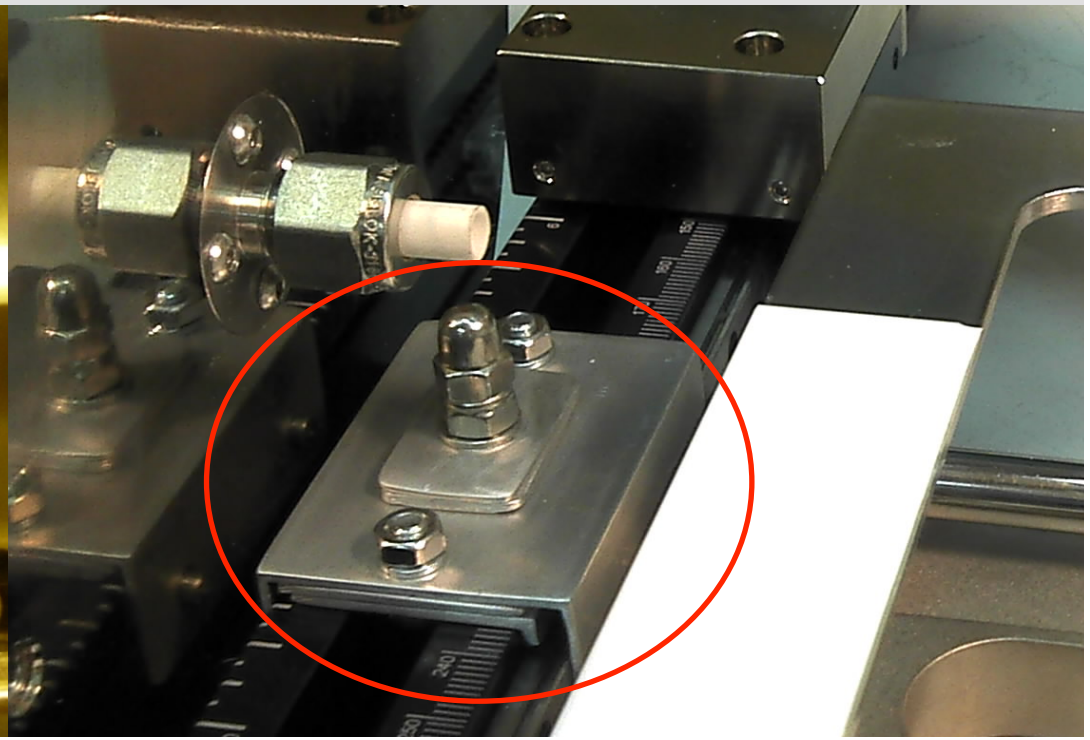
E. Chernetsova, A. Revelsky, G. Morlock, *Rapid Commun. Mass Spectrom.* 25 (2011) 2275–2282

T. Häbe, G. Morlock, in print (2014)

Hyphenations: HPTLC-DART-MS

3 key points

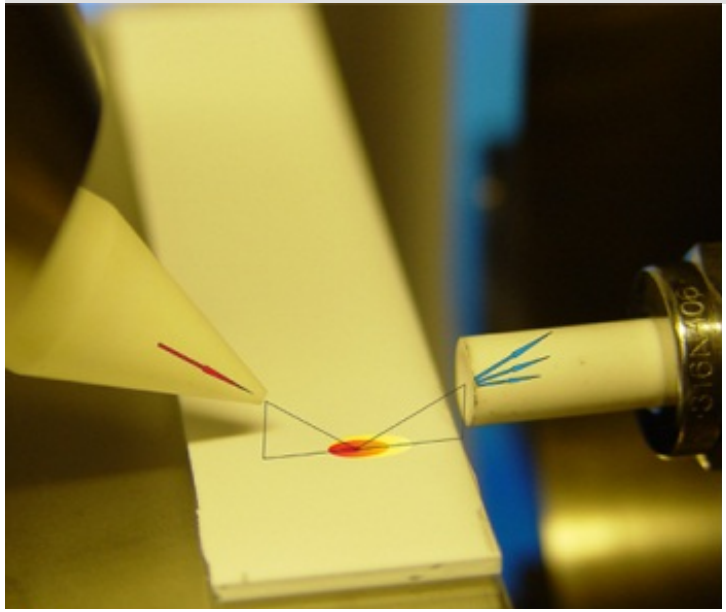




1. Modified **source cap** and **transfer tube** design
2. **Angled y-axis** and **TLC sampling carrier**
3. **Vertical stabilizer** for carrier movements

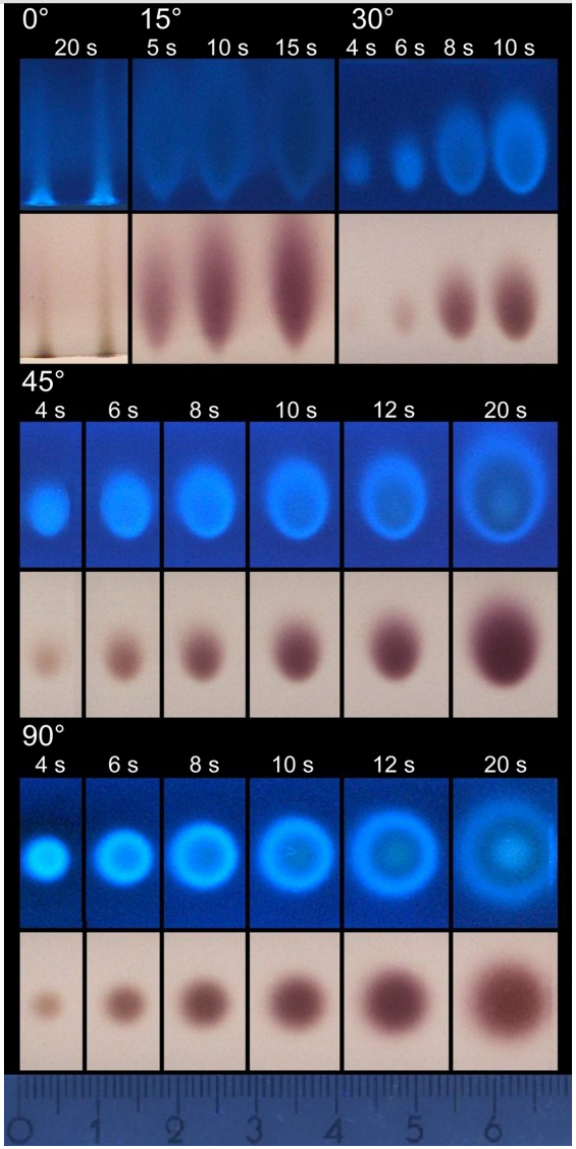
Hyphenations: HPTLC-DART-MS

Impact area and heat distribution



Visualization via heat sensitive HPTLC plate:
gas flow: 3 L/min
gas heater: 300 °C
ion source angles: 0°-90°
exposure time 4-20 s

→ 2-3 mm inner impact zone containing the desorption and ionization region

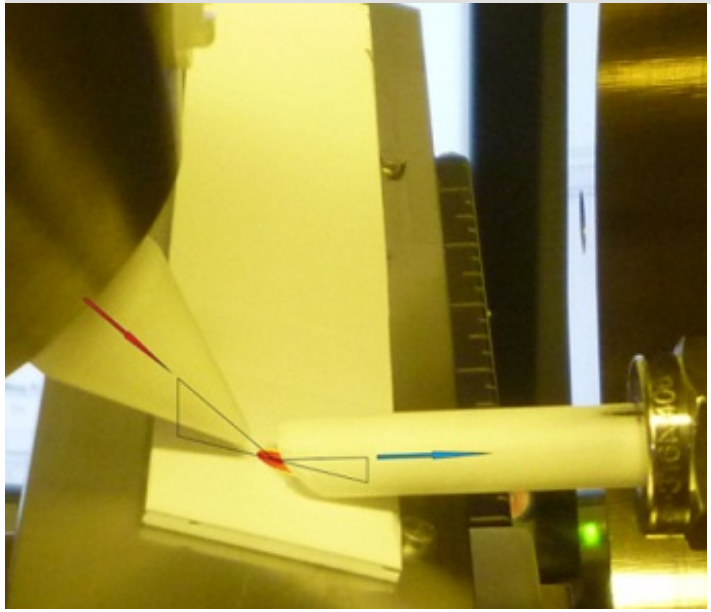


- Modified 1 mm **source cap**
- 40°-**angled ion source**
- Standard transfer tube
- Horizontal 3DS sample carrier

→ **Gas stream scattering** after contact with the impact area

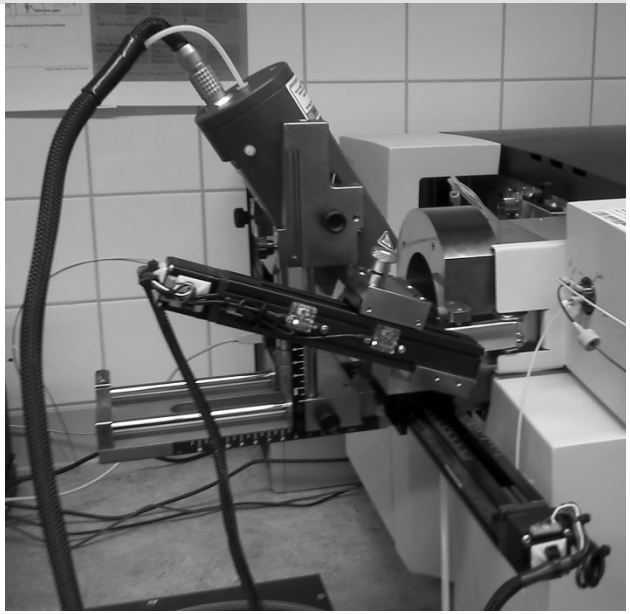
Hyphenations: HPTLC-DART-MS

Impact area with optimized configuration



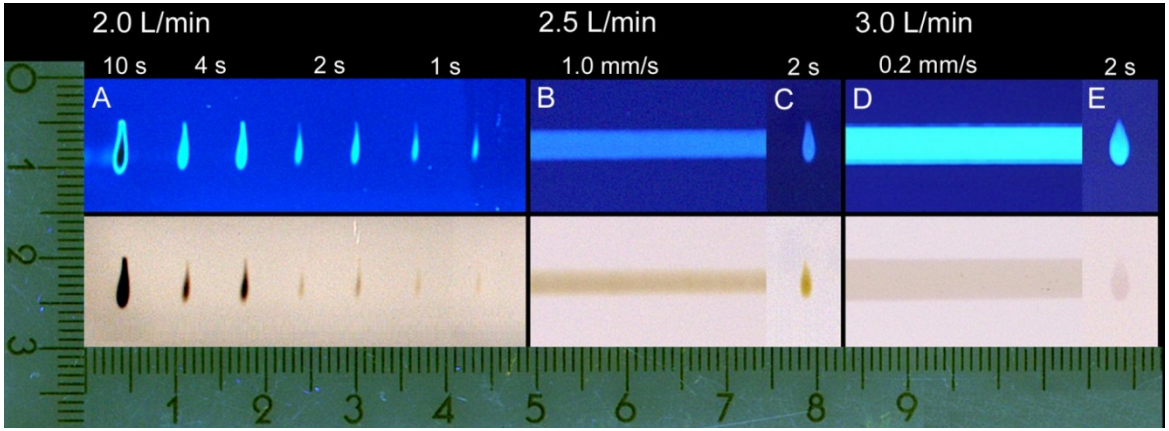
Optimized configuration:
gas flow: 2-3 L/min
gas heater: 400 °C
ion source angle: 60°
carrier angle: 20°
exposure time 1-10 s
Scan speed: 0.2 and 1.0 mm/s

→ Inner impact zone
< 1 x 3 mm



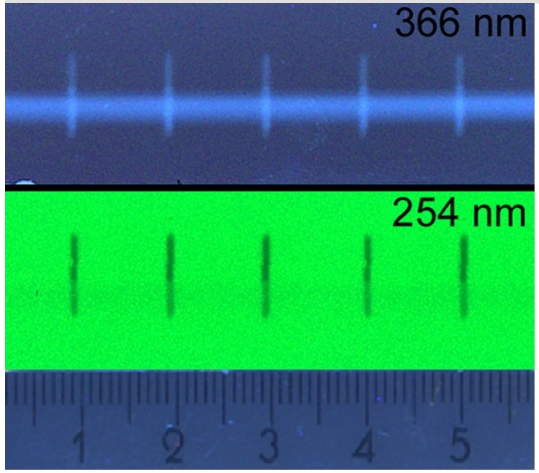
- 60°-**angled ion source**
- Modified **transfer tube nozzle**
- 20°-angled **3DSα TLC carrier**
- **Vertical stabilizer**

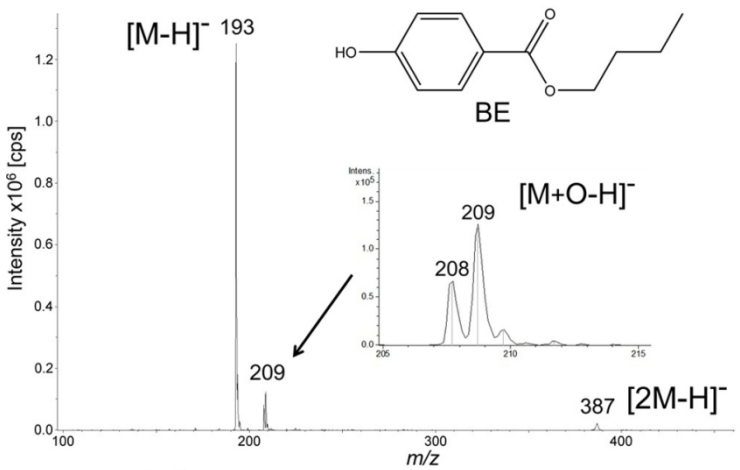
→ **Gas stream focusing** due to lateral air suction **of the transfer nozzle**



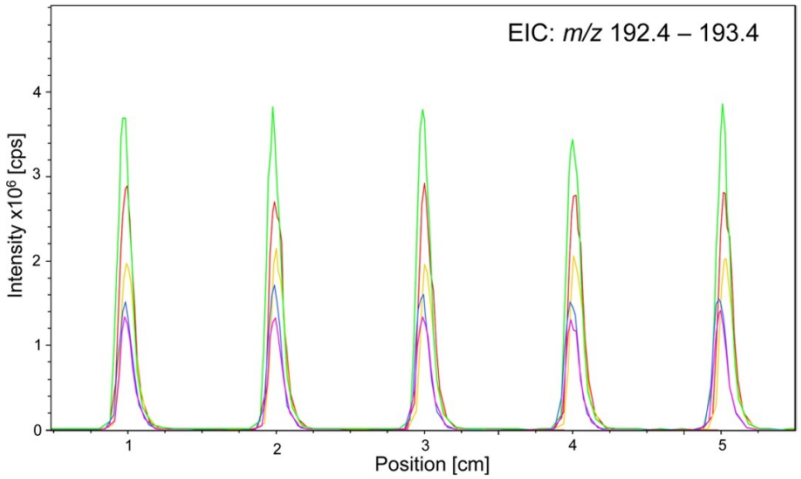
Hyphenations: HPTLC-DART-MS

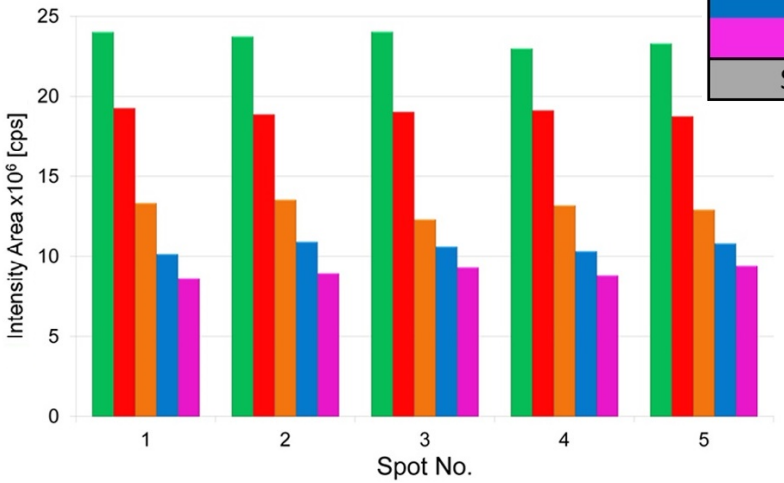
Repeatability results along a track





%RSD (%; n = 5)
1.9
1.1
3.6
3.0
3.7
Mean value: 2.7 ± 1.1
Relative Signal Area (%)
100
80
55
45
38
Signal Decay: 62%

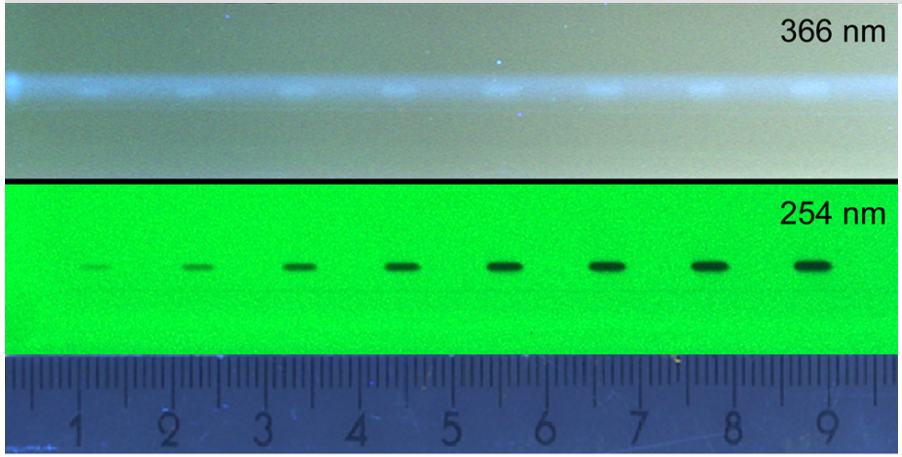


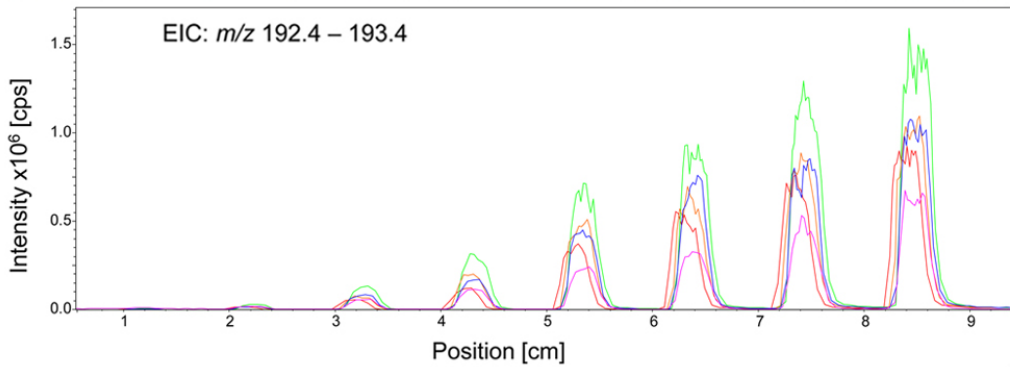


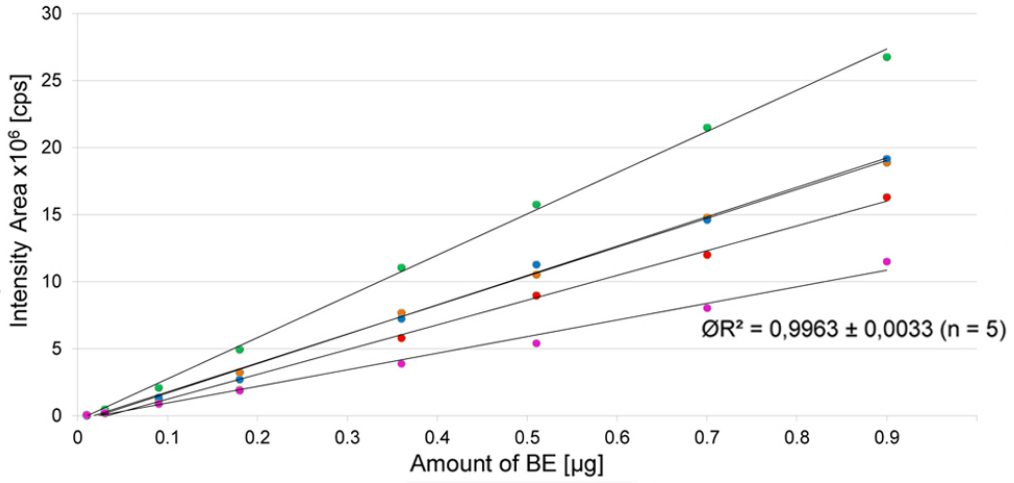
- **5 scan procedures** over 5 bands; each 900 ng/band BE
- Homogenous **signal intensities** and constant **signal decay** for multiple scan runs

Hyphenations: HPTLC-DART-MS

Quantitation results of substance window



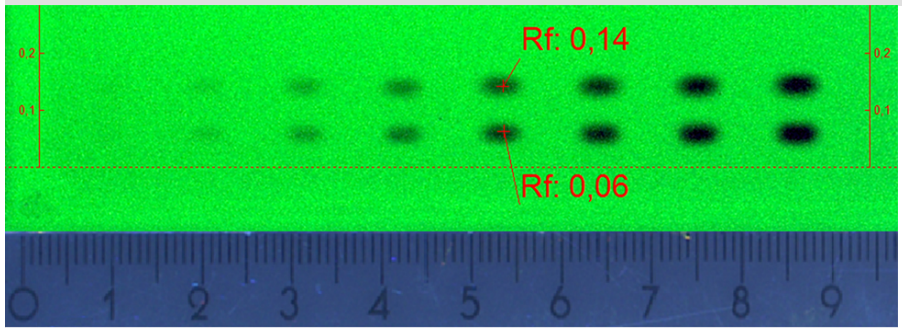




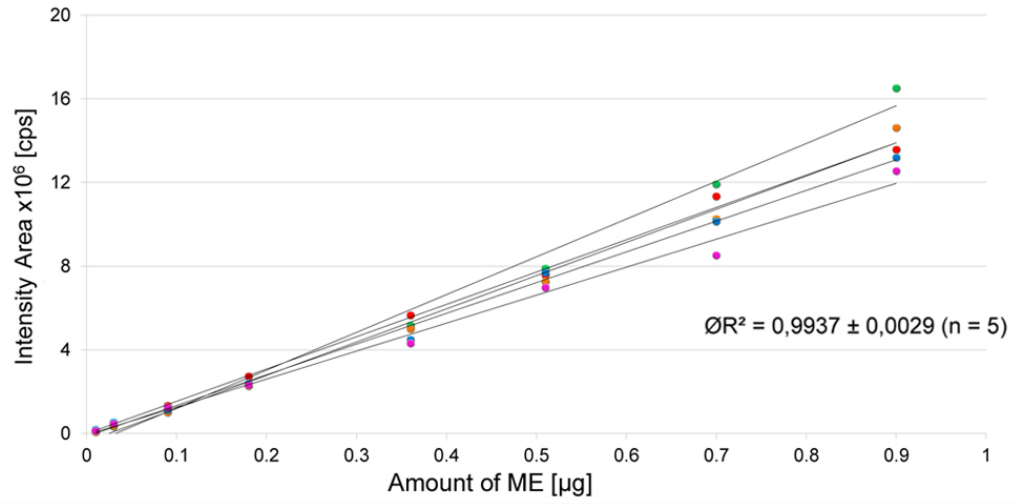
- 8 focused BE bands, **10-900 ng/band**
- Overlaid **EIC chromatograms of 5 plates**
- Good response with determination coefficients of **R² 0.9910 - 0.9990**
- Calibration gradients drift due to deviations of the surface and substance window alignment.

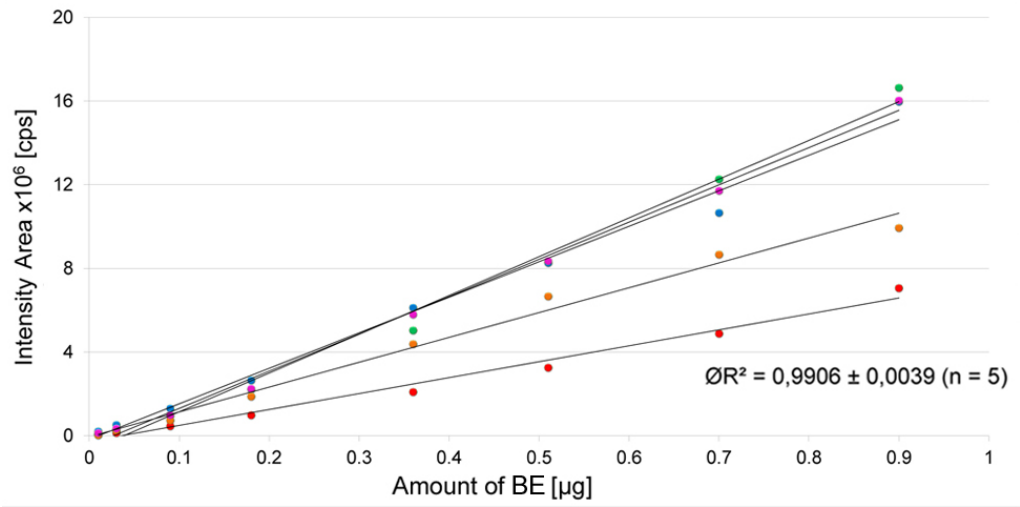
Hyphenations: HPTLC-DART-MS

Quantitation results after chromatography



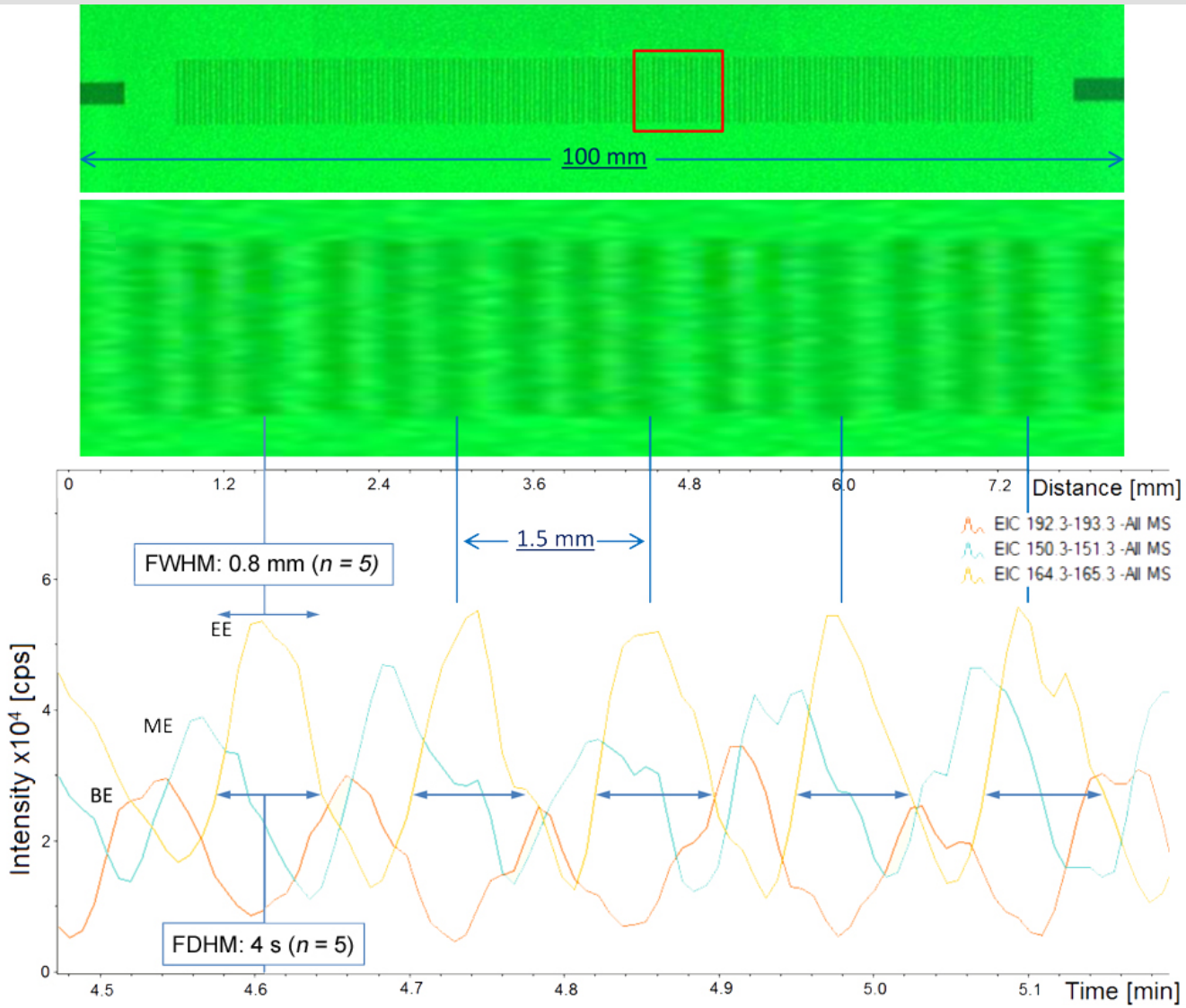
- 8 chromatographed **ME** and **BE** bands, **10-900 ng/band**
- Overlaid **EIC chromatograms** of 2 substance windows **on 5 plates**
- **Good response** with determination coefficients **R² 0.9937 ± 0.0029** and **0.9906 ± 0.0039**





Hyphenations: HPTLC-DART-MS

Spatial resolution

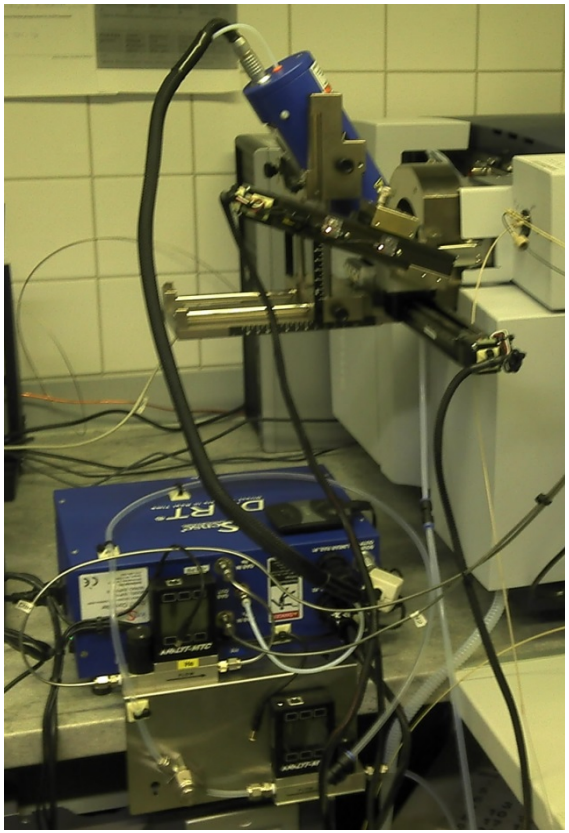


- 3 x 53 band pattern of 30 ng/band **ME, EE** and **BE**
- Overlaid **EIC chromatograms**
- Substance band distance **1.5 mm**
- FWHM: **0.8 mm**
- FDHM: **4 s**

Hyphenations: HPTLC-DART-MS

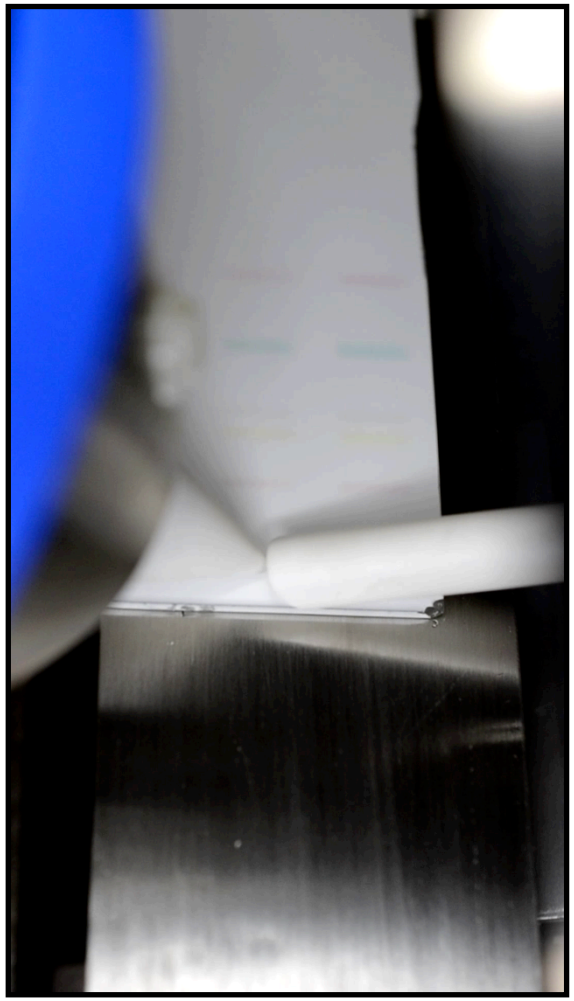
Conclusions

- **Desorption and Ionization** in the flow of the **DART** gas stream
- **No solvents** or **additional Laser** equipment involved
- **Scanning analysis** across **HPTLC track** or **substance window**
- **Semi-destructive**, repeated scans or delay on same zone possible
- Additional information from **DART ionization mass spectra**
- **Quantitation capability** for a wide range of analytes
- **High sensitivity** for analytes adsorbed or dissolved in a matrix
- **No** automated **track positioning** and **ion source control**
- **No ion concentration** from transfer nozzle **to MS inlet**



Hyphenations: HPTLC-DART-MS

Our acknowledgements



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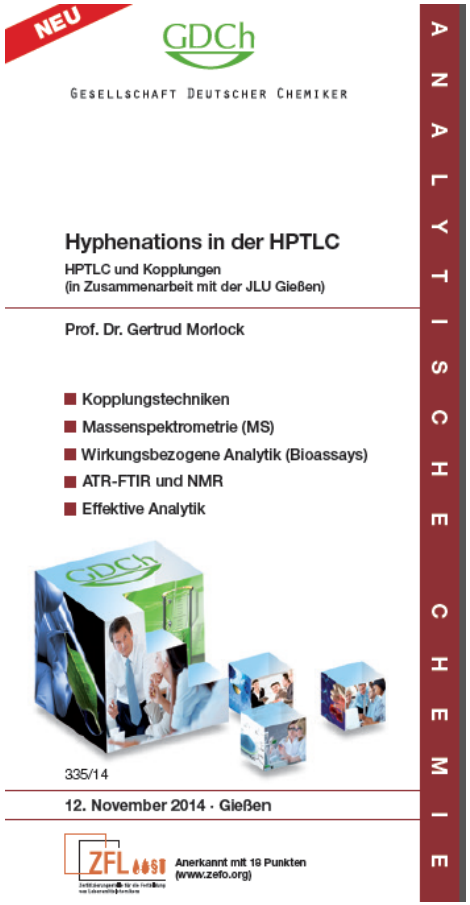

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