Office Chromatography:
Precise sample application on miniaturized phases, image evaluation and DART-MS scanning

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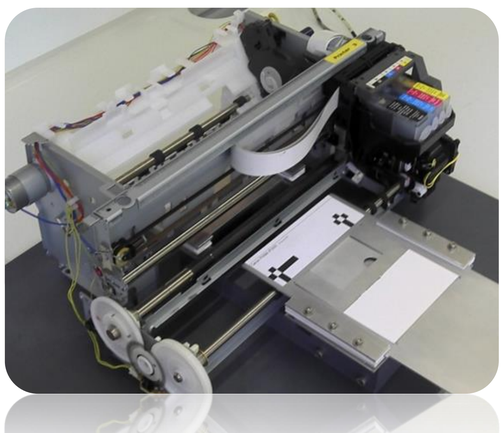
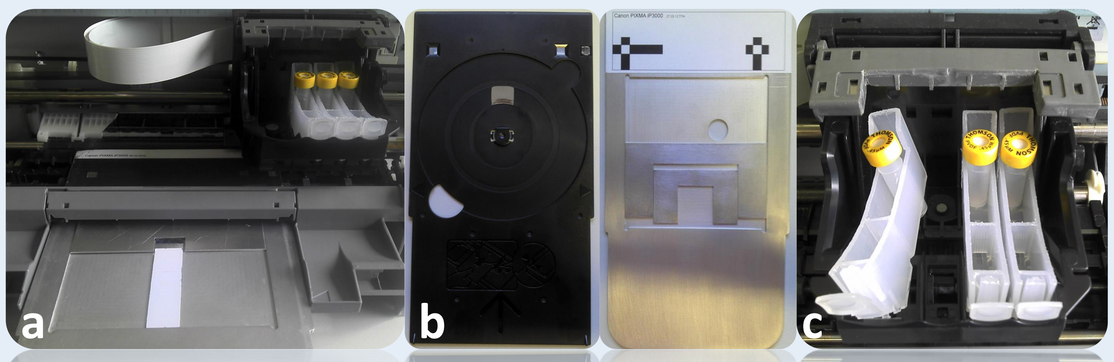


Highlights

- *Technical modifications of an inkjet printer for precise and quantitative HPTLC sample application*
- *Up-to-date miniaturized phases as application substrate and chromatographic layer*
- *High resolution scanner and picture evaluation software for cost-efficient quantitation*
- *Desorption-based UTLC-MS of dyes on a M-CNT-templated ultrathin layer*
- *Printing of finely graduated scales to determine the spatial resolution of DART-MS*

Why modification of the printer?

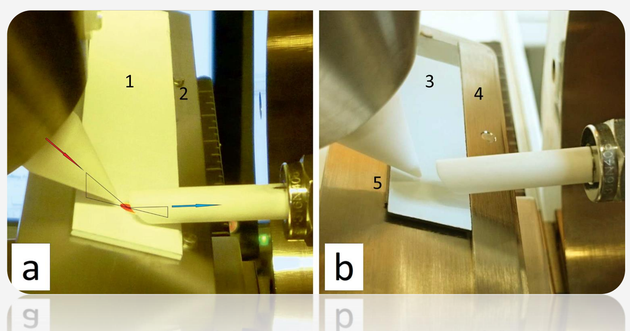
- ✓ *Potential for inexpensive online UTLC*
- ✓ *Current instrumentation cannot handle UTLC*
- ✓ *Green chemistry (quantitatively applied nL-/µL-volumes only)*



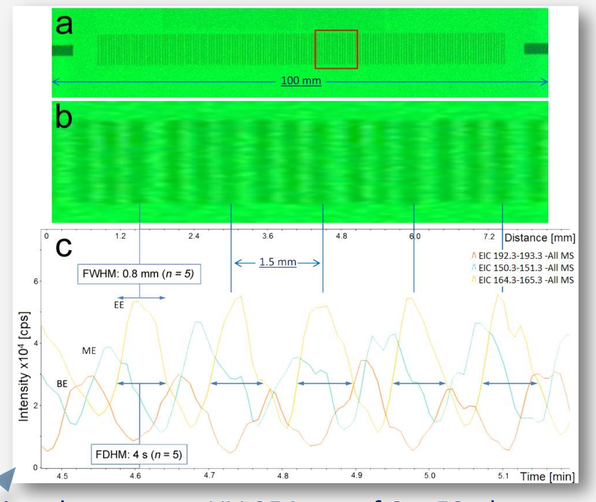
Modified Canon Pixma iP3000 printer with the original tray guide, removed paper feed assembly, dismounted sensors and transport slide cut out for contactless movement of all plate formats (a); original CD-tray and custom-built plate tray (b); print head carrier with Thomson SINGLE StEP® Filter Vials as sample reservoir in cartridge adapters (c)[7].

Why to improve the DART interface for scanning?

- ✓ *Improved ion guidance into the MS orifice → increased reliability*
- ✓ *Contactless scanning and MS evaluation of miniaturized UTLC formats like monolithic[1,2], electrospun[3,4] and nanostructured layers[5,6]*



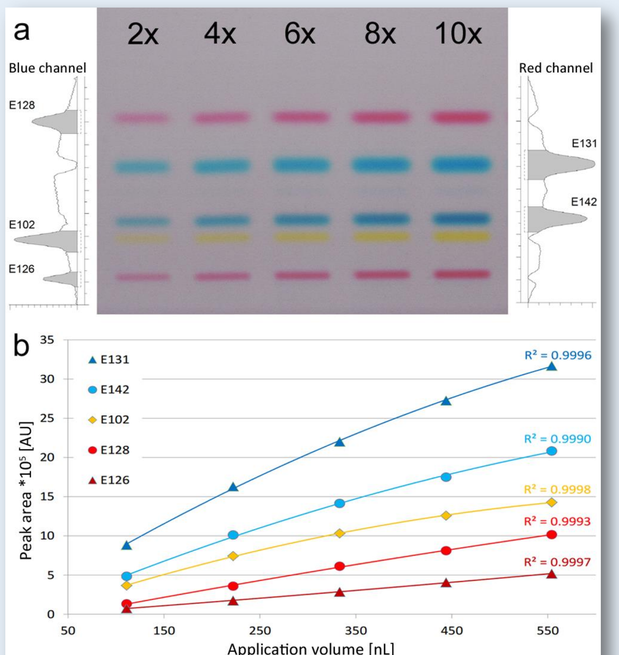
Scanning MS with a substantially modified DART-SVPA-3DS[8]: HPTLC plate (1) aligned at arresters (2) (a) and M-CNT UTLC plate (3) on spacers for scan lane (4) and height (5) adjustment (b).



Spatial resolution of the DART interface: printed pattern at UV 254 nm of 3 x 53 alternating bands (0.25 x 6.0 mm, each 30 ng/band) of methyl/ethyl/butyl parabenes (a) and MS scan along the region of interest (b) showed EIC signals with FWHM of 0.8 mm (c, *n* = 5).

Is this print application reliable?

- ✓ *Highly quantitative, good correlation*

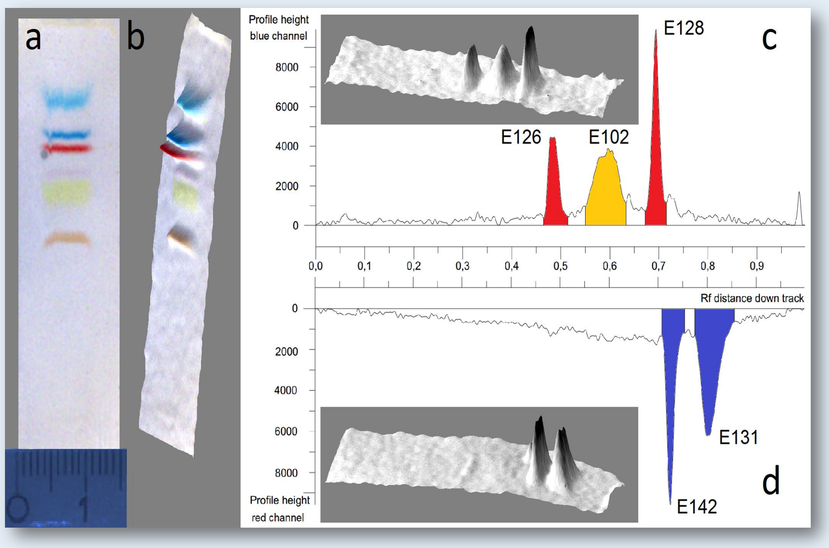


C	hR_F	amount [ng]	mean *RR* [%]	*%RSD* [%]	mean R^2
E126	7	67 - 332	103	4.0	0.9977 ± 0.0033
E102	16	44 - 222	101	3.4	0.9995 ± 0.0008
E142	20	22 - 111	105	6.5	0.9991 ± 0.0007
E131	32	44 - 222	101	2.9	0.9990 ± 0.0007
E128	42	44 - 222	103	7.2	0.9995 ± 0.0005

Calibration curves for 5 food dyes obtained via repeated printjobs and HPTLC analysis; detection via high-resolution flatbed scanner (CanoScan 9000F, Canon) and evaluation with VideoScan (CAMAG) using color channel filters (a, *n* = 4).

Example for miniaturized UTLC: Analysis of water-soluble food dyes

- ✓ *Proof-of-principle worked*

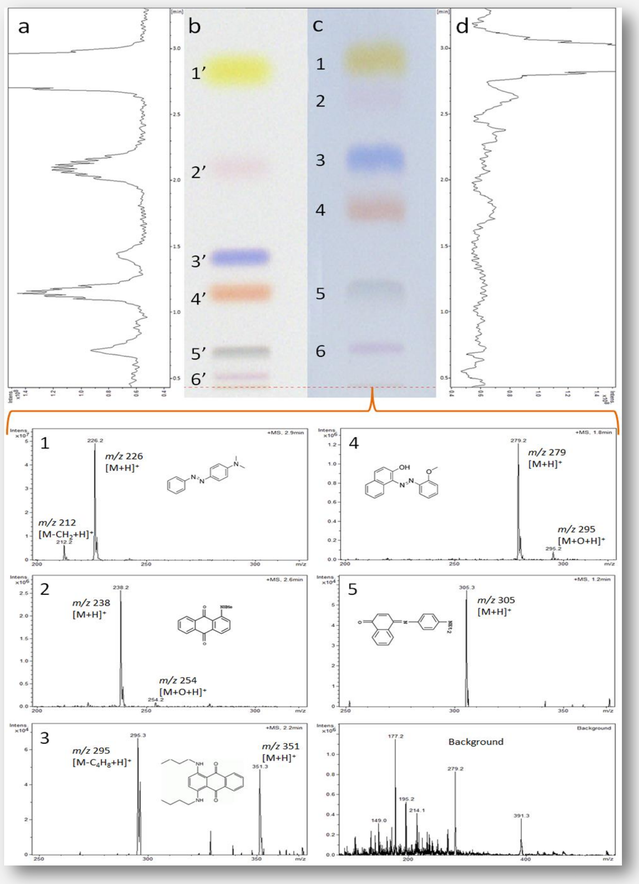


Separation on novel M-CNT UTLC layers of 100 nL hydrophilic dye mixture applied by the modified printer (a), documentation via TLC Visualizer (CAMAG), 3D image profile via Image J[6] (NIH) (b); evaluation via VideoScan (CAMAG) for blue (c) and red filter channel (d).

- E126 (hR_F 48; 30 ng): red
- E102 (hR_F 59; 20 ng): yellow
- E128 (hR_F 69; 30 ng): red
- E142 (hR_F 73; 10 ng): blue
- E131 (hR_F 81; 20 ng): blue

HPTLC-MS versus UTLC-MS

- ✓ *Example: Lipophilic dye mixture*
- ✓ *On both, MS spectra obtainable*
- ✓ *HPTLC-DART-MS more sensitive than M-CNT-UTLC-DART-MS (tight hedges)*



Scanning DART-TIC-MS of separated lipophilic dyes on HPTLC plate (a, b) and UTLC plate (c, d).

References [1]H. Hauck *et al.*, J Planar Chromatogr 14, 234 (2001). [2]A. Frolova *et al.*, J Sep Sci 34, 2352 (2011). [3]X. Fang, S. Olesik, Anal Chim Acta 830, 1 (2014). [4]J. Clark, S. Olesik, Anal Chem 81, 4121 (2009). [5]L. Bezuidenhout, M. Brett, J Chromatogr A 1183, 179 (2008). [6]S. Kanyal *et al.*, J Chromatogr A 1404, 115 (2015). [7] T. Häbe, G. Morlock, J Chromatogr A, in print (2015). [8]T. Häbe, G. Morlock, Rapid Commun Mass Spectrom 29, 474 (2015).

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