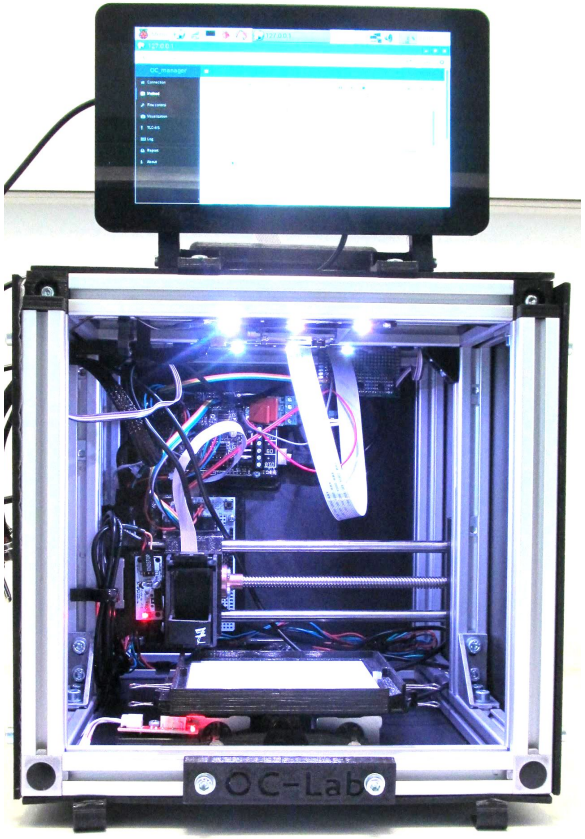
JLU Giessen

Food Science

JLU

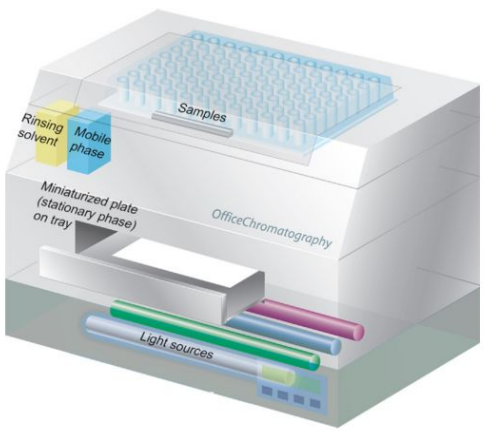
Office Chromatography

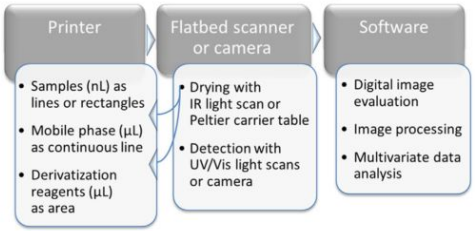


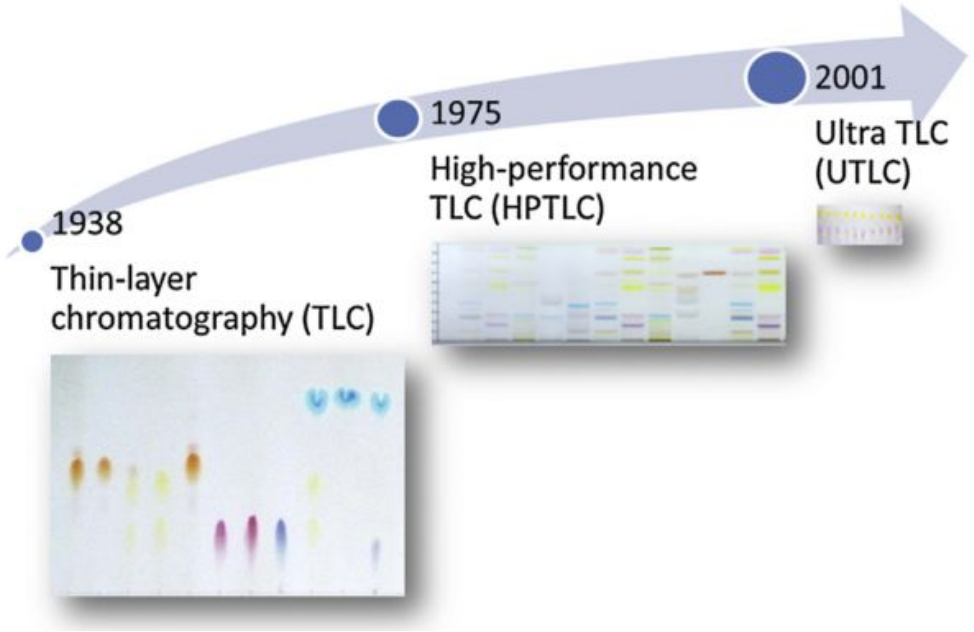
Dimitri Fichou and Gertrud Morlock
Justus Liebig University Giessen

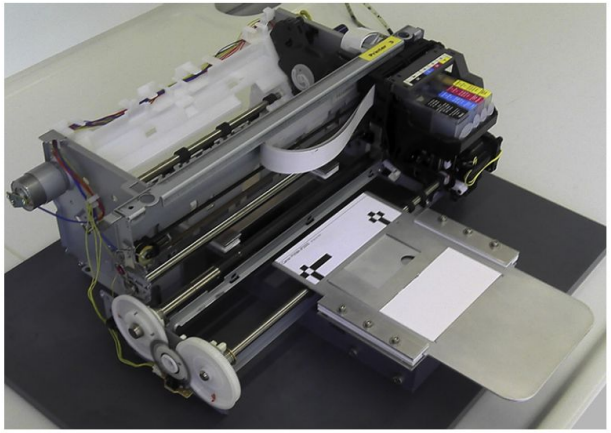
Office Chromatography

- Introduced in 2010 [1]
- Focus on miniaturization of
  - layers (UTLC)
  - apparatus (all steps in one device)
- Inspired from Print and Media Technologies [2]
  - Inkjet printing
  - Scanner





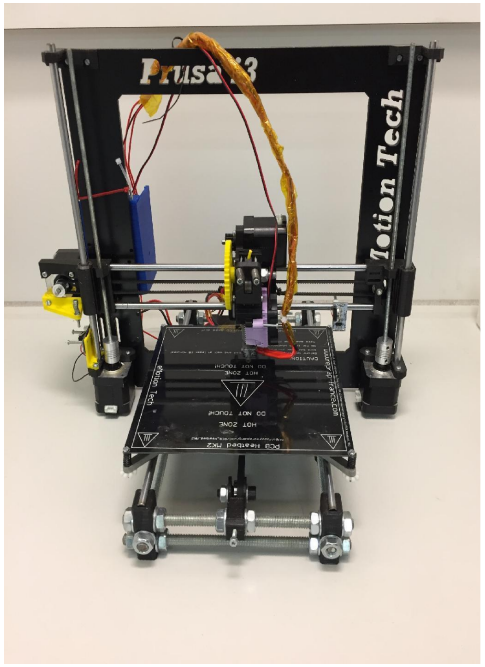


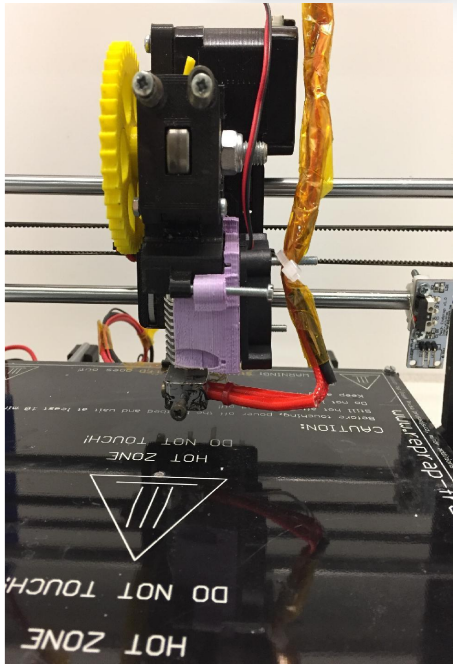


[1] Morlock, G. Anal. Chem. 2010, 82, 2940-2946
[2] Häbe, T. T.; Morlock, G. J. Chromatogr. A 2015, 1413, 127-134

Open source tools

Programming tools



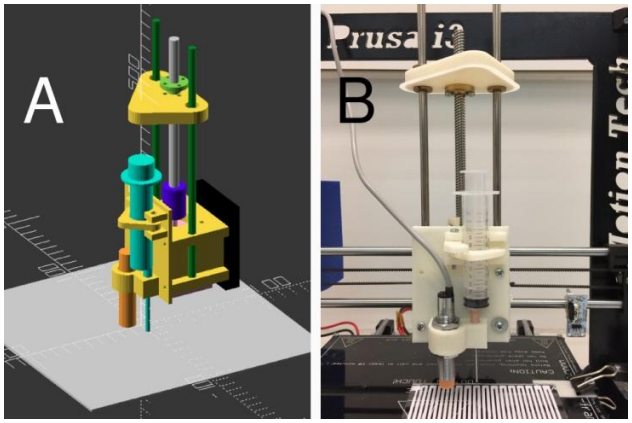


Prusa i3 printer

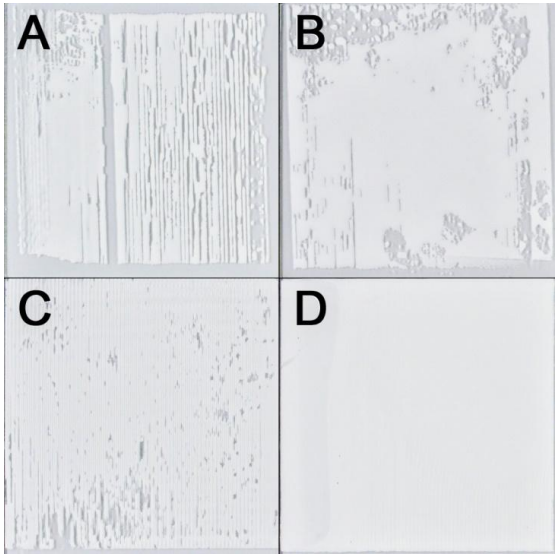
Internet of things with Raspberry pi

Arduino mega 2560 and Ramps 1.4

Layer printing: 3D printer modification

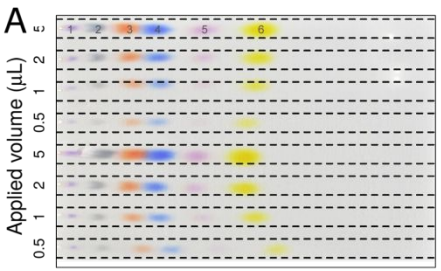


Computer-assisted design and printed version of the prototype

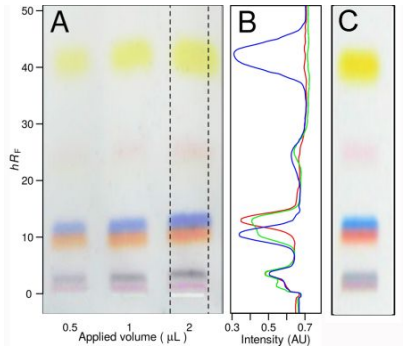


Problems encountered

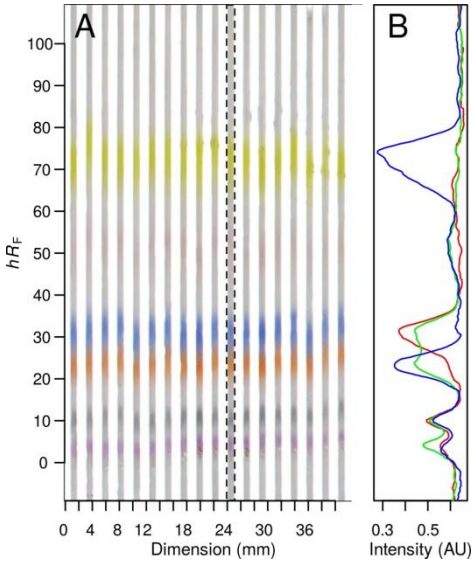
Software pipeline



Semi quantitative analysis



HPTLC silica gel reprinted for spray application

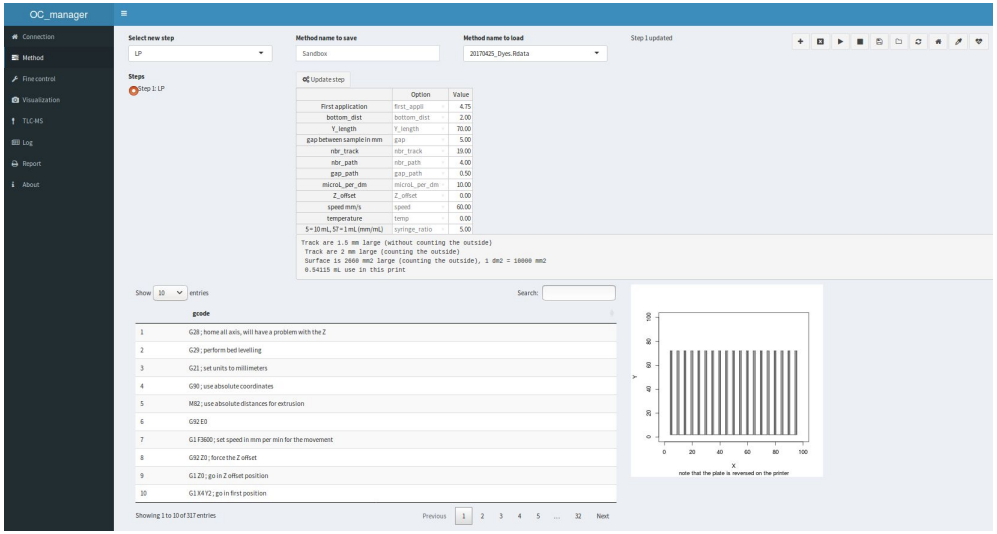


Channel pattern: 40 tracks/10 cm

Exotic pattern

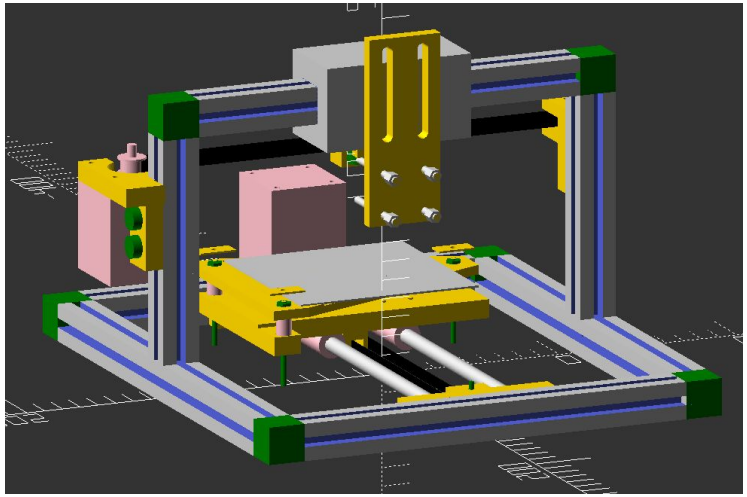
Fichou, D., Morlock, G. Anal. Chem. 2017, 89, 2116–2122

Layer printing: dedicated apparatus and software

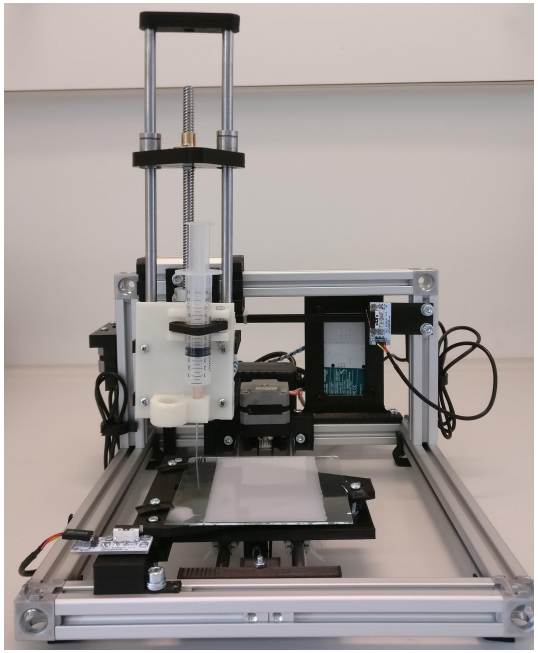


OC manager: control software

	Option	Value
First application	first_appli	4.75
bottom_dist	bottom_dist	2.00
Y_length	Y_length	70.00
gap between sample in mm	gap	5.00
nbr_track	nbr_track	19.00
nbr_path	nbr_path	4.00
gap_path	gap_path	0.50
microL_per_dm	microL_per_dm	10.00
Z_offset	Z_offset	0.00
speed mm/s	speed	60.00
temperature	temp	0.00
5 = 10 mL, 57 = 1 mL (mm/mL)	syringe_ratio	5.00

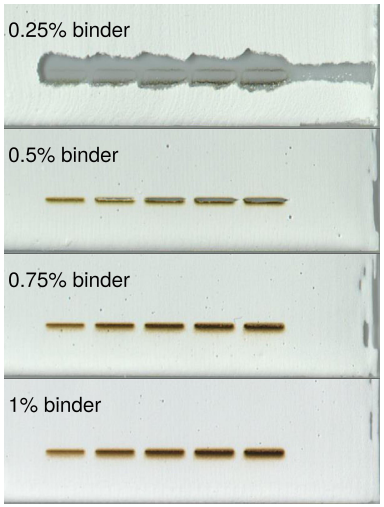


Computer-assisted design of the prototype

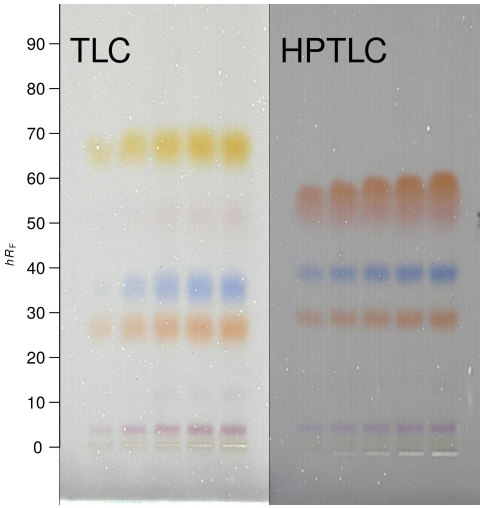


Printed prototype

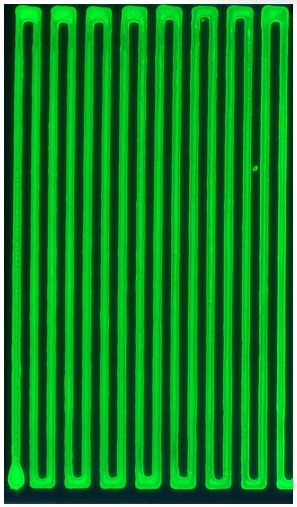
Layer printing: New slurry



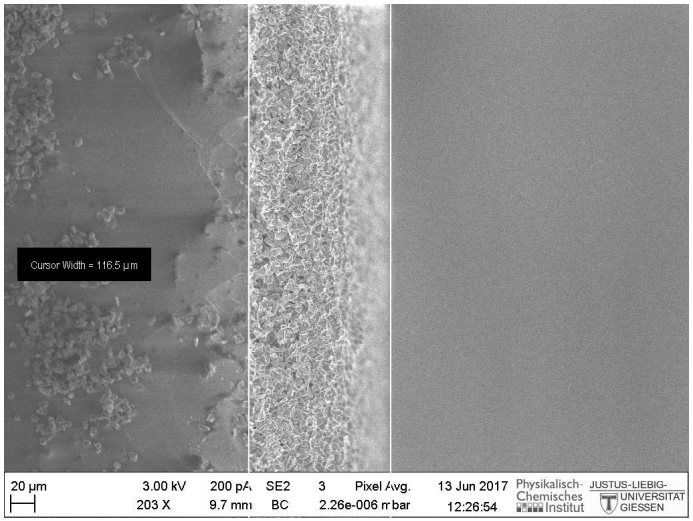
Organic binder



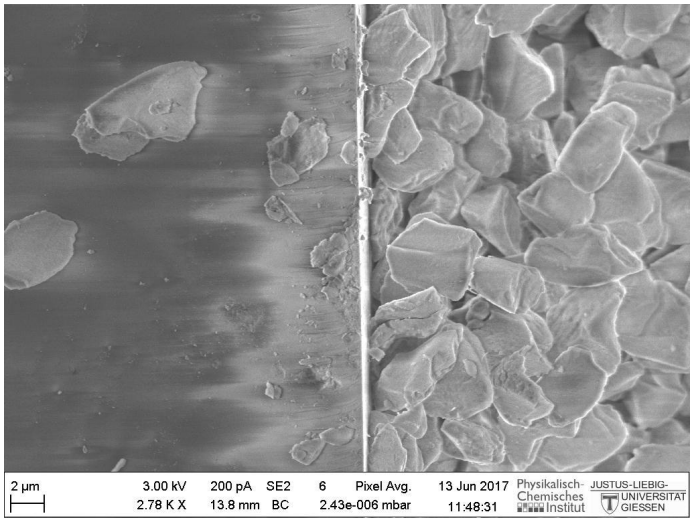
TLC *versus* HPTLC particles



Fluorescent indicator

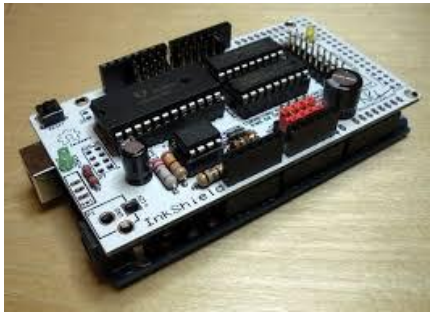


Layer thickness: 120 µm

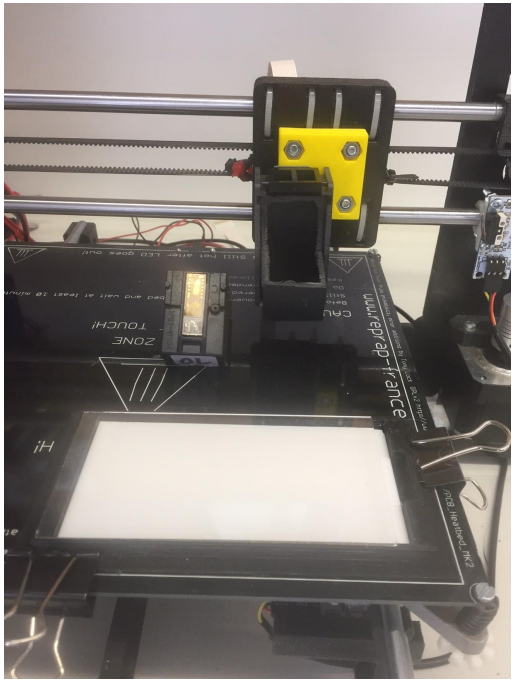


Glass-layer border

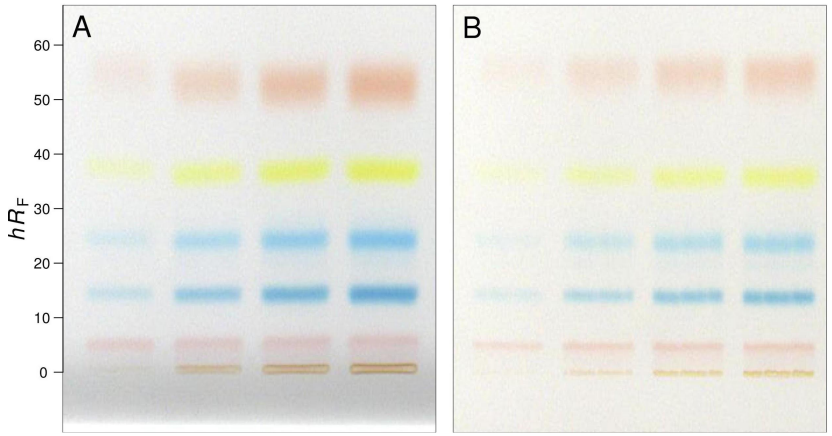
Inkjet printing: 3D printer modification



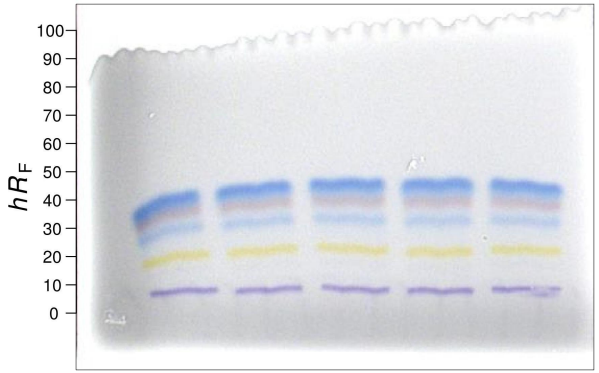
InkShield board



Cartridge holder and elution chamber

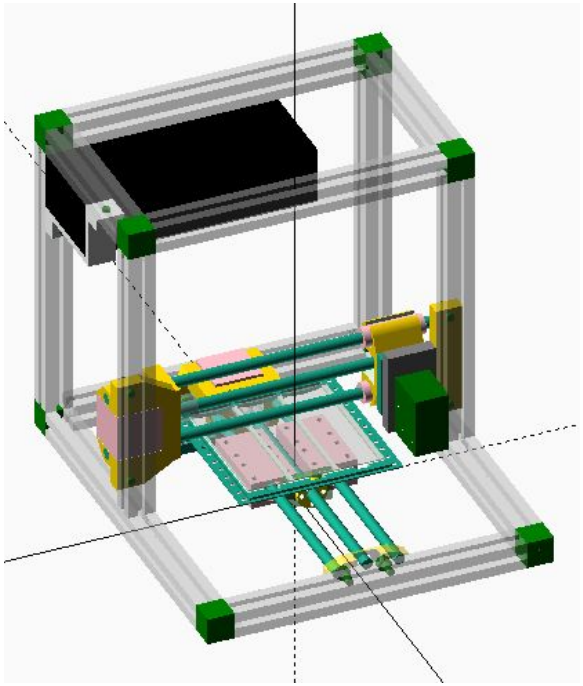


Dyes separation: comparison syringe (A) and inkjet (B) application

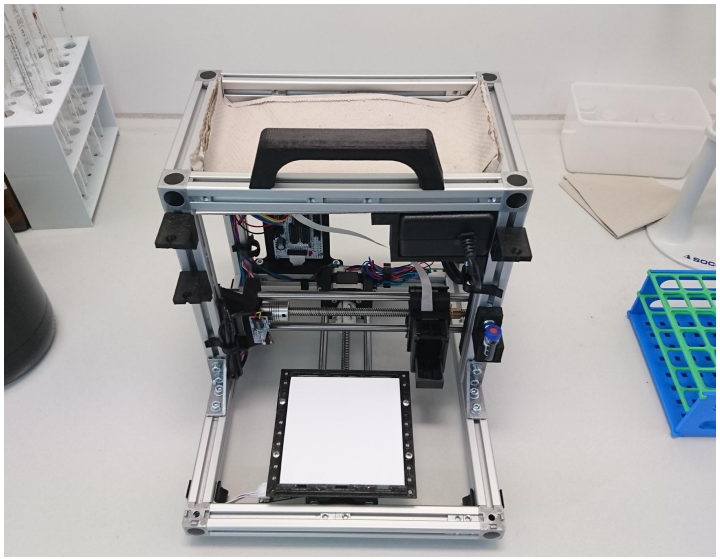


Dyes separation: printing of samples and mobile phase

Inkjet printing: dedicated apparatus and software



Computer-assisted design of the prototype



First OC prototype

Update step

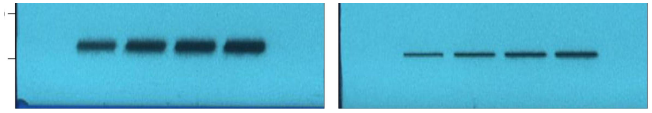
	Option	Value
Distance to lower edge	dist_bottom	6.00
First application position	dist_gauche	10.00
Band length	band_length	6.00
Distance between track	gap	2.00
Numbre of band	nbr_band	4.00
Speed (mm/s)	speed	10.00
I (number of firing)	I	10.00
L (pulse delay <20)	L	5.00
Wait between path (s)	wait	5.00
Used Nozzle	nozzle	12.00

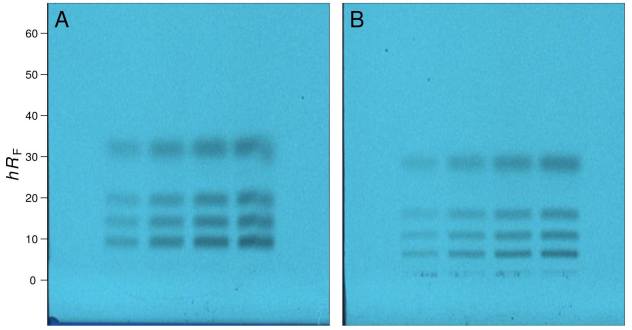
	Band	Vial	Repeat	Content	Use
1	1	1.00	10	pesticide in H2O 25pc 100 ppm	☑
2	2	1.00	20	pesticide in H2O 25pc 100 ppm	☑
3	3	1.00	30	pesticide in H2O 25pc 100 ppm	☑
4	4	1.00	40	pesticide in H2O 25pc 100 ppm	☑

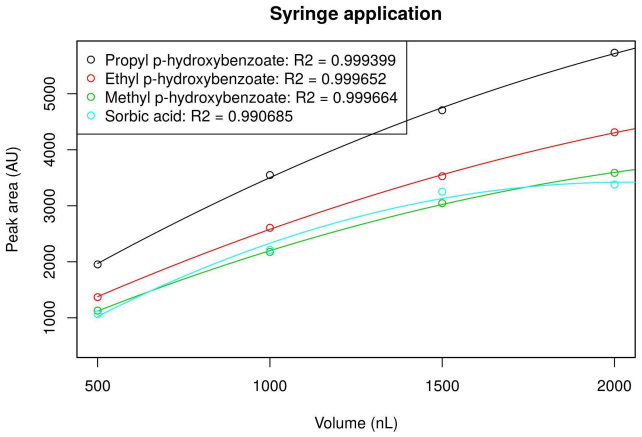
OC manager: control software

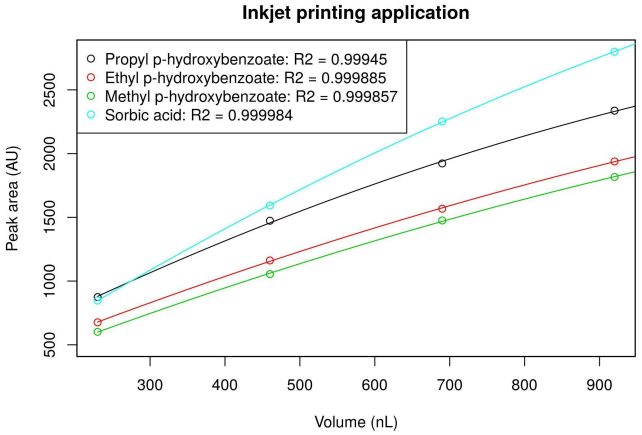
Inkjet printing: Preservatives analysis

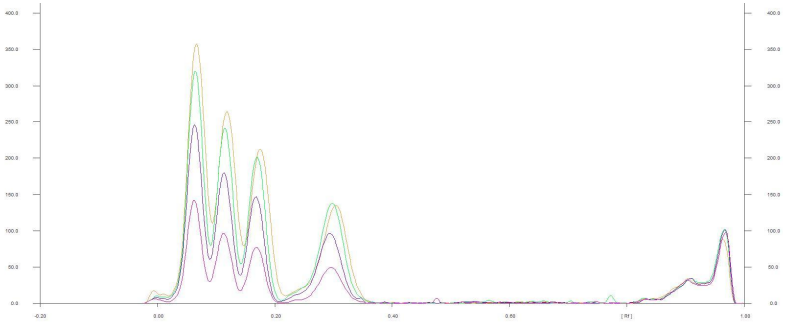
- Sample diluted in MeOH/H_2O 1:3
- Stationary phase: Silica gel 60 CN F_{254} s
- Inkjet application: 2300 - 9200 drops per track
- Mobile phase: EtOH/H_2O/AcOH 14:40:0.1
- Absorption measurement at 257 nm

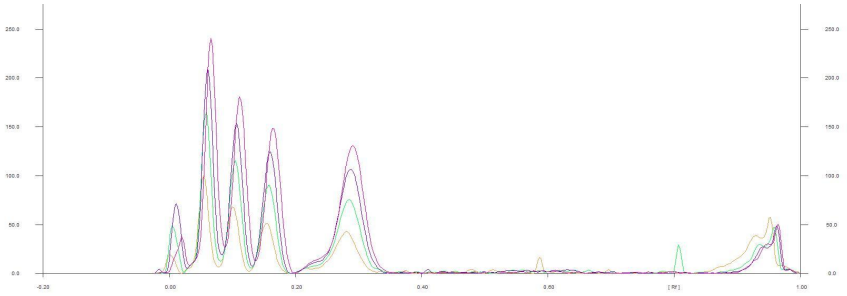




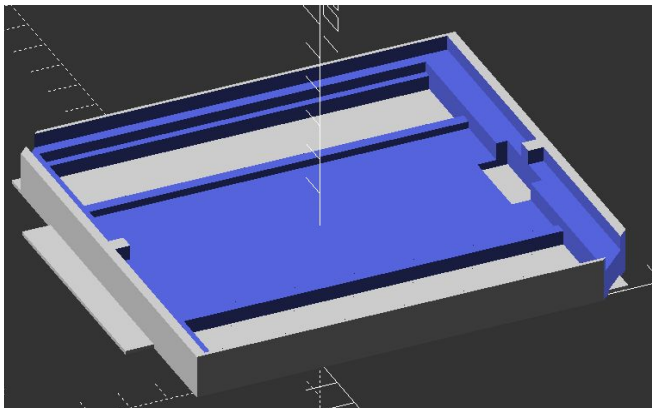




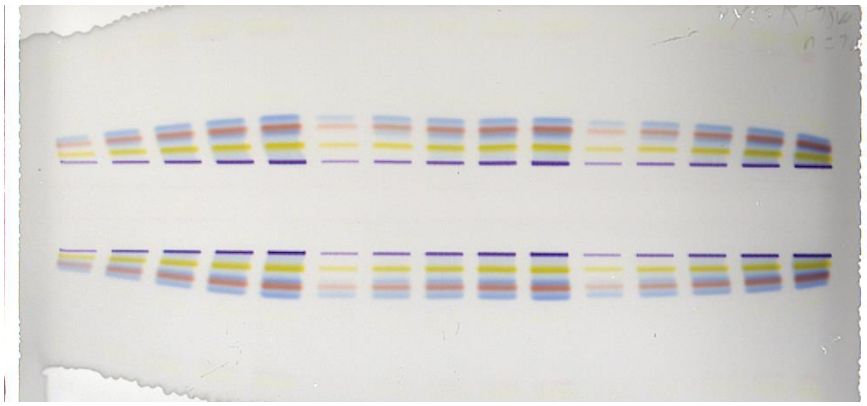




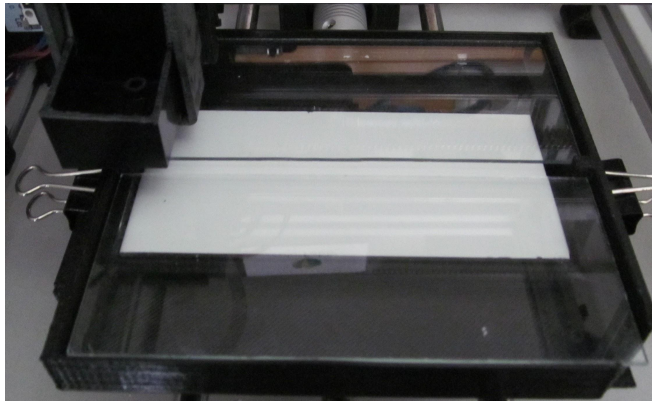
Inkjet printing: Mobile phase printing



Computer-assisted design of the development chamber



Dyes: MeOH/H_2O 5% Na_2SO_4 3:4
Silica gel 60 RP 18 W

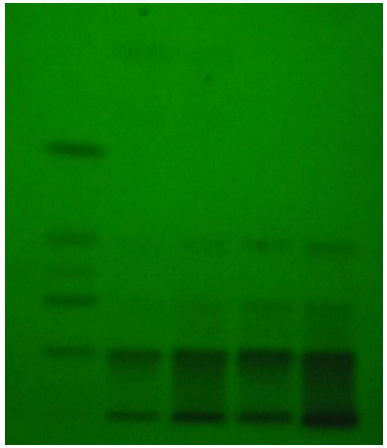


Printed development chamber with counter plates

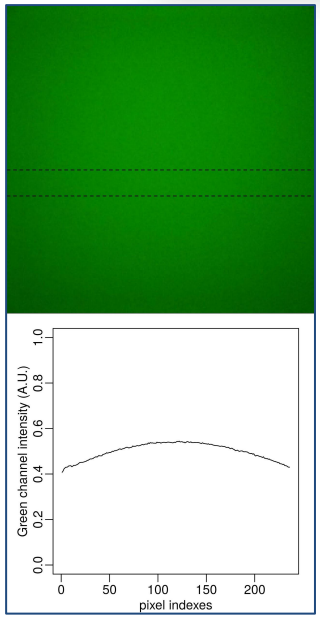
Parabens: EtOH/H_2O/AcOH, 14:40:0.1
Silica gel 60 CN F_{254} s

Detection at 254 nm

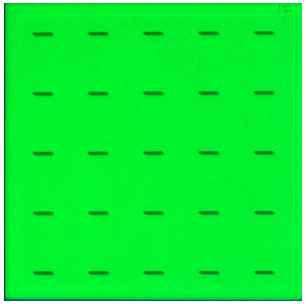
Visualization chamber with raspberry pi, camera and UV-LED



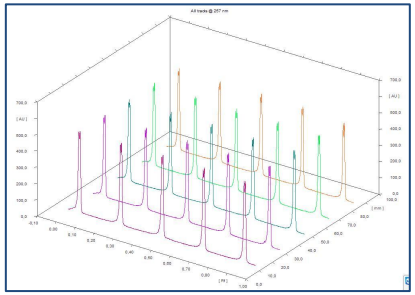
First chromatograms recorded



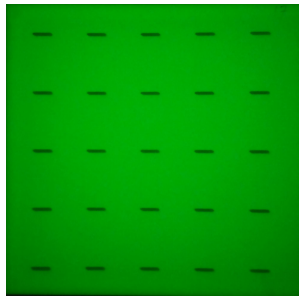
Inhomogeneous illumination



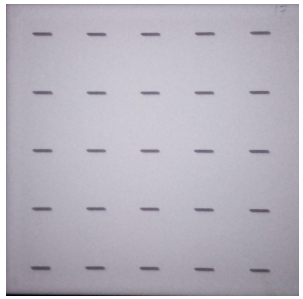
Commercial visualiser
automatic exposure
%RSD = 3.5%



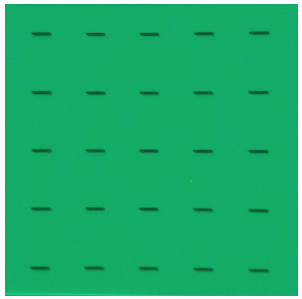
Scanner
257 nm
%RSD = 3.8%



VisuLab
200 ms, ISO 800
no image correction
%RSD = 6.8%

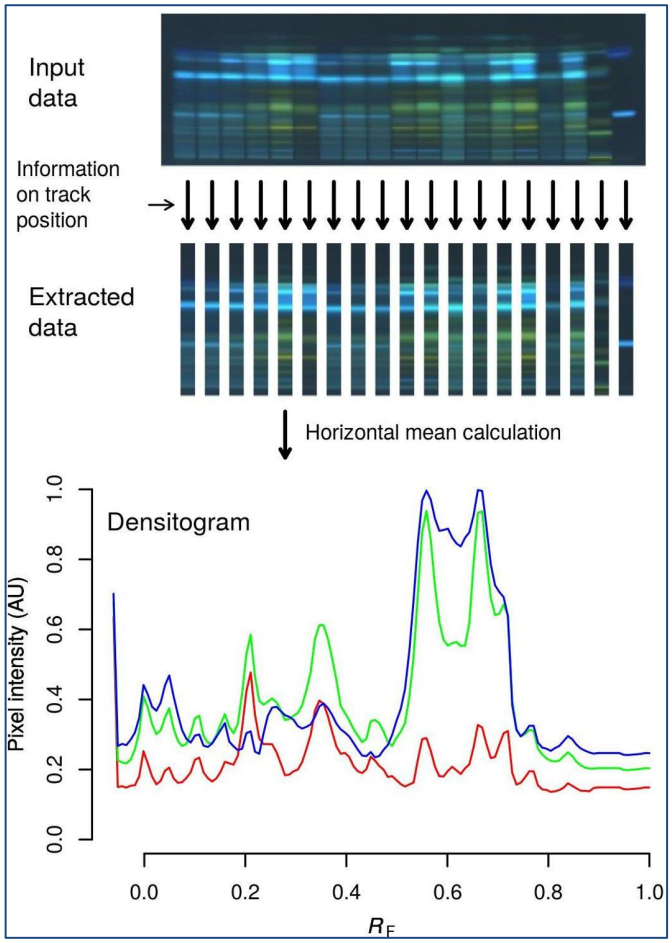


VisuLab
800 ms, ISO 200
no image correction
%RSD = 4.6%

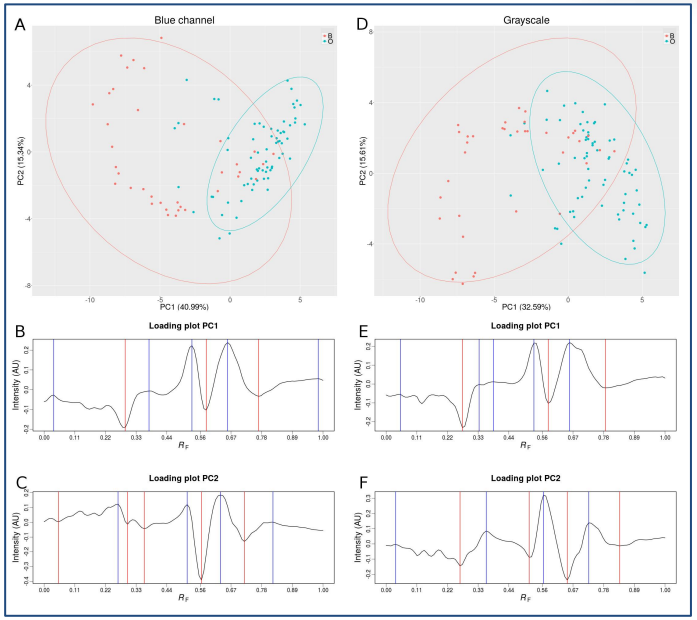


VisuLab
800 ms, ISO 200
background correction
%RSD = 3.4%

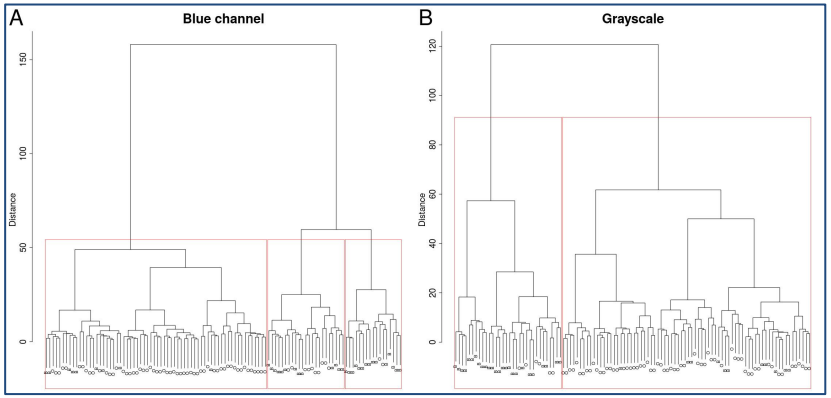
Data analysis: rTLC



Automatic video densitogram extraction



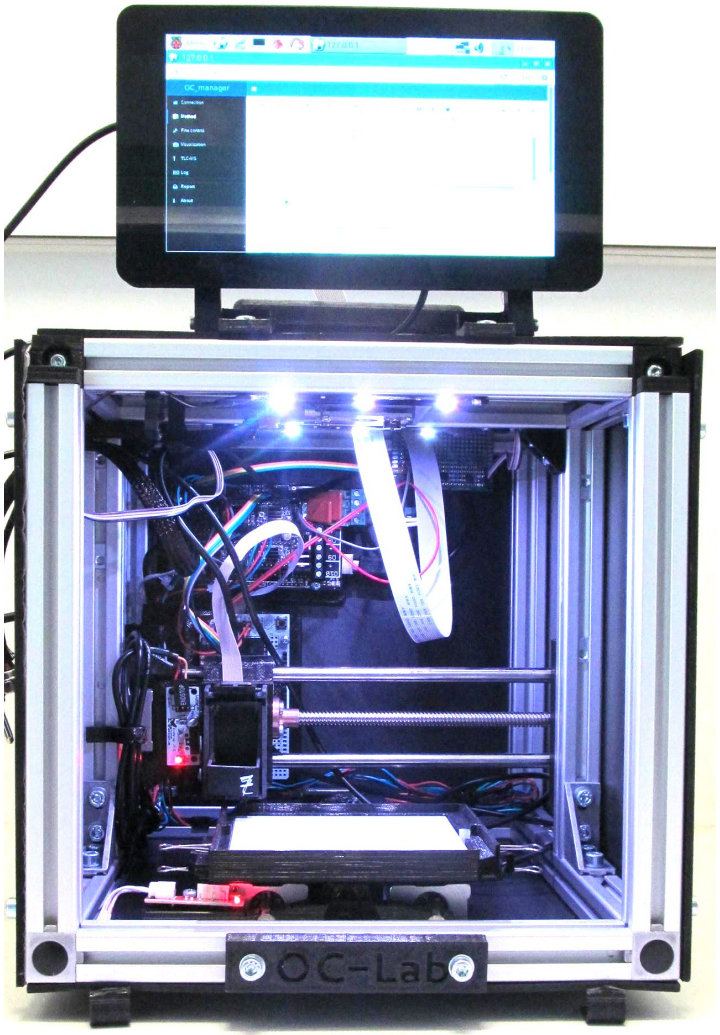
Principal component analysis of German propolis samples



Hierarchical cluster analysis of German propolis samples

Fichou, D., Ristivojević, P., Morlock, G. Anal. Chem. 2016, 88, 12494-12501

Office Chromatography: apparatus features



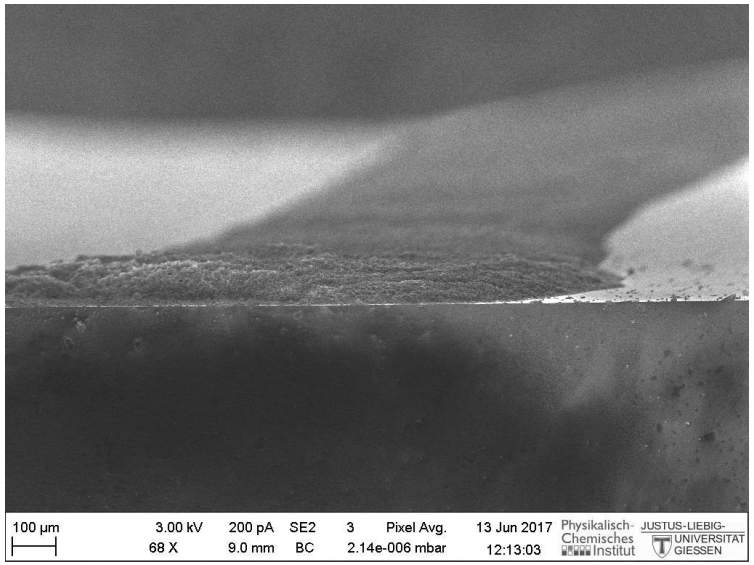
Steps:

- Layer printing
- Sample application
- Mobile phase printing
- Derivatization (in preparation)
- Plate heating (in preparation)
- Visualization
- Data analysis

Specifications:

- 96 dpi inkjet resolution (250 µm)
- 50 µm mechanical resolution
- ≅ 100 pL per drop
- Open source
- Evolutive
- Compact (260 x 310 x 260 mm)

JUSTUS-LIEBIG-UNIVERSITÄT GIESSEN
Food Science



Thank you!