Office Chromatography: Precise printing of sample solutions on miniaturized thin-layer phases

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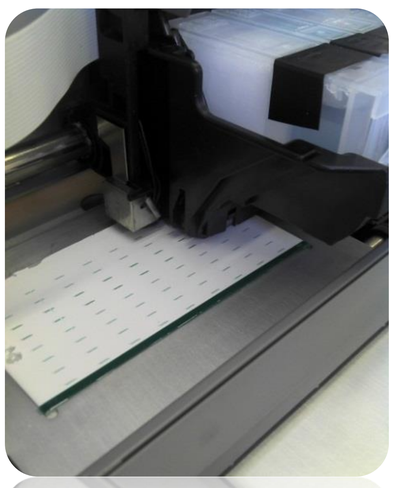


Introduction

HPTLC is a separation technique routinely used to identify and quantify components in a wide range of analytical samples. Its full analytical power is based on the combination of automated devices. The progress in planar chromatography affords miniaturized plates like ultrathin monolithic[1,2], electrospun[3,4] and nanostructured[5,6] layers. However, common automated HPTLC devices for application and development can not handle the miniaturized and sensitive ultrathin layers. Moreover, only small volumes of sample solution are needed and can be applied quantitatively via bubble jet printing. For this reason, a high-resolution office printer has been modified for applying sample solutions in a precise process without carryover. The evaluation of the plates is based on a high-resolution office scanner and respective image evaluation software. The aim of this project was to combine new achievements of print & media technology and planar chromatography on miniaturized plates.

Results and discussion

Modifications on the printer

The first step was the modification of the Pixma 3000 printer for safe and precise sample application followed by image evaluation with the flatbed scanner CanoScan 9000F (both Canon) with transmitted light option (Fig. 1). Chemical inkjet printing was employed for food dye samples[7,8], sugar solutions[9] and application of derivatization reagents[10] so far. Evaluation via an office scanner and software was demonstrated for hydrazines[11], ochratoxine A[12], aflatoxines[13] and staining solutions[14].

Fig. 1 Canon Pixma 3000 with original CD-tray, Canon CanoScan 9000F and modified Pixma 3000 and custom-built plate tray with mounted 10 x 5 cm thin-layer plate in the new tray guide system

Unnecessary parts of the original printer were removed for free access to the printing unit under operating conditions. The waste fluids were collected in a tank, instead of rinsing into sponges under the printing unit. All electronic parts were assembled at the rear side, unnecessary sensors for the housing and paper movement were removed. As the cartridge fill level sensor was dismounted, clear solutions and cartridge imitations were accepted. Unnecessary parts of the paper feed system were removed obtaining enough space for a safe movement of the plate tray (Fig. 2, left) and space for the tray guide system and the intended scanner unit.

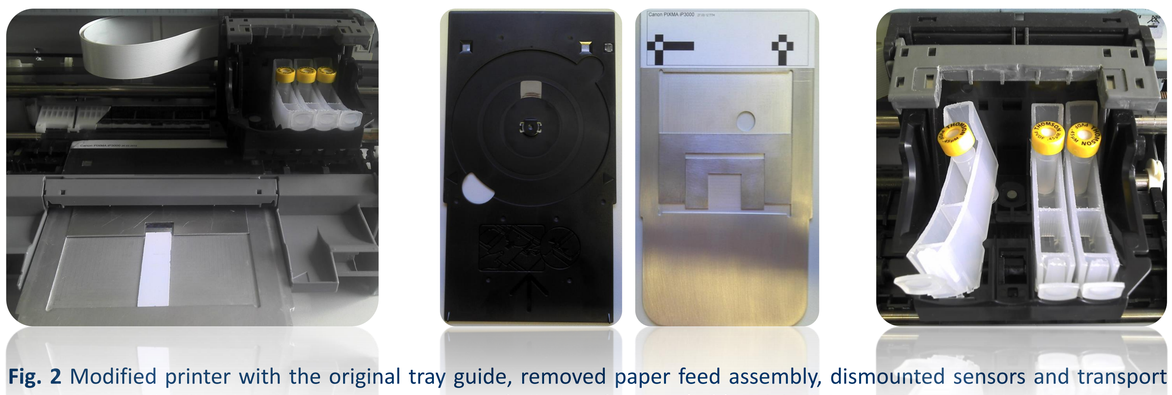


Fig. 2 Modified printer with the original tray guide, removed paper feed assembly, dismounted sensors and transport slide cut out allowing the contactless movement of all plate formats (left); original CD-tray and custom-built plate tray (center); print head carrier with Thomson SINGLE StEP® Filter Vials as sample reservoir in cartridge adapters (right)

The original CD print tray was replaced by a custom-built plate tray for eased handling of miniaturized plates (Fig. 2, middle). In combination with the new tray guide system, the precision during repeated printings was improved. Filter Vials were used to handle small volumes of samples with different viscosities allowing quick exchange of printing medium and purge liquid (Fig. 2, right). The purge unit was modified by attaching four waste lines at the purge head for quick and complete cleaning of the print head via vacuum line.

Application for HPTLC

A mixture of five food dyes (E102, E126, E128, E131 and E142) in water - methanol 9:1 (*V*/*V*) was applied in increasing amounts by multiple printjobs on a HPTLC plate silica gel 60, 10 x 7 cm (Fig. 3). All the repeated, vector graphic-based printjobs (2x to 10x) had to be applied precisely to the same start zone. These print areas of 6.0 x 0.7 mm were developed with ethyl acetate – methanol – water – acetic acid 65:23:11:1 (*V*/*V*/*V*/*V*) up to a migration distance of 55 mm. The chromatogram under white light illumination (transmission) was digitized with a resolution of 1200 dpi. Evaluation via video densitometry (VideoScan, CAMAG) showed determination coefficients R^2 of 0.9990 - 0.9998 (Fig. 4).

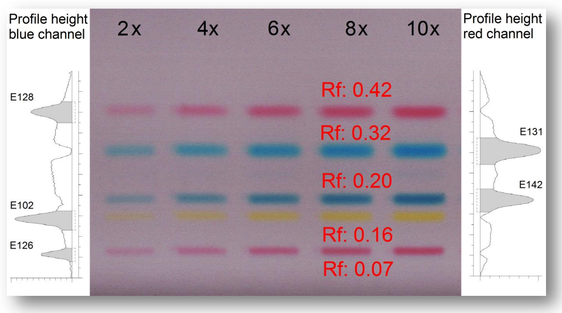


Fig. 3 Repeated printjobs and HPTLC analysis of E126 (hR_F 7; 30-150 ng), E102 (hR_F 16; 20-100 ng), E142 (hR_F 20; 10-50 ng), E131 (hR_F 32; 20-100 ng) and E128 (hR_F 42; 30-150 ng)

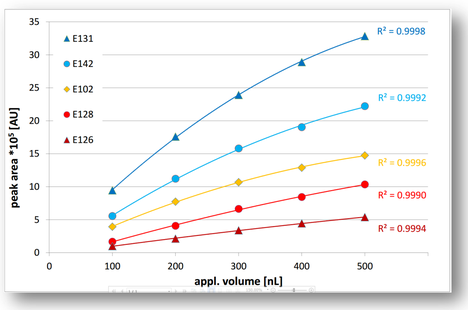
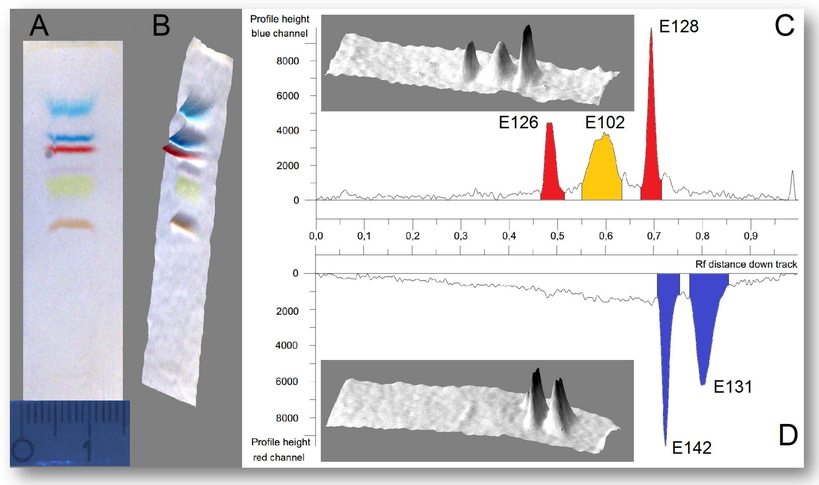


Fig. 4 Calibration curves for the 5 food dyes showing good correlations between signal areas and application volumes (100-500 nL/band, *n* = 4)

Application on nanostructured layers

The custom-built plate tray was equipped with adapters for all common formats of miniaturized plates. The previous HPTLC method was modified and adjusted to 15 x 50 mm miniaturized silicon-carbon thin-layer phases manufactured by carbon-nanotube-templated microfabrication[6]. Food dye mixture (100 nL) was applied and developed with 300 µL ethyl acetate – methanol – water – acetic acid 150:35:25:1 (*V*/*V*/*V*/*V*) up to 46 mm (Fig. 5).

Fig. 5 (A) Chromatography on novel layers of 100 nL food dye mixture applied by the modified printer: E126 (hR_F 48; 30 ng), E102 (hR_F 59; 20 ng), E128 (hR_F 69; 30 ng), E142 (hR_F 73; 10 ng) and E131 (hR_F 81; 20 ng); documentation via TLC Visualizer (CAMAG); (B) 3D image profile using ImageJ (NIH); evaluation via VideoScan (CAMAG) for (C) blue filter and (D) red filter channel



Outlook

The Office Chromatography concept was used for investigation of novel plates. Combining all the items in a miniaturized scale will disclose the real performance of miniaturized planar chromatography.

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] D. Jensen *et al.*, J. Chromatogr. A **1257**, 195 (2012). [7] G. Morlock *et al.*, Anal. Chem. **82**, 2940 (2010). [8] J. Wannenmacher *et al.*, J. Chromatogr. A **1318**, 234 (2013). [9] S. Kirchert *et al.*, Anal. Bioanal. Chem. **405**, 7195 (2013). [10] G. Morlock, C. Stiefel, W. Schwack, J. Liq. Chromatogr. Relat. Technol. **30**, 2171 (2007). [11] A. Abbaspour *et al.*, Anal. Methods **2**, 349 (2010). [12] J. Welke *et al.*, J. of Planar Chromatogr. **23**, 116 (2010). [13] J. Stroka *et al.*, J. of Planar Chromatogr. **14**, 109 (2001). [14] R. Johnsson *et al.*, J. Chromatogr. A **1164**, 298 (2007).

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