# Separation of pigment formulations by HPTLC/AMD

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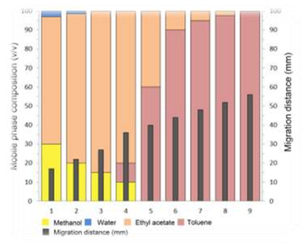
## Highlights

- *Separation and characterization of different pigment formulations (124 samples) and additives (8)*
- *Fast and simple sample preparation: Direct solving or suspension with 15 min ultrasonic extraction*
- *Additional gain of information by post-chromatographic derivatizations*
- *Selective method suitable for application in routine analysis during quality control*
- *Hyphenation of HPTLC with MS, FTIR, and NMR supports the assignment of unknown pigment components*

### *Selective and sensitive separation of different pigment formulations*

- ✓ *Sharp zones due to a 9-step gradient using the AMD 2 System*
- ✓ *Separation of 18 different colored, matrix-rich pigment formulations (Fig. 1)*
- ✓ *Gain of information by multi-detection: white light, UV 366 and 254 nm (A-C)*
- ✓ *Analysis time of less than 5 min per sample*



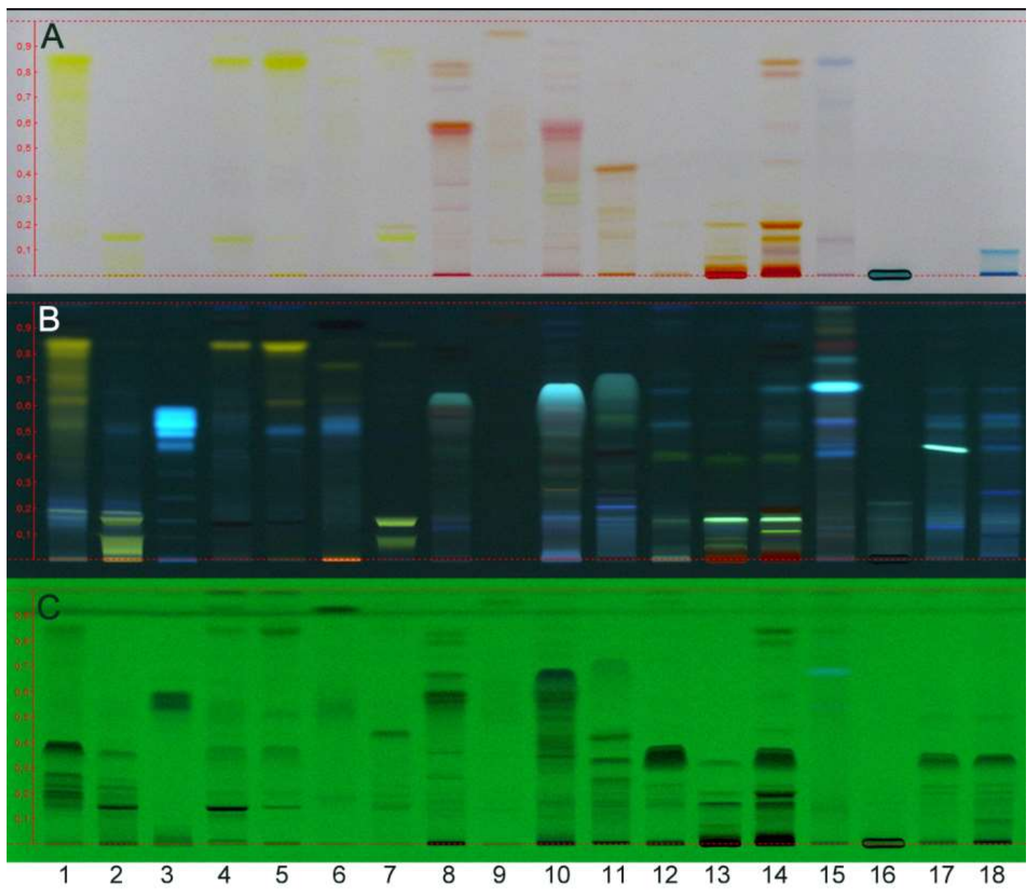




**Fig. 1** HPTLC chromatograms of pigment formulations PY 12 (1), PY 13 (2), PY 83 (3), PY 174 (4), PY 188 (5), PO 16 (6), PO 34 (7), PR 146 (8), PR 166 (9), PR 184 (10), PR 210 (11), PR 48:1 (12), PR 48:2 (13), PR 57:1 (14), PV 23 (15), PG 7 (16), PB 15:3 (17) and PB 15:4 (18)

### *Comparison of different suppliers and batches during routine analysis*

- ✓ *Significant differences between single pigment suppliers and batches (Fig. 2)*
- ✓ *Evident differences in the area of coatings and binders after detection with sulfuric acid reagent (Fig. 2C)*
- ✓ *Fast comparison of several pigment batches as part of regular quality control*

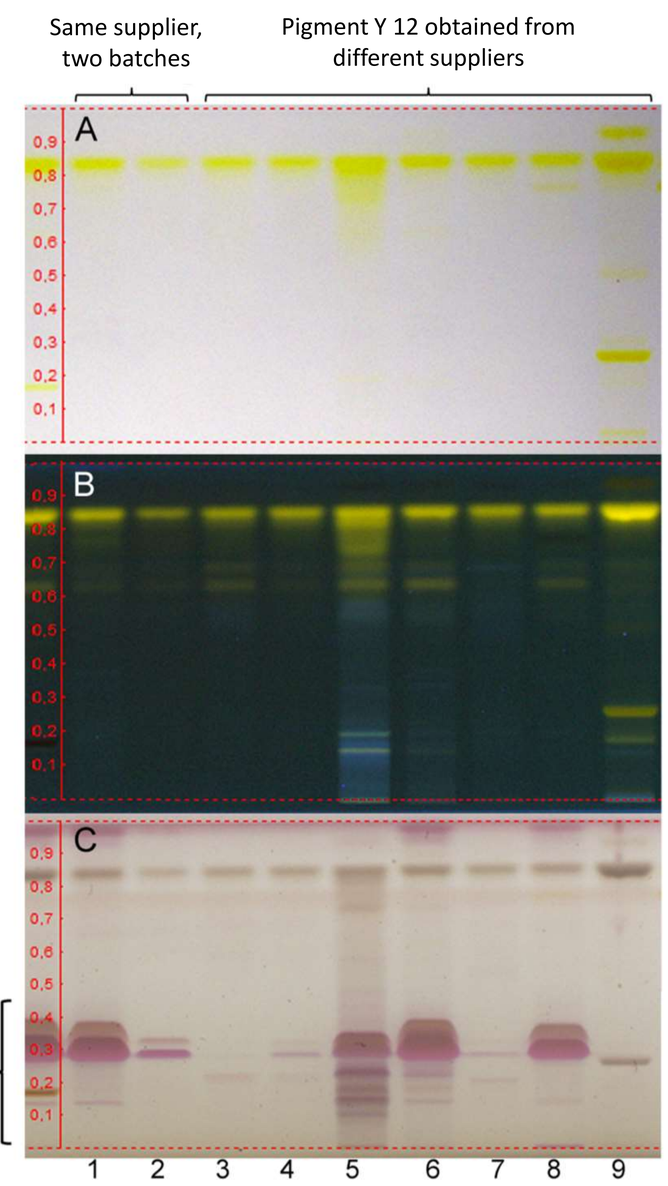



**Fig. 2** Comparison of different samples of PY 12 under white light (A), UV 366 nm, (B) and after derivatization with sulfuric acid reagent under white light (C)

### *Post-chromatographic multi-detection*

- ✓ *First indication of contained substance classes*
- ✓ *Effect-directed analysis (EDA) of bioactive components*
- ✓ *Sulfuric acid reagent especially suitable (Fig. 3)*

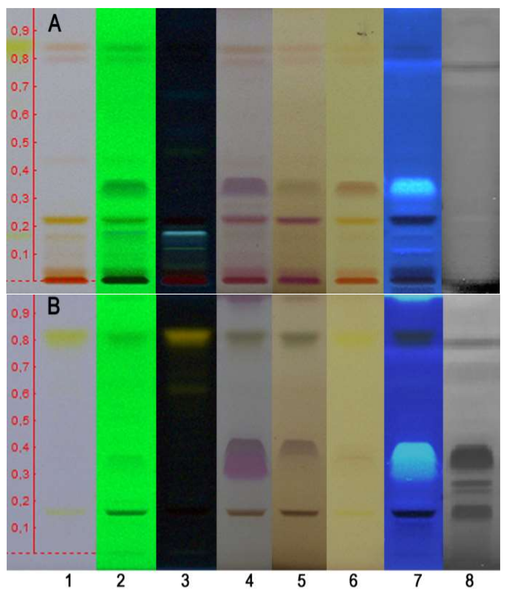



**Fig. 3** HPTLC chromatograms documented after multi-detection of pigment formulations PR 57:1 (A) and PY 12 (B)
1 under white light (as for 4-6)
2 at UV 254 nm
3 at UV 366 nm
4 via sulfuric acid reagent
5 via β-naphthol reagent
6 via ninhydrin reagent
7 via primulin reagent at UV 366 nm
8 after EDA using *A. fischeri (bioluminescent image in greyscale)*

### *Characterization by HPTLC/ESI-MS*

*Exemplarily, measured mass spectrum of an unknown, strongly fluorescent substance zone of PV 23 at $hR_F$ 66 (Fig. 4) → basepeak at m/z 180*

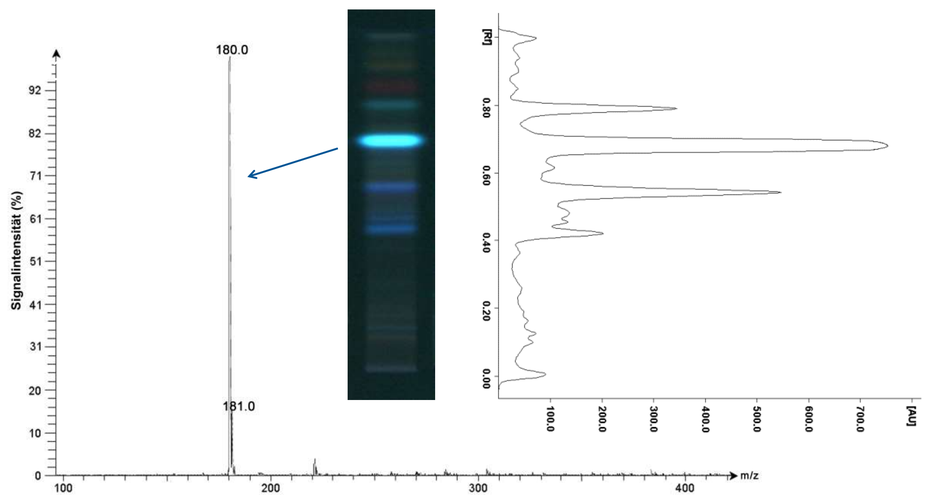



**Fig. 4** HPTLC-ESI⁺-MS of the blue fluorescent substance zone of pigment formulation PV 23 as well as HPTLC chromatogram and densitogram

**Reference** [1] C. Stiefel, S. Dietzel, M. Endress, G.E. Morlock, Separation of pigment formulations by high-performance thin-layer chromatography with automated multiple development, J. Chromatogr. A 1462 (2016) 134-145.

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