HPTLC-MS analysis using a novel compact single quadrupole mass spectrometer

Frank Porbeck and Dr. Andreas Wiesner, Advion, Harlow, UK, presented a new and very compact single quadrupole mass spectrometer named expressionCMS, which can optionally be used with an ESI or APCI ion source. Via the TLC-MS Interface it was coupled to HPTLC. Its performance was exemplarily shown for isopropylthioxanthone (ITX) and caffeine in cooperation with the working group of Professor Gertrud Morlock, Justus Liebig University Giessen, Germany.

HPTLC analysis

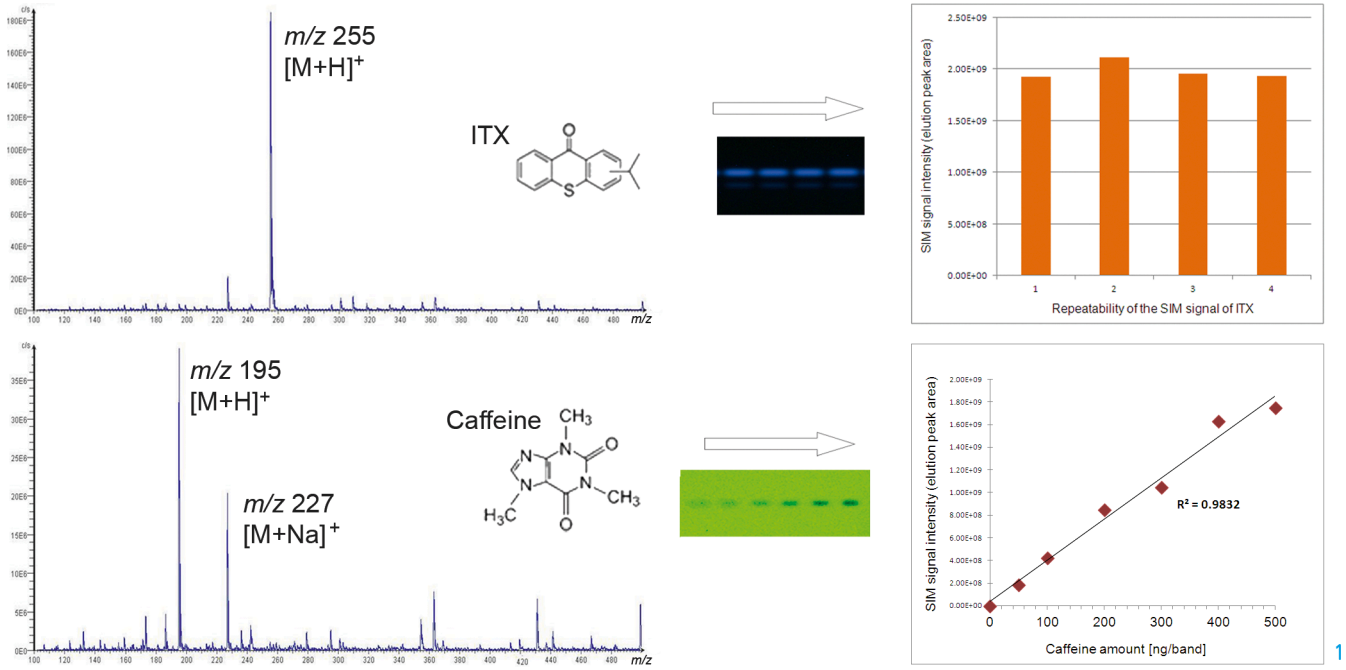
Layer: HPTLC silica gel 60 F_{254} plates (Merck) pre-washed with methanol – water 4:1; application as 6 mm bands with ATS 4; development with toluene – *n*-hexane 4:1 for ITX and acetonitrile containing 2.5 % ammonia for caffeine; evaluation with TLC Scanner 4 by fluorescence measurement at 366/>400 nm (ITX) and absorbance measurement at 275 nm (caffeine) to check the HPTLC performance.

MS analysis

Elution with TLC-MS Interface (circular elution head) with methanol – ammonium formate buffer (10 mM, pH 4) 19:1 at a flow rate of 0.1 mL/min, online connected to the MS, which was run in ESI^+ mode for selected ion monitoring (SIM) and full scan.

Results and Discussion

The analytical response showed a determination coefficient (R^2) of 0.9966 (ITX, 5–100 ng/band) and 0.9832 (caffeine, 50–500 ng/band). The repeatability (*%RSD*) was determined to be 3.9 % (ITX, 50 ng, *n* = 4) and 8.8 % (caffeine, 300 ng, *n* = 6). Based on this good performance data, the compact MS enabled mass-over-charge (*m/z*) signal intensities of HPTLC zones in a concentration-dependent (quantitative) and reliable manner. As a very compact MS, it will help to establish MS in the workflow of TLC/HPTLC laboratories.



Up: Mass spectrum of ITX and its repeatability in the SIM mode (%RSD = 4 %)
Down: Mass spectrum of caffeine and its analytical response in the SIM mode (R^2 = 0.9832)

Further information is available from:

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