Simultaneous Determination of Caffeine, Ergotamine and Metamizol by HPTLC/UV/FLD and HPTLC/ESI-MS

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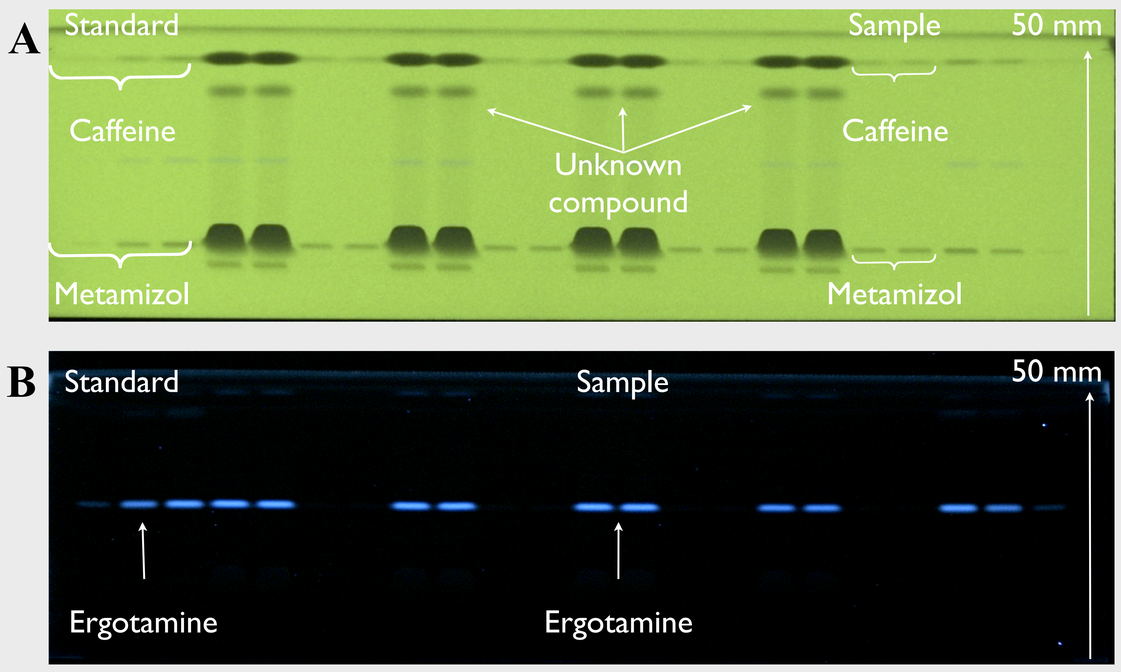
Introduction

Headache is one of the most frequently reported symptoms; epidemiologic studies have found that 57% of males and 76% of females had at least one or even more headache attacks per month [1]. Different kinds of drugs are used for the treatment of headache disorders. Among the most common pharmacological agents for acute therapy are: analgesic, nonsteroidal anti-inflammatory drugs and ergot alkaloids. The objective of this work was to develop a high-throughput analytical method to detect simultaneously caffeine, ergotamine tartrate and metamizol in headache tablets by HPTLC-UV/FLD with mass confirmation via HPTLC-ESI/MS.

Method

The compounds were dissolved with 100 mL of methanol-water mixture (7:3 v/v) and to complete the dissolution process, the flask was shaken in a mechanical shaker for 20 min and sonicated for 10 min in an ultrasonic bath. Chromatography was carried out on 20 x 10 cm HPTLC plate silica gel 60. Solutions of samples and the standard mixture were applied with Automatic TLC Sampler IV (ATS IV) from CAMAG (Muttenz, Switzerland). Detection was performed by multi-wavelength scanning using the Scanner 3 (CAMAG).

Results

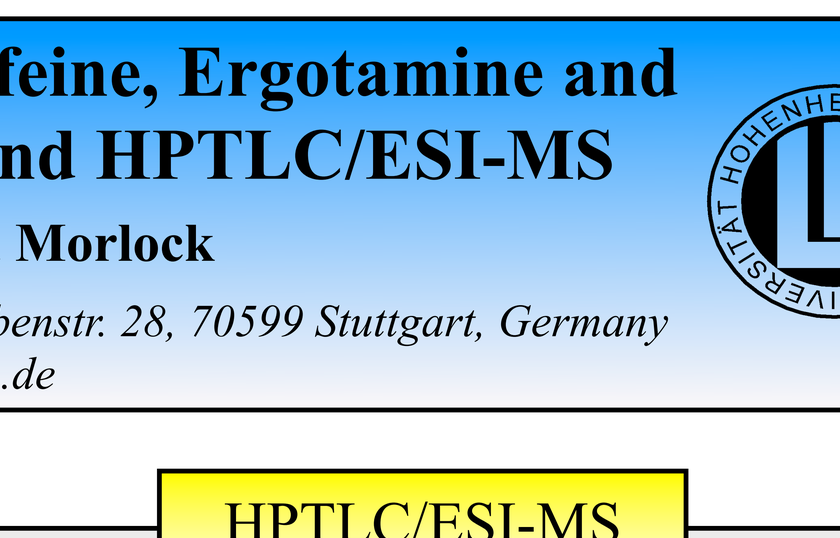


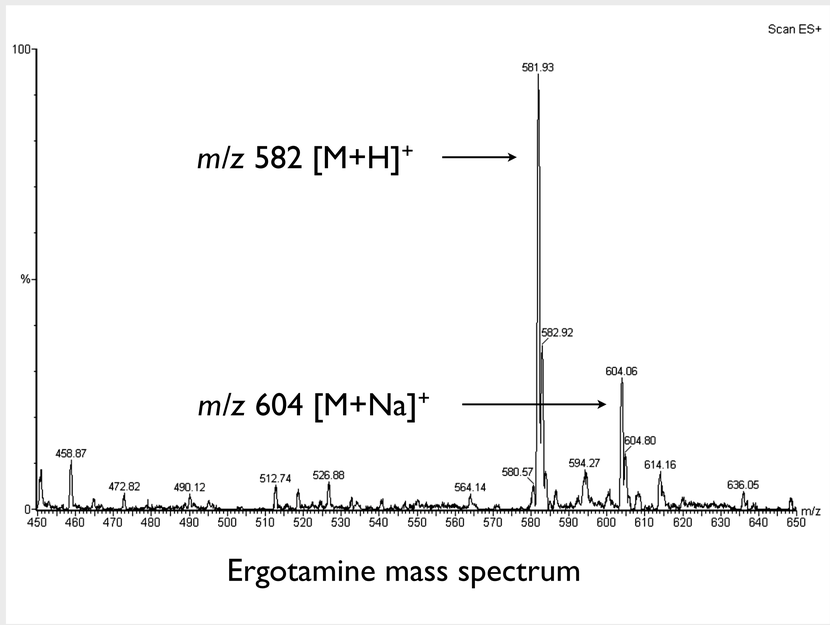
HPTLC plate silica gel 60 illuminated at UV 254 nm (A) and 366/>400 nm (B)

Calibration plots showed regressions with determination coefficients (r^2) > 0.999. Recoveries in commercially available pharmaceutical products were 102.8 % ± 2.8 % for ergotamine, 106.6 % ± 3.2 % for caffeine and 104.7 % ± 2.2 % for metamizol.

HPTLC/ESI-MS

The compounds mass spectra were obtained using a plunger-based extraction device for HPTLC/ESI-MS [2] which employment was recently demonstrated in food [3] and trace analysis [4].





Examples of mass spectra obtained directly from the compound band.

Conclusion

This new HPTLC method offers a reliable and low-cost high-throughput alternative.

The compounds online identification via HPTLC/MS gives a reliable approach to ensure correct peak identification and is also helpful for decipher the type of impurities present.

References:
[1] J. O'Sullivan and J. T. Mccabe, AANA J. 2006, 74:61-69
[2] H. Luftmann, Anal. Bioanal. Chem. 2004, 380:964-968.
[3] M. Aranda and G. Morlock. J. Chromatogr. A (submitted)
[4] G. Morlock, W. Schwack, Anal. Bioanal. Chem. 2006, 385:586-595.

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