



# HPTLC/DART-TOF compared to HPTLC/ESI-MS

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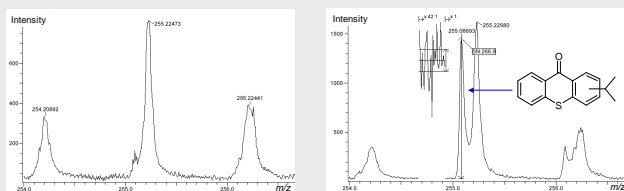


## Introduction

DART (Direct Analysis in Real Time) was introduced at the Pittcon 2005 [1]. This kind of versatile new ion source is working in open air under ambient conditions. The employment of DART in the field of planar chromatography was successfully demonstrated in this study for the first time [2] and compared to a plunger-based device.

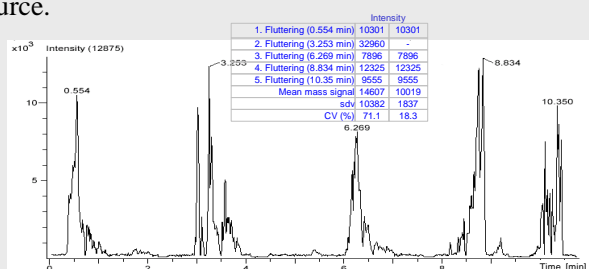
## HPTLC/DART-TOF

Within seconds the mass spectrum of a substance was obtained provided that the substance of interest was positioned on the plate edge. Sensitivity was obtained in the low ng-range on the example of isopropylthioxanthone, a photoinitiator known as a new food contaminant.



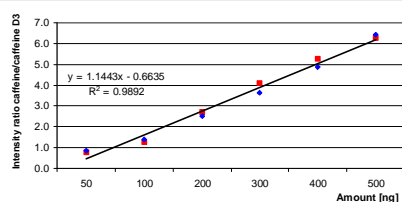
HPTLC/DART-TOF spectra of a blank plate position (left) and a 3.2 ng ITX zone at  $m/z$  255.08693 with S/N of 267 by accumulation (right)

Spatial resolution was better than 7 mm. The analytical response ( $r^2$  0.8202) and repeatability (RSD 18.1 %) were strongly dependent from proper positioning of the HPTLC/TLC plate into the charged gas stream of the ion source.



Mass chromatogram at  $m/z$  255  $[M+H]^+$  showing repeatability of the mass signals of 5 ITX zones at 32 ng/zone; RSD of 71.1 % or 18.3 % if corrected according to Nalimov outlier test ( $P = 0.05$ )

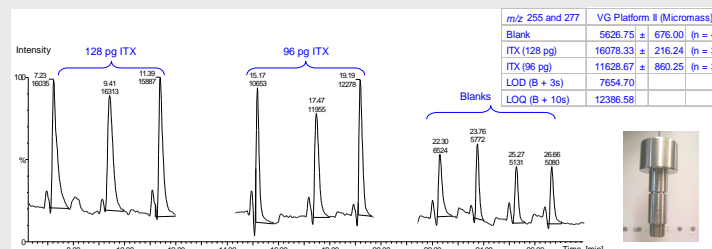
This drawback was overcome by using stable isotope labeled standards shown on the example of caffeine. This way, analytical response ( $R^2$  0.9892) and repeatability (RSD  $< \pm 5.4$  %,  $n = 6$ ) were improved to a high extent.



HPTLC/DART-IDA-TOF: Analytical response (calibration) of caffeine at  $m/z$  195  $[M+H]^+$  in the range of 50 – 500 ng/zone corrected by the stable isotope labeled internal standard caffeine D3 at  $m/z$  198  $[M+H]^+$

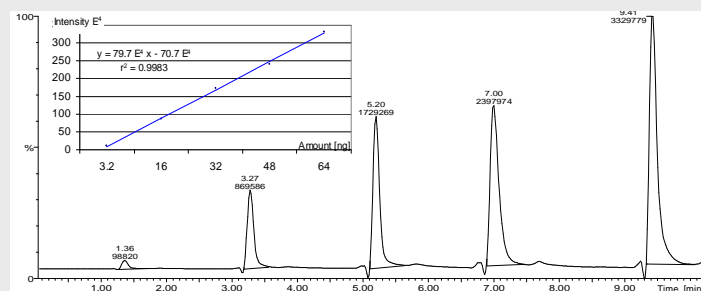
## HPTLC/ESI-MS

This new hyphenation was compared to a plunger-based extraction device for HPTLC/ESI-MS [3] which employment was recently demonstrated in trace analysis [4]. LOQ was found to be 96 pg.



HPTLC/ESI-MS elution profiles of 3 ITX zones 96 and 128 pg each; recording in SIM mode at  $m/z$  255  $[M+H]^+$  and 277  $[M+Na]^+$ ; in comparison 4 extractions of blank positions

The repeatability of 5 ITX extractions at 6.4 ng/zone recorded in the SIM mode at  $m/z$  255  $[M+H]^+$  and 277  $[M+Na]^+$  showed a RSD of 6.7%, analytical response showed a determination coefficient  $r^2$  of 0.9983. The spatial resolution was 2 mm or 4 mm depending on the plunger head used.



Analytical response of ITX in the range of 3.2 to 64 ng/zone showing linear correlation with  $r^2 = 0.9983$ ; recorded by HPTLC/ESI-MS in the SIM mode at  $m/z$  255  $[M+H]^+$  and 277  $[M+Na]^+$

## Conclusion

The comparison showed that repeatability, analytical response and sensitivity of HPTLC/DART-MS were heavily influenced by manual positioning and so far not as well as those results obtained by the plunger-based device. Consequently a plate stage or holder for proper positioning or even scanning of 4 cm-short HPTLC strips is highly required to meet the immense potential of the new HPTLC/DART-MS coupling. Both presented techniques – compared to other approaches – allow mass selective detection in the lower pg-range on silica gel phases.

### References:

- [1] R.B. Cody, J.A. Laramée, H. Dupont Durst, Anal Chem 2005, 77:2297-2302.
- [2] G. Morlock, Y. Ueda, 2006 in submission
- [3] H. Luftmann, Anal Bioanal Chem 2004, 380:964-968.
- [4] G. Morlock, W. Schwack, Anal Bioanal Chem 2006, 385:586-595.

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