

Using Electrospray-Assisted Laser Desorption/Ionization Mass Spectrometry To Characterize Organic Compounds Separated on Thin-Layer Chromatography Plates

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This presentation explains a new technique in mass spectrometry...

ELDI (Electrospray-assisted laser desorption/ionization)

Some abbreviations used in this presentation

TLC : Thin-layer chromatography

MS : Mass spectrometry

ESI : Electrospray ionization

MALDI : Matrix-assisted laser desorption/ionization

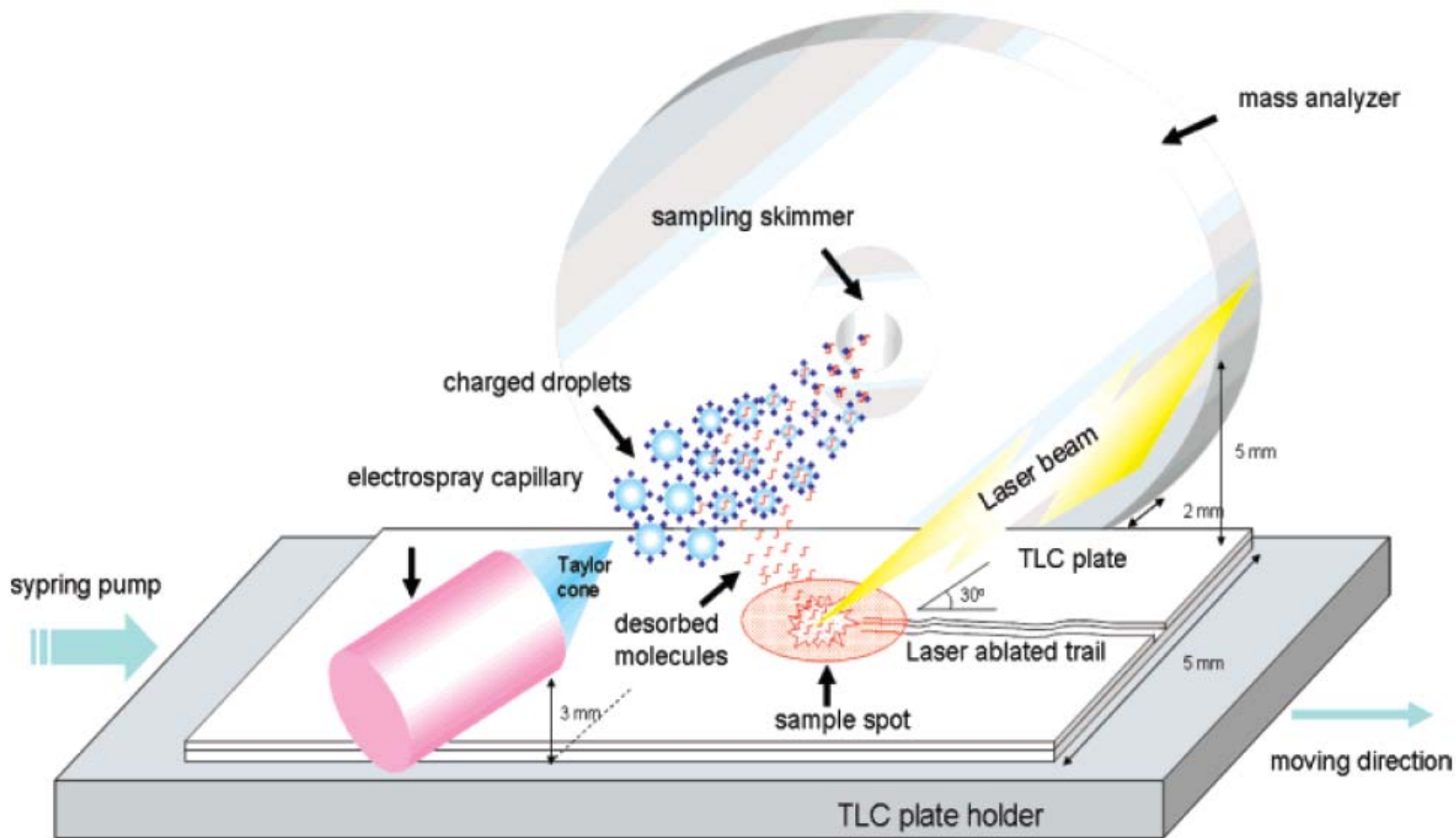
Thin-layer chromatography (TLC) is rapid, simple, and economical separation technique for most organic, inorganic, and biochemical mixtures

Identification of the separated product after TLC is a tedious job

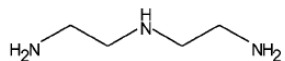
Coupling of TLC to detective instruments for the rapid identification of trace sample is an active area of investigation

MS contributes significantly for the identification of samples after TLC

Direct analysis of TLC plates by traditional MS is difficult because of some reasons

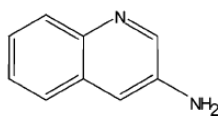


Schematic illustration of the ELDI-MS approach to the desorption and ionization of organic compounds separated on a TLC plate.



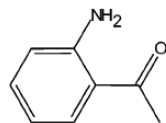
2,2'-Diaminodiethylamine

$[M+H]^+ = 104$



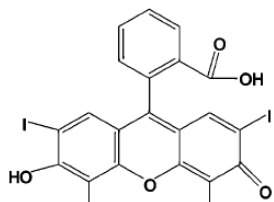
3-Quinolinamine

$[M+H]^+ = 145$



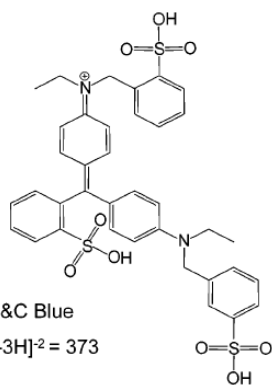
2-Acetylaniline

$[M+H]^+ = 136$



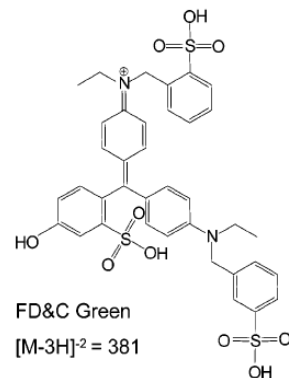
FD&C Red

$[M-H]^- = 835$



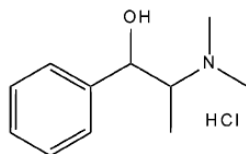
FD&C Blue

$[M-3H]^{-2} = 373$



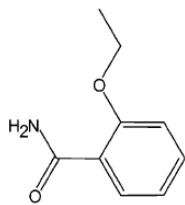
FD&C Green

$[M-3H]^{-2} = 381$



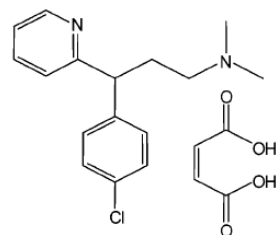
DL-Methylephedrin Hydrochloride

$[M-HCl+H]^+ = 180$



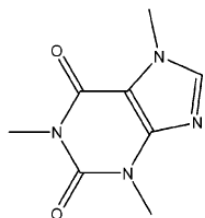
Ethoxybenzamide

$[M+H]^+ = 166$



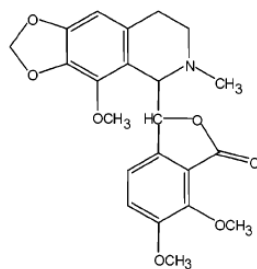
Chlorpheniramine Maleate

$[M-C_4H_4O_4+H]^+ = 275$



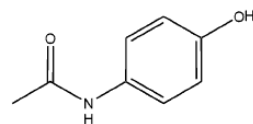
Caffeine

$[M+H]^+ = 195$



Noscapine

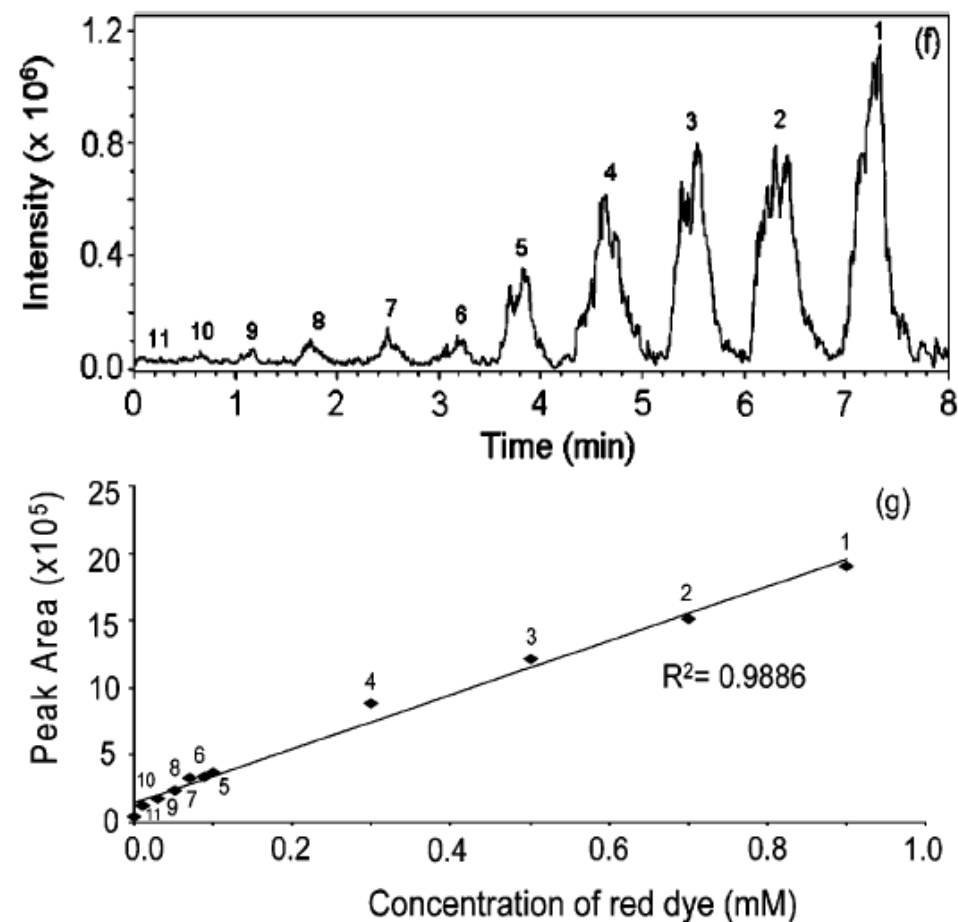
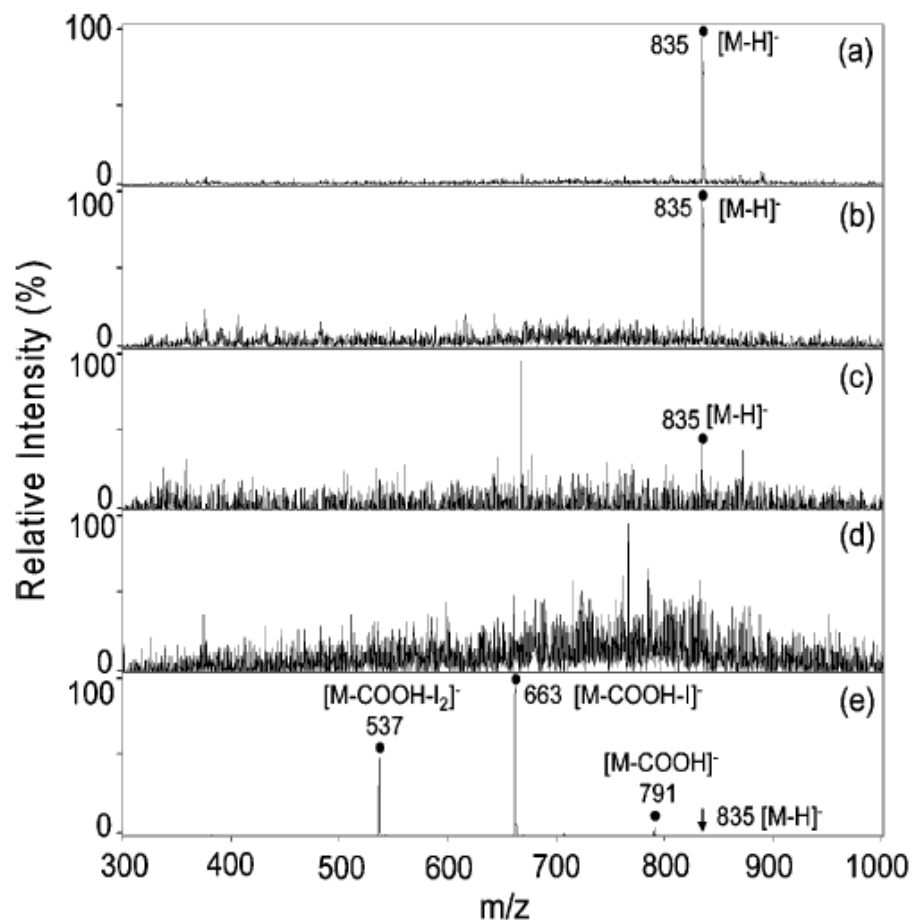
$[M+H]^+ = 414$



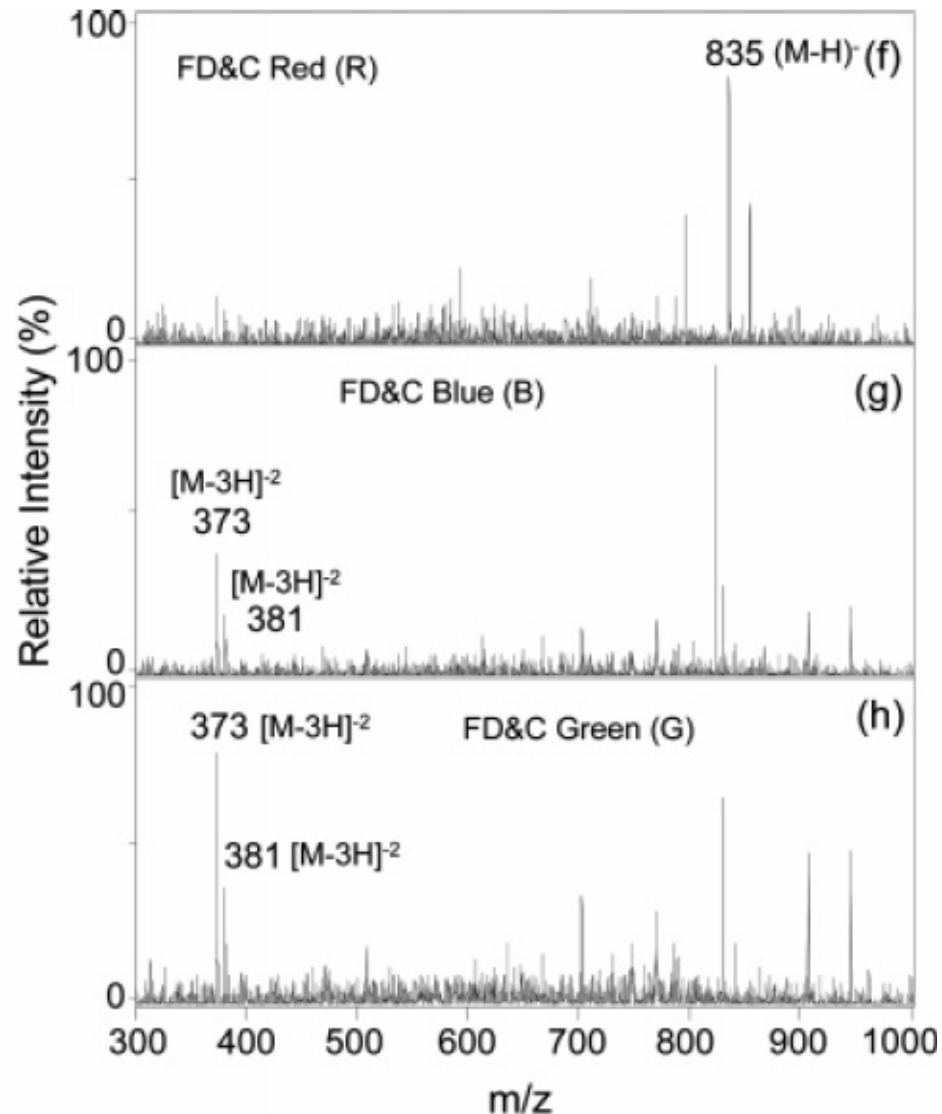
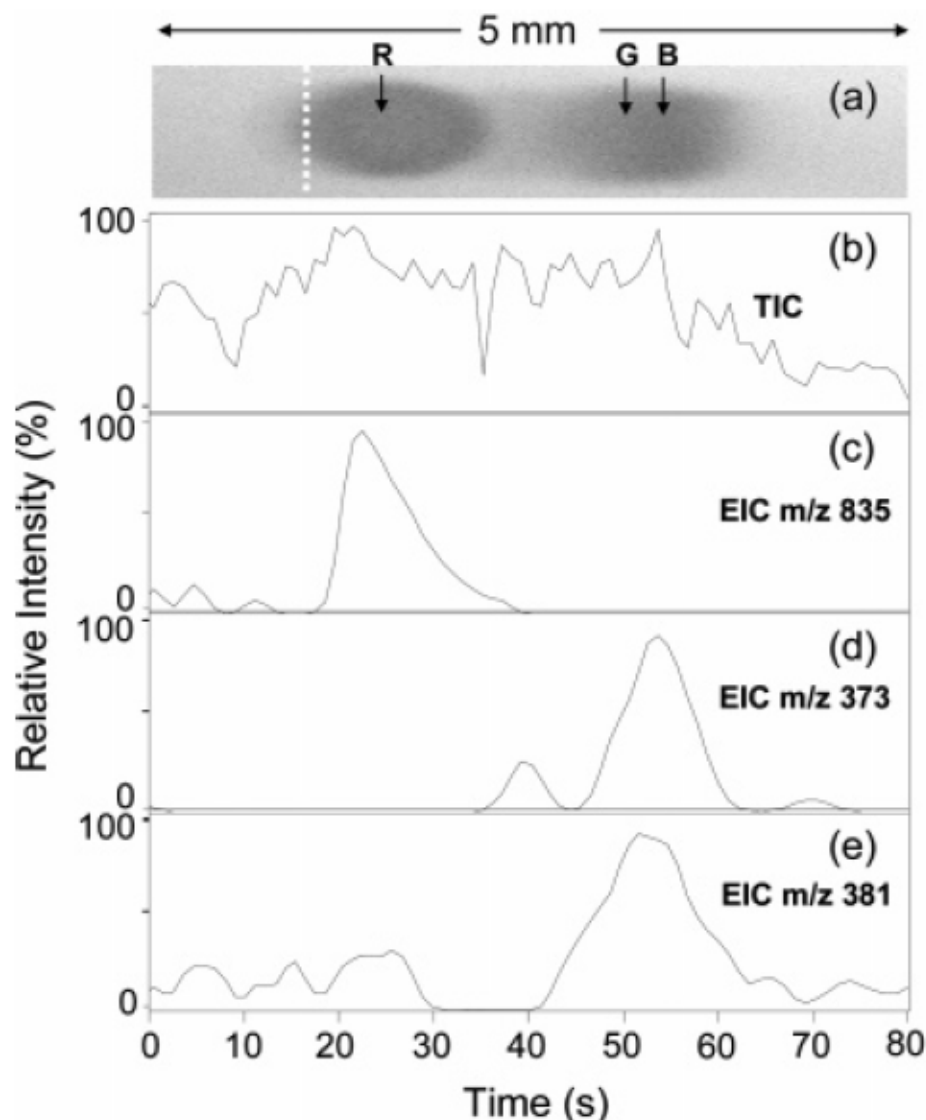
Acetaminophen

$[M+H]^+ = 152$

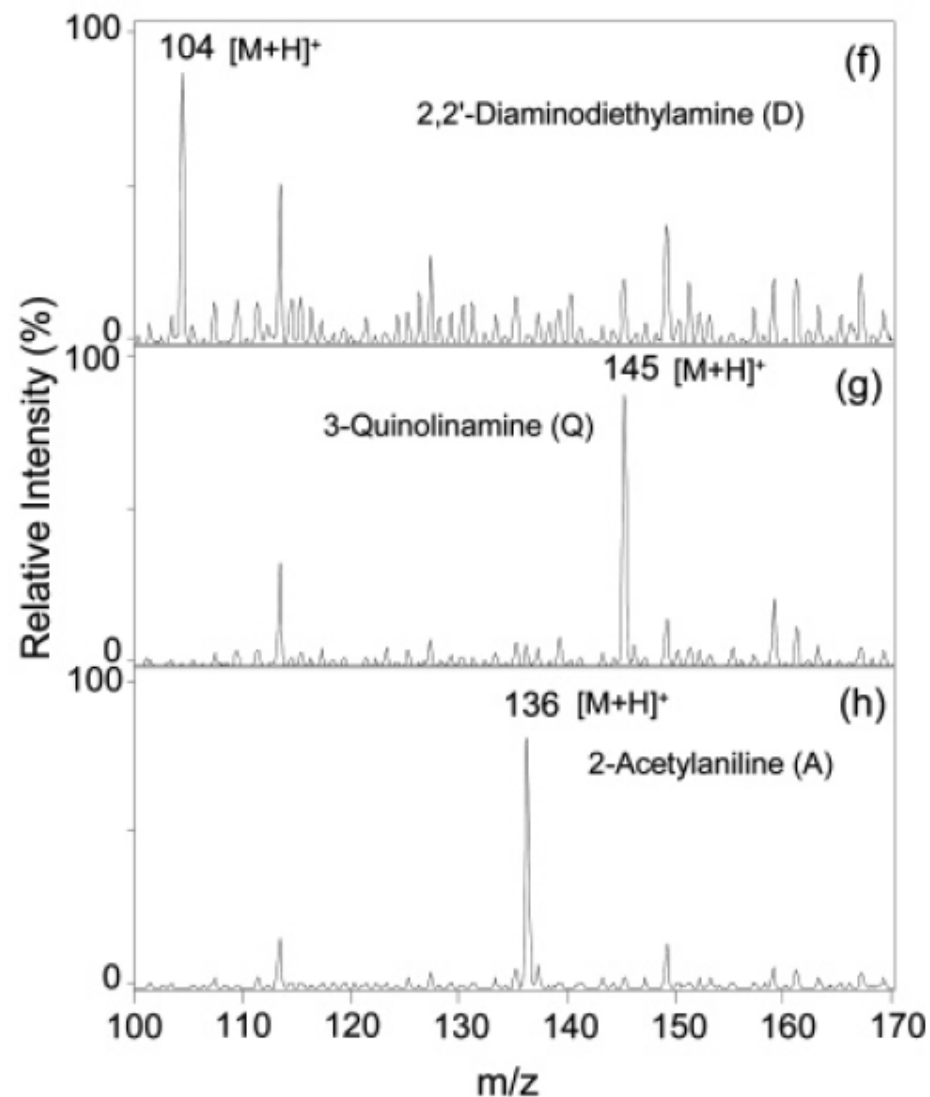
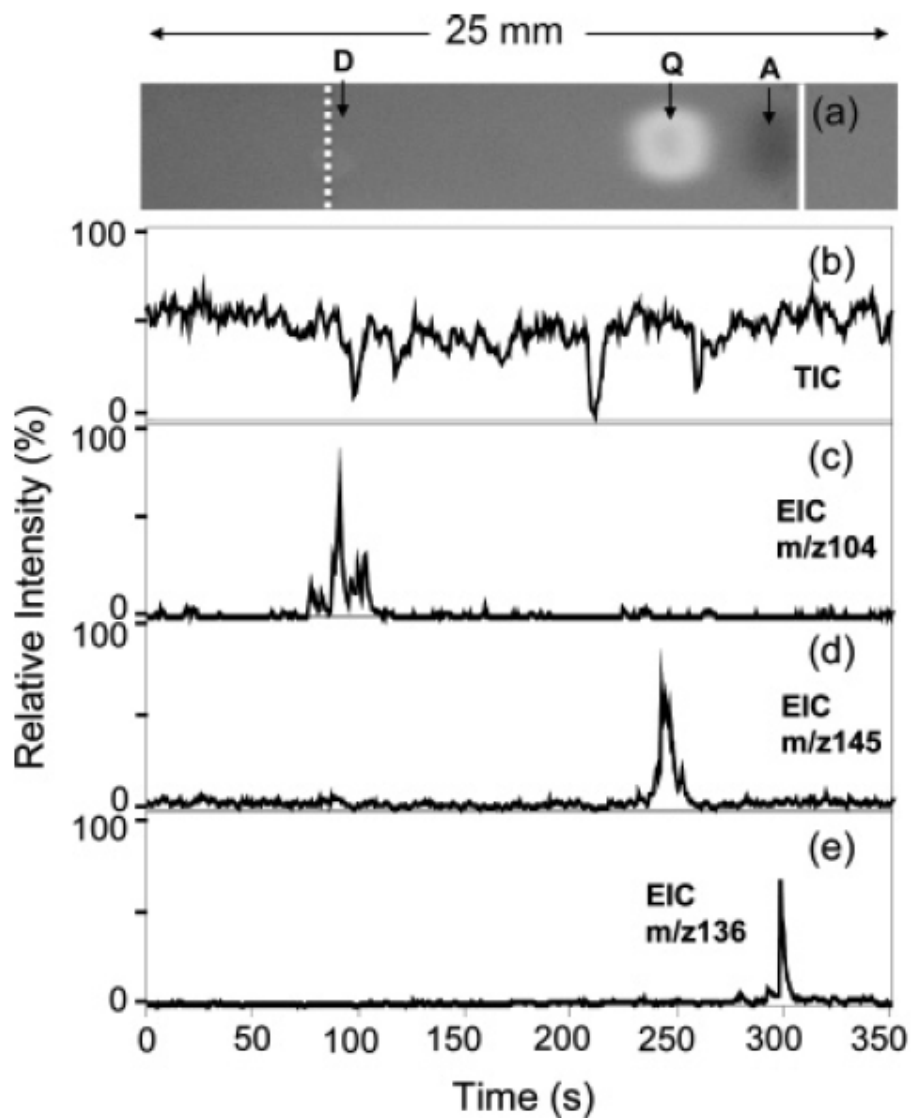
Chemical
compounds
analyzed in
this study.



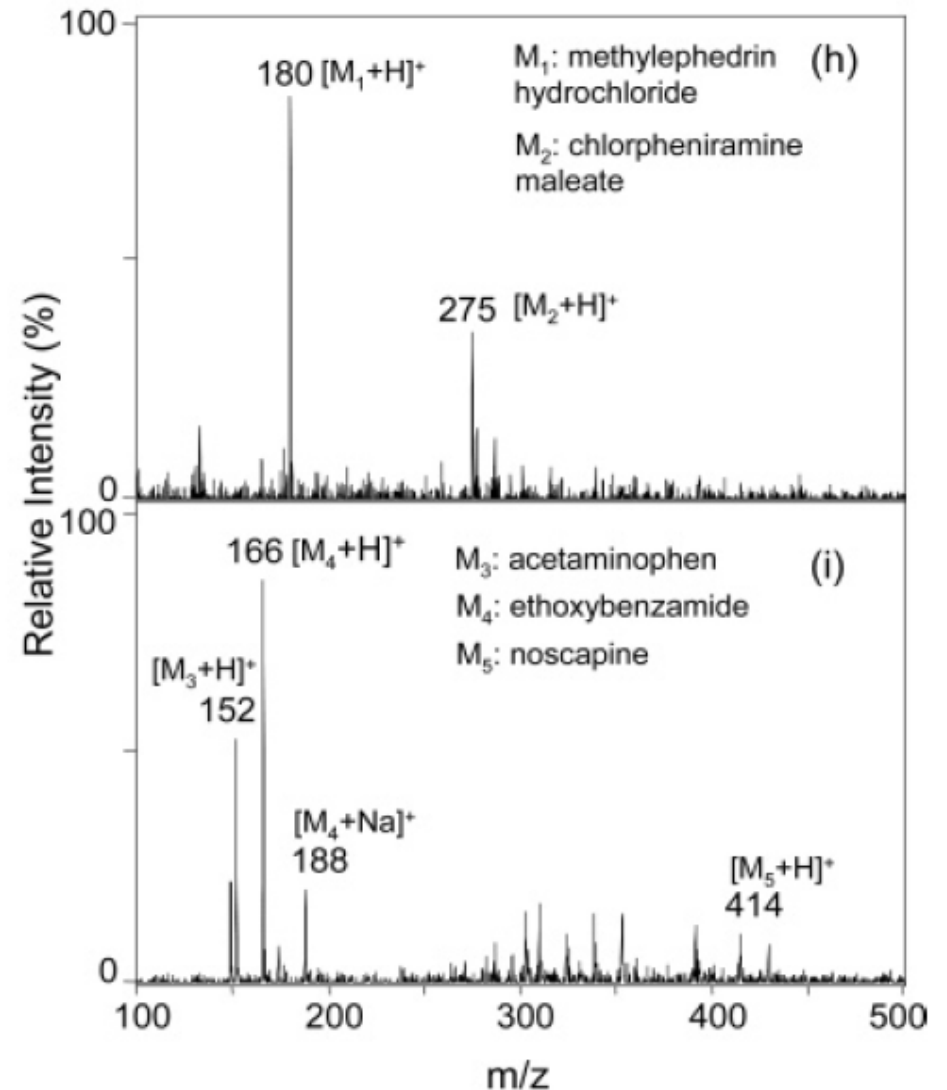
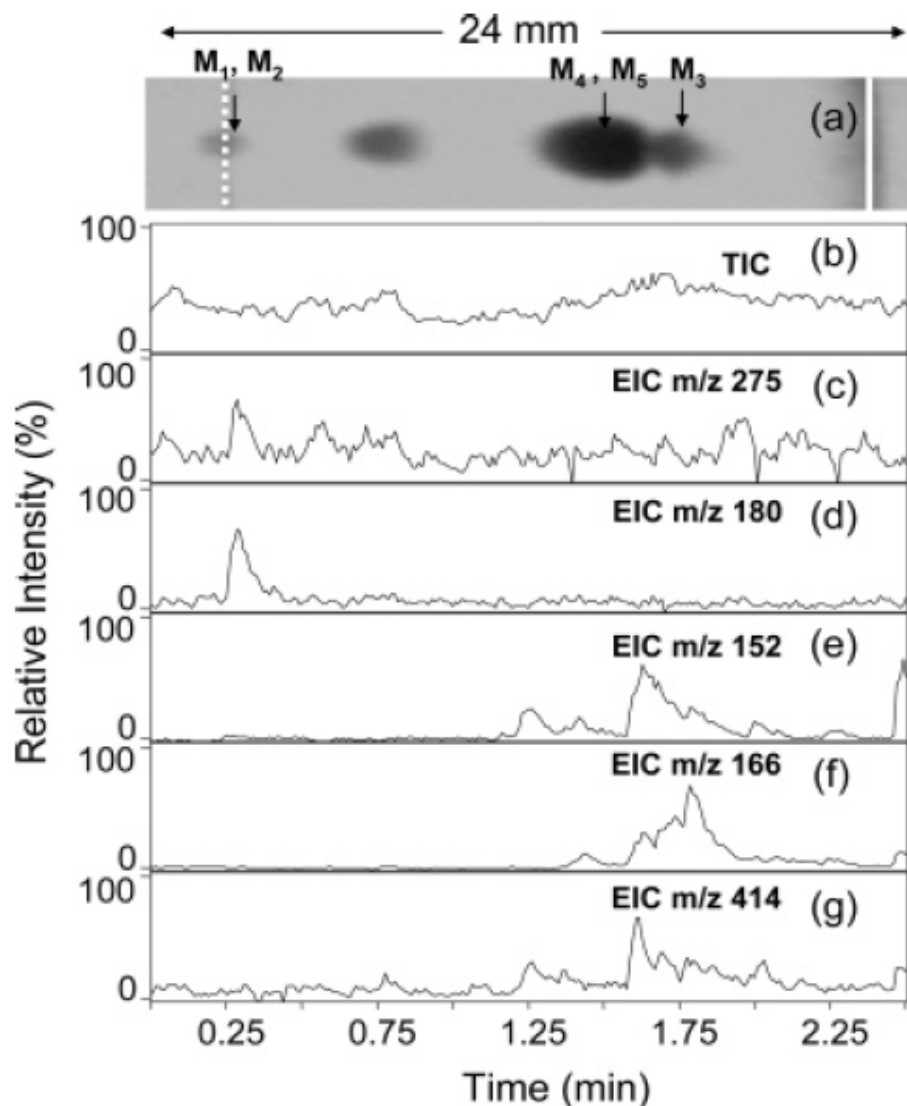
FD&C Red dye standards (m/z 835 for [M-H]⁻) dissolved in water at concentrations of (a) 10^{-4} , (b) 10^{-5} , (c) 10^{-6} , and (d) 10^{-7} M. The MS/MS spectrum of the parent ion of FD&C Red dye (m/z 835) is presented in (e). (f) The ion intensity (m/z 835) of the sample spots on the plate vs scanning time. (g) The calibration curve based on the peak area of m/z 835 in (f) vs the concentration of the sample spots.



FD&C dyes (b) Total ion chromatogram and (c-e) extracted ion chromatograms for the (c) singly charged red dye ion (R, m/z 835), (d) doubly charged blue dye ion (B, m/z 373), and (e) doubly charged green dye ion (G, m/z 381) obtained from full-scan negative-ion-mode data acquired by scanning the developed lane indicated in (a). (f-h) mass spectra (negative-ion mode) corresponding to the respective spot positions on the TLC plate: (f) red dye, (g) blue dye, and (h) green dye.



(b) Total ion chromatogram and (c-e) extracted ion chromatograms for (c) 2,2'-diaminodiethylamine (D, m/z 104), (d) 3-quinolinamine (Q, m/z 145), and (e) 2-acetylaniline (A, m/z 136). (f-h) mass spectra corresponding to the respective spot positions on the TLC plate: (f) 2,2'-diaminodiethylamine, (g) 3-quinolinamine, and (h) 2-acetylaniline.



anti-cold tablet. (b) Total ion chromatogram and (c-g) extracted ion chromatograms for (c) chlorpheniramine maleate (M₂, m/z 275) (d) methylephedrin hydrochloride (M₁, m/z 180), (e) ethoxybenzamide (M₄, m/z 152), (f) acetaminophen (M₃, m/z 166), and (g) noscapine (M₅, m/z 414). (h, i) mass spectra corresponding to the respective spot positions on the TLC plate: (h) methylephedrin hydrochloride and chlorpheniramine maleate (i) acetaminophen, ethoxybenzamide, and noscapine.

Conclusion

They have demonstrated that ELDI-MS can be used to directly characterize molecules separated on TLC plates

Advantage over ESI and MALDI

Can be used with almost all mass analyzers