Developing an LC-FAIMS-MS strategy for non-targeted urinary metabolomic studies

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FAIMS: The principles

- Field asymmetric waveform ion mobility spectrometry (FAIMS) is a technique that separates ions in the gas-phase based on differences in their mobility while subject to alternating high and low electric field as they travel through a pair of electrodes in a flow of buffer gas at atmospheric pressure (Figure 1).
- Different dispersion field (DF) and compensation field (CF) conditions allow the transmission of selected ions to the mass spectrometer.
- FAIMS separation is highly orthogonal to liquid chromatography (LC) and mass spectrometry (MS), giving third dimension to the acquired data without adding additional time to the analysis.

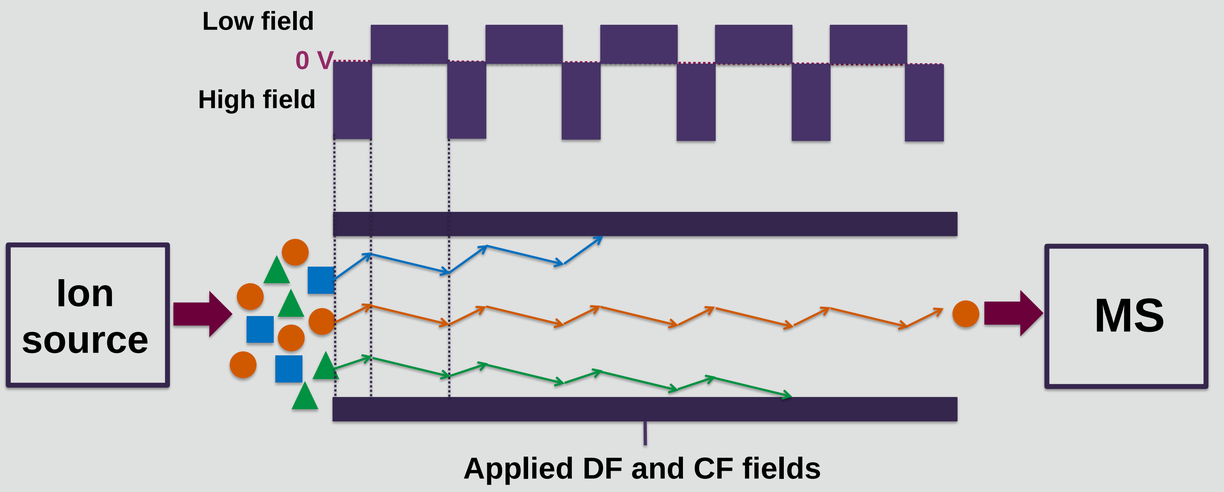

Figure 1. The scheme of ion transmission through the planar FAIMS into the MS.

Methodology

- The chip-based FAIMS device (Owlstone ultraFAIMS, Owlstone Medical Ltd, Cambridge, UK) consisting of multiple electrode pairs (100 µm gap x 700 µm long) was located in the front of the TOF MS heated capillary inlet, behind the modified electrospray source (Fig. 2b).[1]

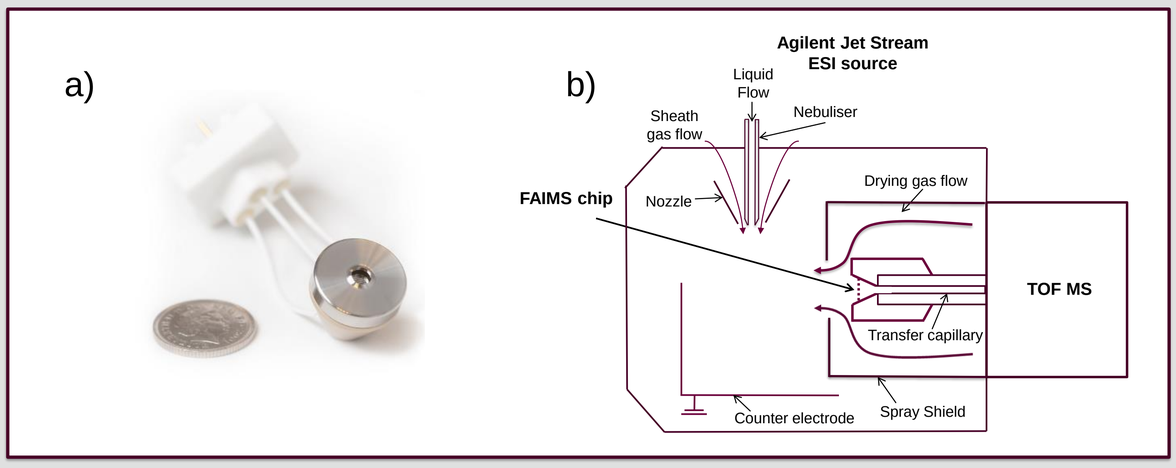

Figure 2. a) FAIMS chip, b) the scheme of FAIMS-MS interface (Agilent 6230 TOF).

- Urine sample preparation was performed by protein precipitation with acetonitrile.
- Chromatographic separation for non-targeted metabolomics approach was carried out on a HILIC column with a total run time of 13 minutes.
- Full CF scans (-0.5 to 3.5 Td) were acquired within 1 seconds, resulting in 19 CF steps + one step for re-initialization.
- MS scan rate of 20 scans/sec allows a mass spectrum to be acquired at every CF step.
- Data were processed using in-house Matlab script and Simca.

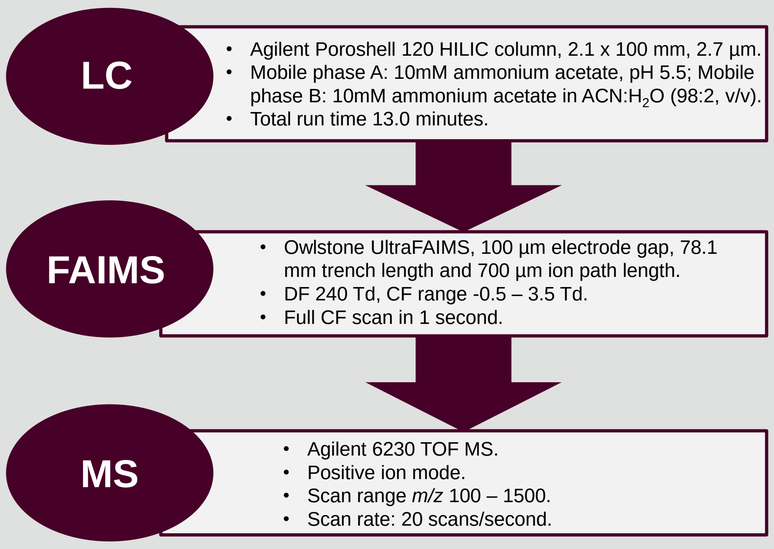

Figure 3. LC-FAIMS-MS parameters.

Results

- Full scan LC-FAIMS-MS was applied to the non-targeted metabolomic analysis of urine samples.
- FAIMS increases peak capacity by separating isobaric and isomeric ions not resolved by LC-MS. Figure 4 presents the response for *m/z* 137.07. In LC-MS, one peak was detected (Fig. 4a) at a retention time (RT) of 7.85 min. However, LC-FAIMS-MS analysis resulted in the detection of two peaks, at CF 0.17 Td (RT 7.88 min) and CF 1.5 Td (RT 7.80min), in the FAIMS CF spectrum (Fig. 4b).

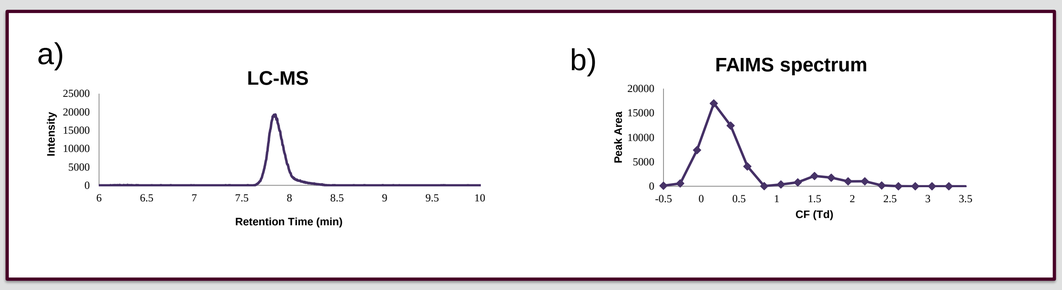

Figure 4. a) LC-MS extracted ion chromatogram (*m/z* 137.071), b) CF spectrum with two peaks for *m/z* 137.07.

- Nested LC-FAIMS-MS data sets were acquired for aged and fresh urine samples and subjected to bioinformatics analysis.
- The principal component analysis (PCA) (Fig. 5) shows the clear separation of aged and fresh urine samples.

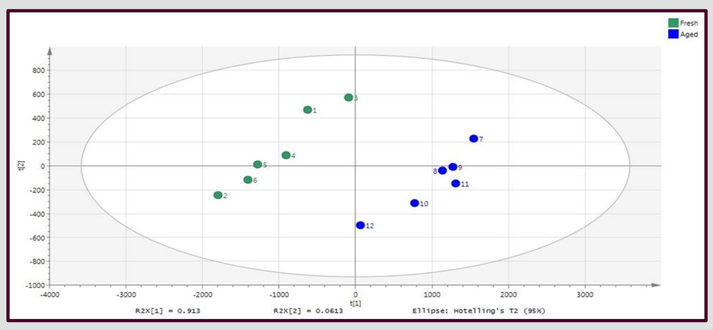

Figure 5. Scores plot for of urine samples at CF 0.83 Td.

Conclusions

- Combining full scan FAIMS with LC-TOF MS increased the number of detected peaks.
- The applied method allows for the separation of fresh and aged urine samples using multivariate statistics.

Acknowledgements

- The authors thank Owlstone Ltd. for financial and technical support.

REFERENCES

1. Arthur, KA, Turner, MA, Reynolds, JC, Creaser, CS.; Anal. Chem., 2017, 89, 3452-3459.

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