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MULTI-MATRIX SAMPLE PREPARATION FOR FAMES PROFILING BY TWO-DIMENSIONAL GAS CHROMATOGRAPHY (GCxGC)

Axel RAUX¹, Justine MASSIAS¹, Emmanuelle BICHON¹, Yann GUITTON¹, Bruno Le BIZEC¹



LABERCA, Oniris, INRAE, Nantes, France
Fax : +33 (0)2 40 68 78 78 - Tél : +33 (0)2 40 68 78 80
laberca@oniris-nantes.fr

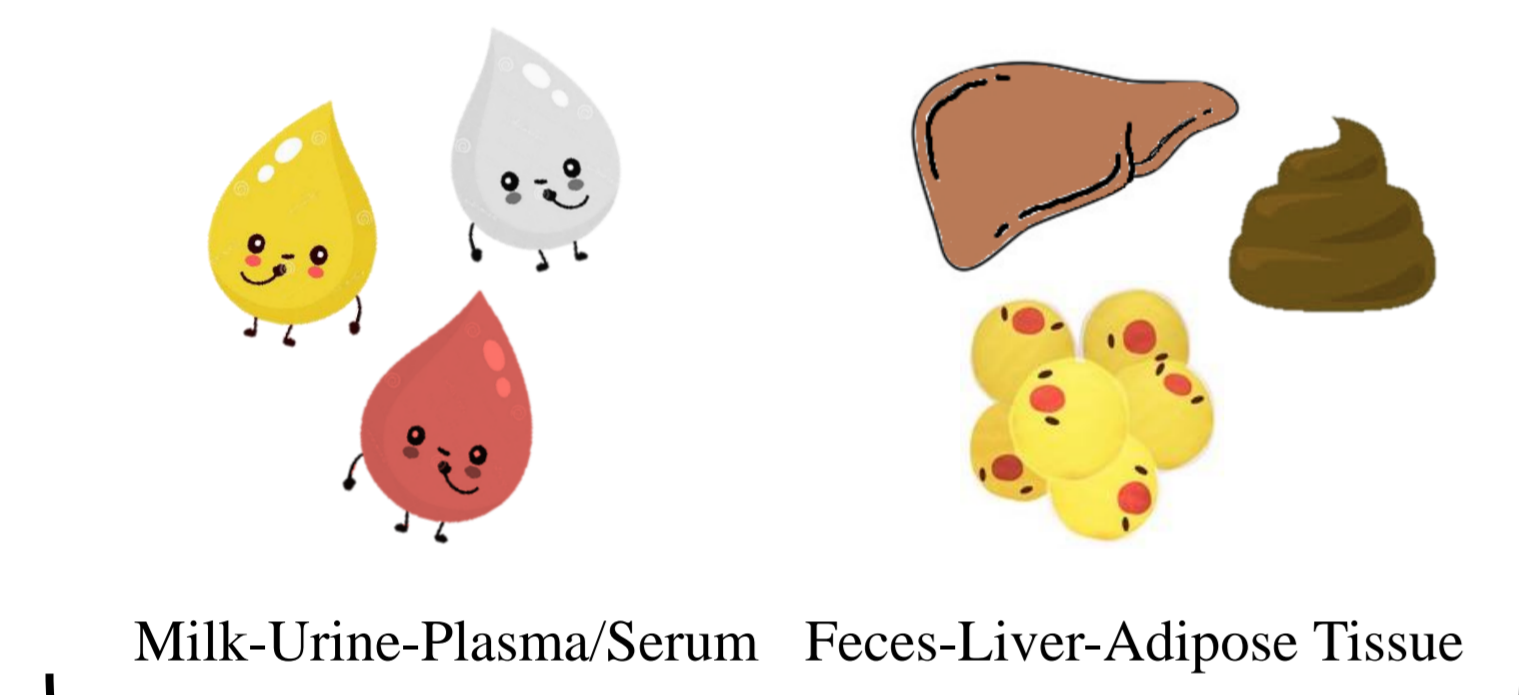


Introduction

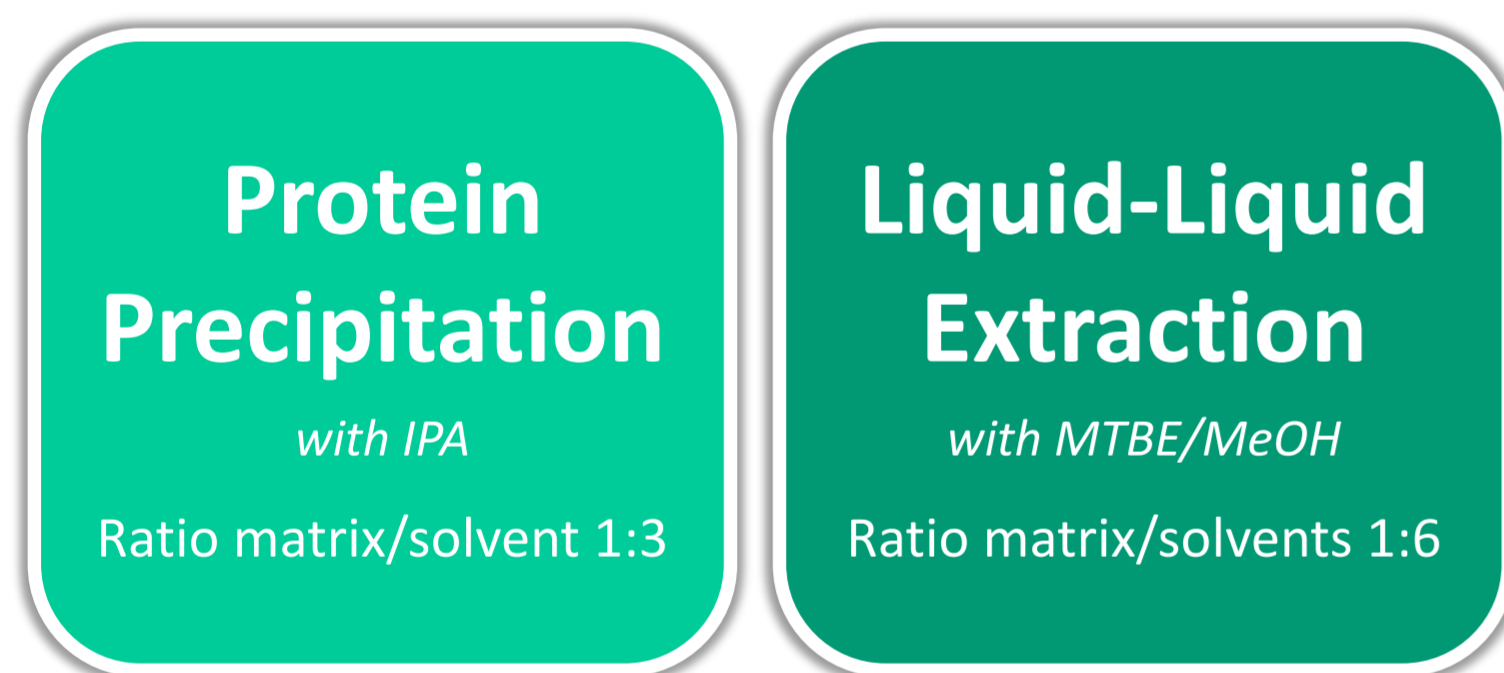
Fatty acids methyl esters (FAMES) analysis is of a special interest for monitoring the metabolic health of a population. As lipidomic studies are often based on the separation of the different lipid classes by LC or GC-HRMS, it appears that the use of a more resolutive chromatographic technique, like two-dimensional gas chromatography (GCxGC), can be beneficial to enhance resolution and improve FAMES discrimination in lipidomic studies. GCxGC coupled with MS offers an additional separation dimension allowing lipids isomers differentiation in complex matrices.

Therefore, GCxGC was assessed for FAMES analysis in several matrices. Indeed, liquid matrices (milk, urine and plasma/serum) as well as solid matrices (lyophilized liver and feces, and adipose tissue) were prepared following two protocols performed in lipidomic studies.

Experimental



2 sample preparations for each



Supernatant recuperation (25µL)
Internal standards (lipids) addition
Evaporation at 25°C under nitrogen flow

Derivatization

25µL MOX (2mg/mL) 30min ; 70°C ; 800rpm
25µL MSTFA 30 min ; 37°C ; 800rpm

GCxGC-TOF (Markes BenchTOF-Select-eV)

1D : BPX70 (60m ; 0.25mm ; 0.25µm)
2D : DB-1MS (1,5m ; 0.25mm ; 0.10µm)
Electron Ionization 70eV and FID



Raw data acquisition
MS-TOF data
FID data

Baseline Compensation

Deconvolution

Library Matching
NIST database

dataMatrix
mz, RT1, RT2,
intensity, match score

RStudio

Quantification : calculation,
plots

Results and Discussion

Preliminary studies were conducted on the mix FAMES 37 (from Supelco). Sufficient chromatographic resolution was obtained allowing the identification with NIST matching of 36 FAMES, only missing C4:0 which is not retained with this gradient. Gradient optimizations occurred however, no improvement was obtained.

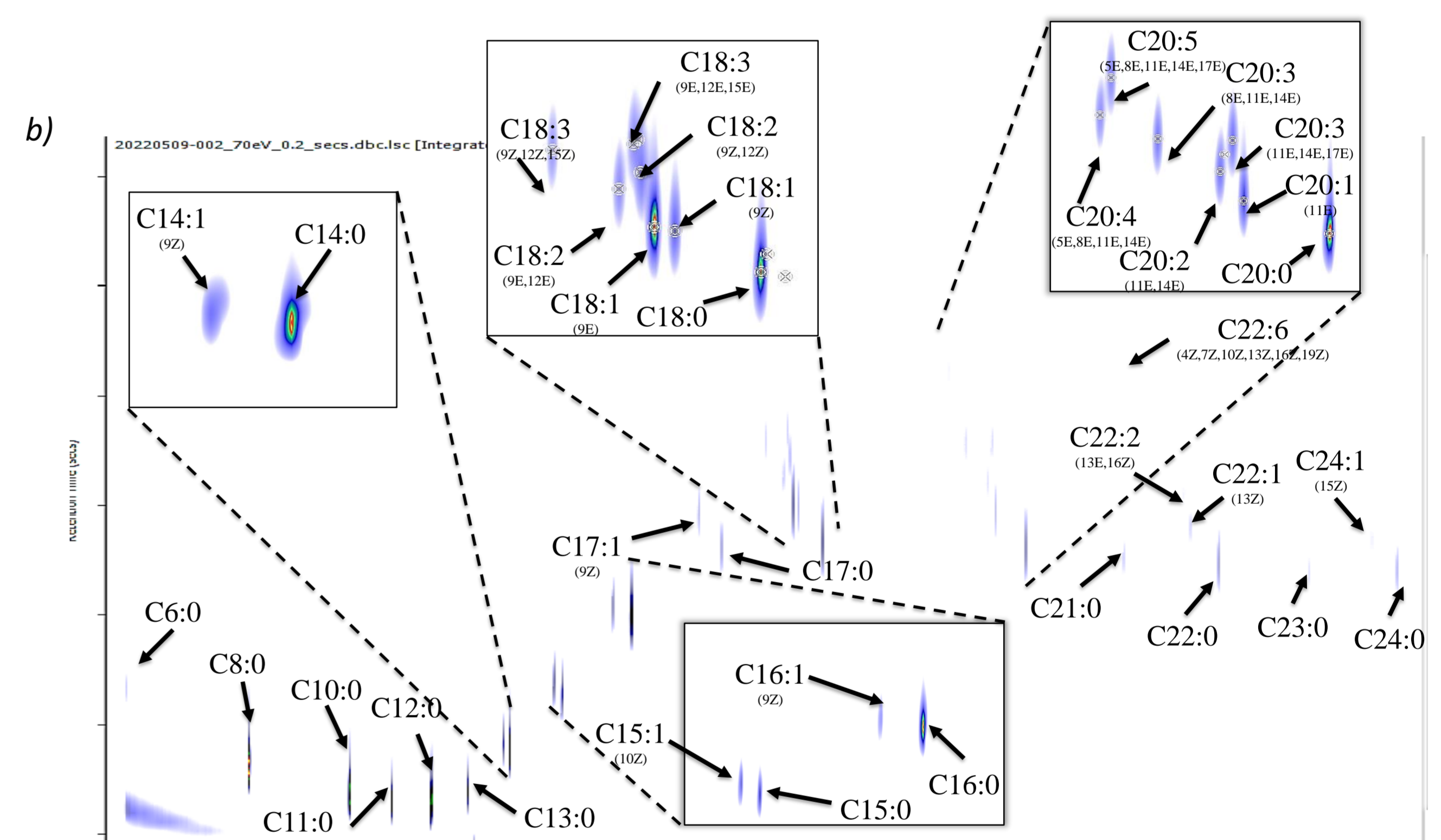
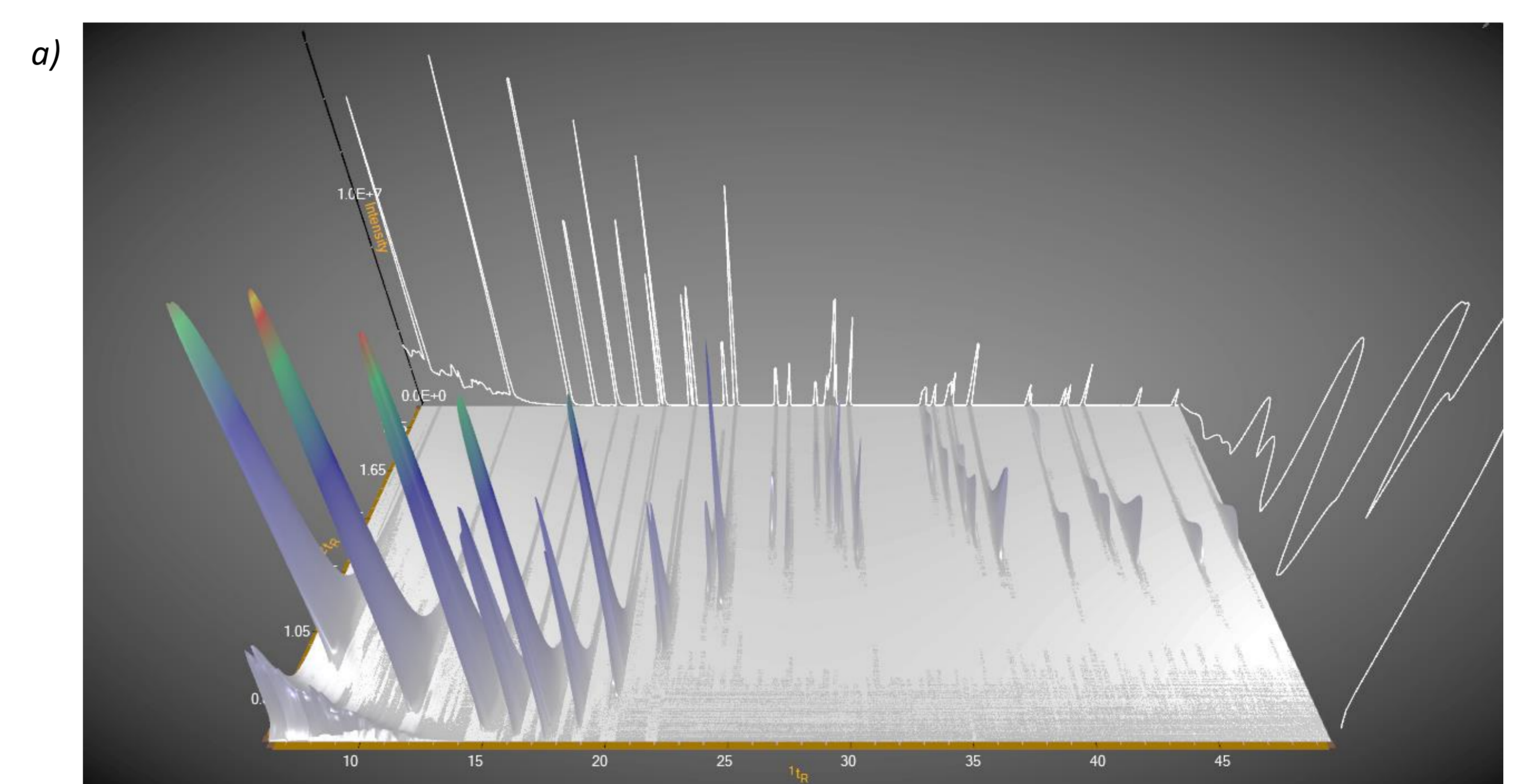
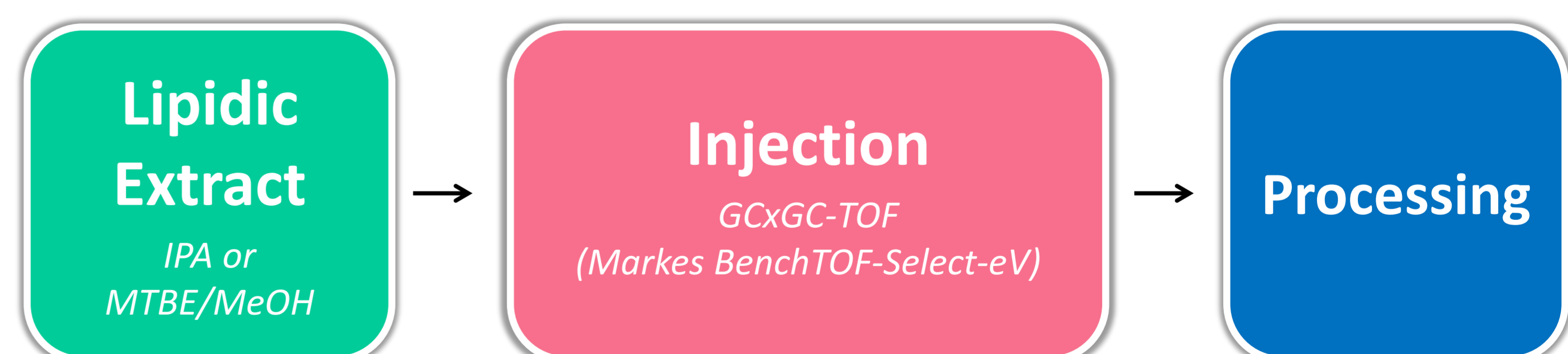


Figure: EI-MS (70eV) spectra of mix FAMES 37 in a) 3D plot b) 2D plot with NIST annotation

For the matrices, no signal could be detected. As standards were injected between the samples, and successfully detected, sample preparation may be involved in the issue. Next injections will be performed without prior derivatization.



Conclusion

Two-dimensional chromatography hyphenated with TOF-MS was evaluated for FAMES detection. Sufficient chromatographic resolution was successfully achieved for discriminant FAMES identification performed with NIST database. The same method was applied to several matrices. However, no detection signal could be properly obtained. A sample preparation issue could be involved here. This underlines the significance of sample preparation in GCxGC-MS studies. Further tests will be conducted for FAMES detection in the matrices and then applied to lipidomic studies.