SEPARATION, DETERMINATION AND COMPOSITION PROFILE OF LIPIDS IN BIODIESEL USING HYPHENATION OF GRADIENT-HPTLC WITH FLUORESCENCE DETECTION BY INTENSITY CHANGES AND MASS SPECTROMETRY

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Introduction

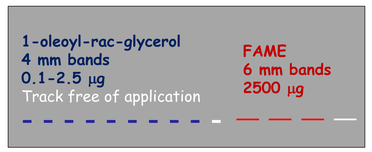
❖Biodiesel is a lipid-based renewable fuel mostly composed of methyl esters of fatty acids (FAME). Detailed knowledge of lipid profiles and structures of each class **[mono-(MG), di-acylglycerides (DG), and fatty acids (FA)]** is required for finding impurities affecting biodiesel operation.

❖HPTLC is a suitable technique for these types of samples. As all compounds in a sample are stored on the plate after chromatographic development, a quantitative analysis may be possible.

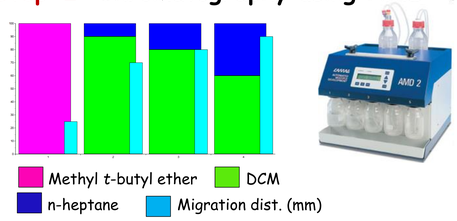
❖HPTLC is now a fully automated and computer-controlled analytical technique that makes it possible to design original hyphenated instrumental methods well suited to a particular analytical issue.

Separation, detection and identification of lipid classes in FAMEs using a hyphenated AMD-FDIC-ESI-MS system

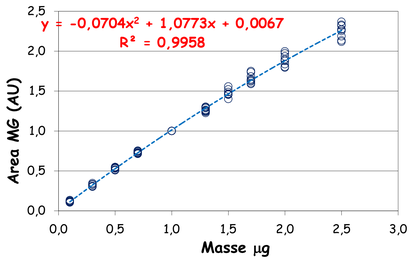
Step-1: Standard and FAME were applied on a silica gel plate



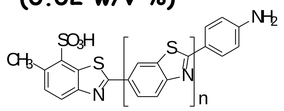
Step-2: Chromatography using AMD



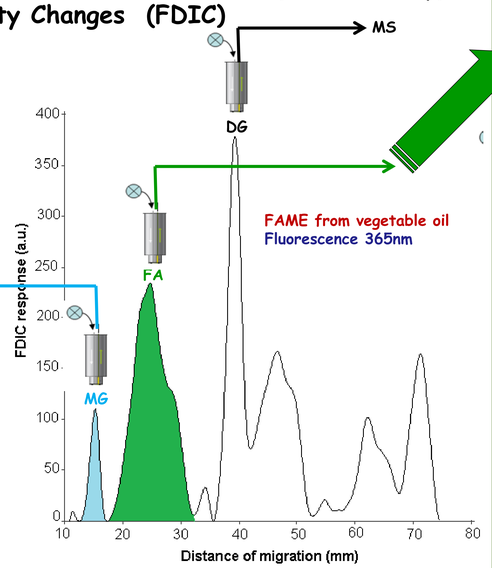
Step-5: Quantitative determination of MG in FAMEs



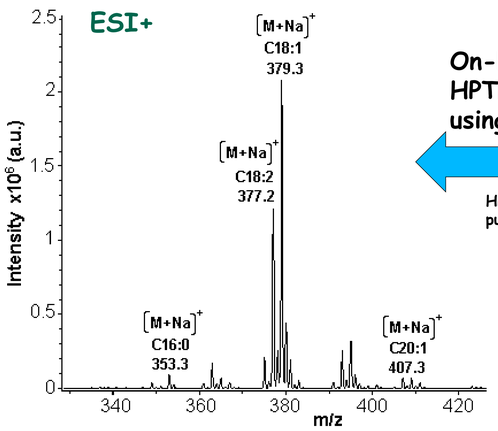
Post-impregnation: Primuline in MeOH (0.02 w/v %)



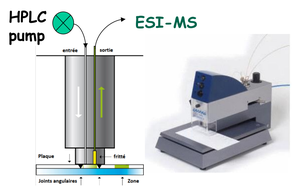
Step-3: Fluorescence Detection (densitometry) by Intensity Changes (FDIC)



Step-4: Identification of species in MG peak



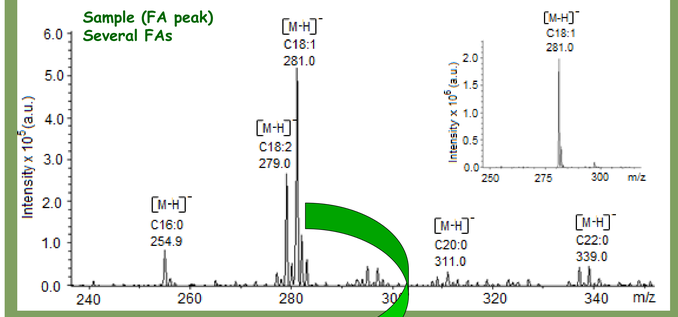
On-line, on-plate HPTLC-MS extraction using CAMAG interface



Lipid profile of each class peak
Structural identification of FAs in FAMEs directly from the plate

ESI-MS

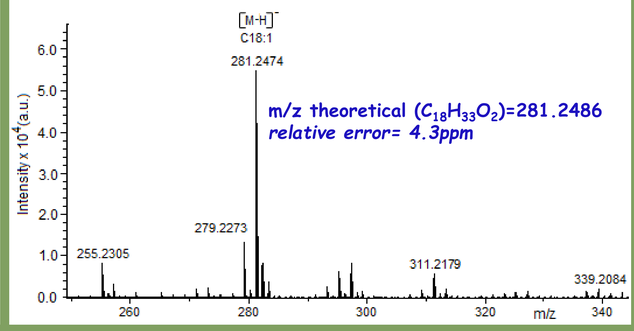
Ion-Trap ESI-



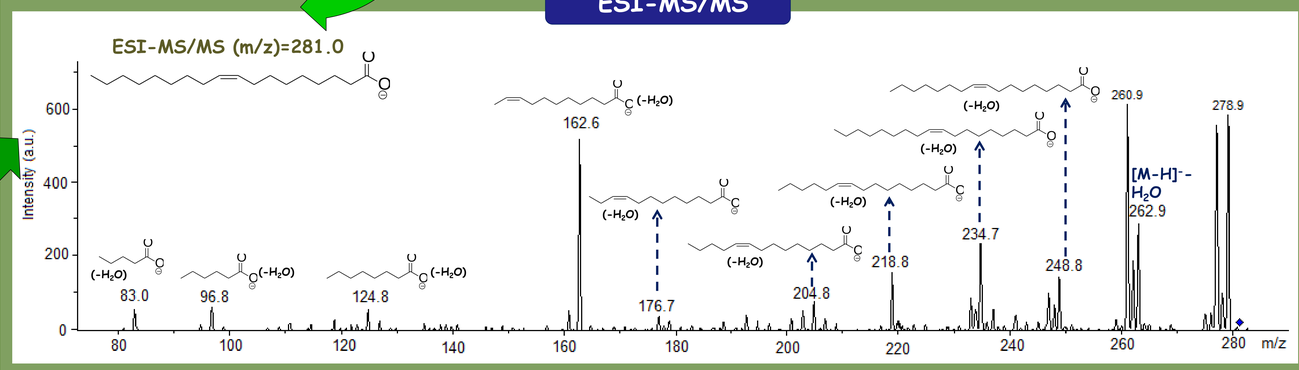
Standard Oleic Acid

High Resolution ESI-MS

Micro QToF



ESI-MS/MS



Conclusions

❖ Lipids are separated in classes and from diesel using HPTLC-AMD. Classes can be quantified in BX (X>5) by fluorescence densitometry. Method is sensitive to determine, for example, MG according to biodiesel standards (<0.8 wt%).

❖ ESI-MS/MS and HR ESI-MS spectra can be obtained directly from the plate. FA in negative mode; MG and DG in positive mode.

❖ The whole analysis is done on a unique silica gel plate.

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