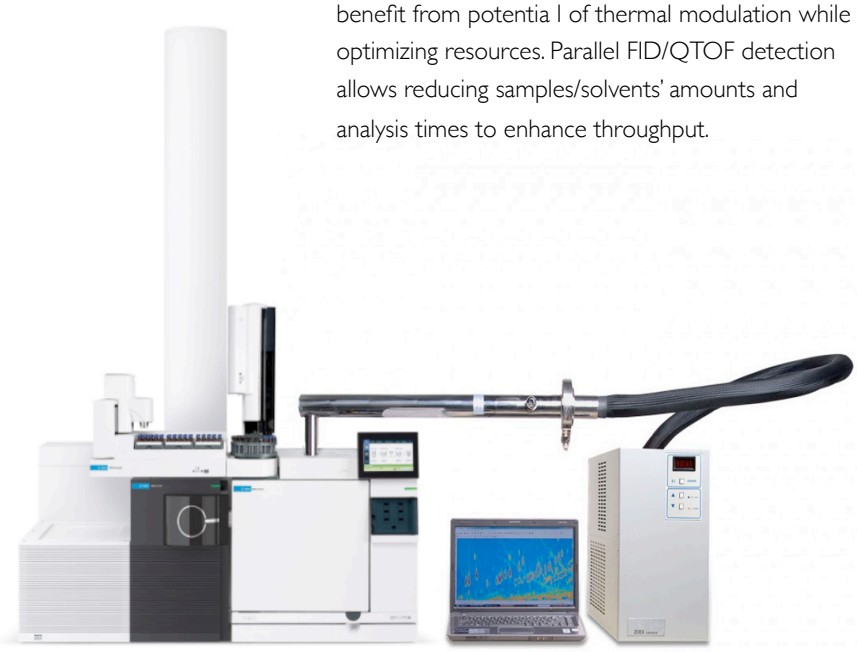
2D speciation of MOSH/MOAH with GC×GC-FID/QTOF

Mineral oil (MO) hydrocarbons, which can be divided in saturated (MOSH) and aromatic (MOAH), are a well-known source of food contamination from raw material to harvest, processing, or packaging. This issue has been for years at the center of attention in Europe for its potential impact on consumers' health. In particular the presence of MOAH is linked to increased risks in terms of toxicity due to their suspect carcinogenicity and genotoxicity, especially when species with 3 or more aromatic rings are present.

Current instrumental platforms, based on the LC-GC-FID hyphenation, allow individual quantification of the aliphatic and aromatic contaminant fractions. Nevertheless, the task is often challenging due to matrix complexity and the presence of interferences. Moreover, FID detection alone does not permit to obtain qualitative information about the type of MOSH or MOAH present in order to reach valuable conclusions on toxicity. Laboratories tasked with performing MOSH/MOAH analysis need, for contaminated samples, access to advanced, more insightful investigation tools for improved characterization of both fractions.

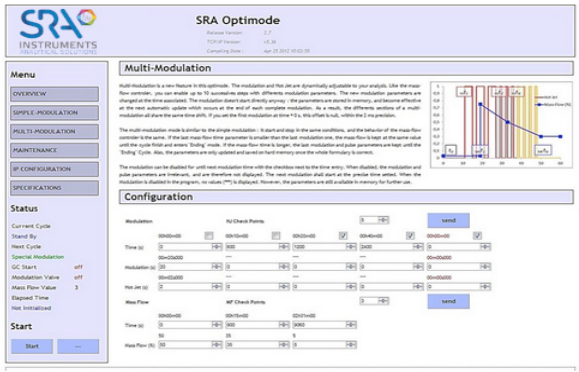
SRA Instruments developed configuration and analytical methodology to match the challenging qualitative and quantitative needs of MOSH/MOAH analysis. The solution is based on a two-dimensional GC×GC platform in combination with FID detection and High Resolution Mass Spectrometry. This technique significantly increases the capacity to characterize the two fractions preliminarily separated by HPLC thanks to its superior chromatographic resolution, offering a more detailed classification of the hydrocarbon profiles.

Zoex thermal loop modulator with close-cycle refrigeration (no liquid nitrogen needed) grants excellent chromatographic performance combined to high flexibility, hereby assisted by the Optimode, a product of SRA Instruments developed to fully benefit from potentia l of thermal modulation while optimizing resources. Parallel FID/QTOF detection allows reducing samples/solvents' amounts and analysis times to enhance throughput.

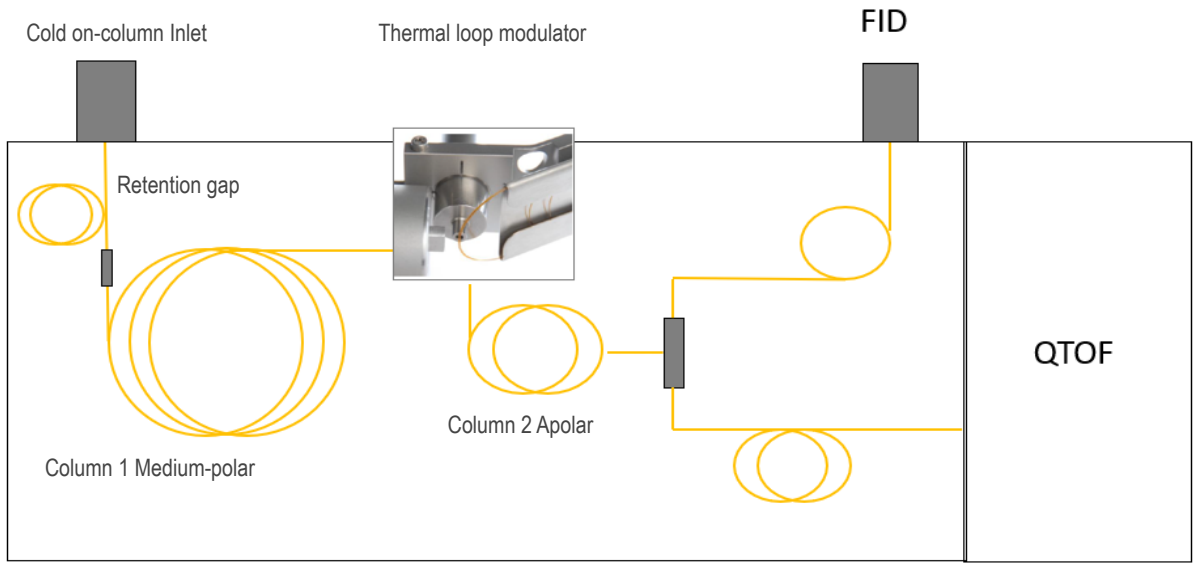


SRA GC×GC-QTOF solutioni:

- *Agilent GC 8890 - QTOF 7250*
- *Zoex thermal loop modulator ZX2 (o liquid nitrogen needed)*
- *SRA Optimode*



SRA OPTIMODE Control Interface

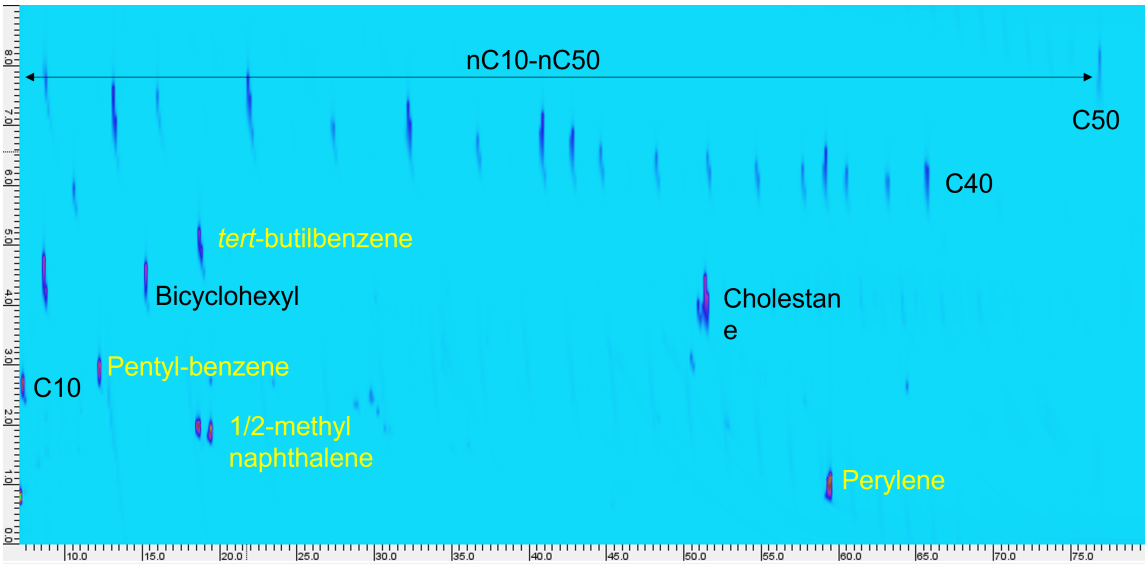


SRA GC×GC-QTOF solution diagram.

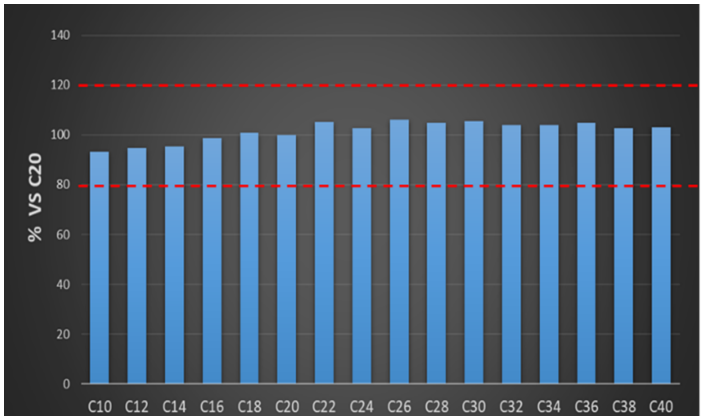
Analytical performance

Instrumental set-up and method parameters were finely tuned to balance parallel detection and achieve consistent FID e QTOF signals. This ensures a seamless translation of information between the detection channels in order to facilitate efficient integration of qualitative and quantitative workflows.

The absence of discrimination was successfully verified for aliphatic and aromatic components in the C10-C50 range. Recovery, calculated for FID response relatively to alkane C20 picked as reference, was in all instances between 80 and 120%.

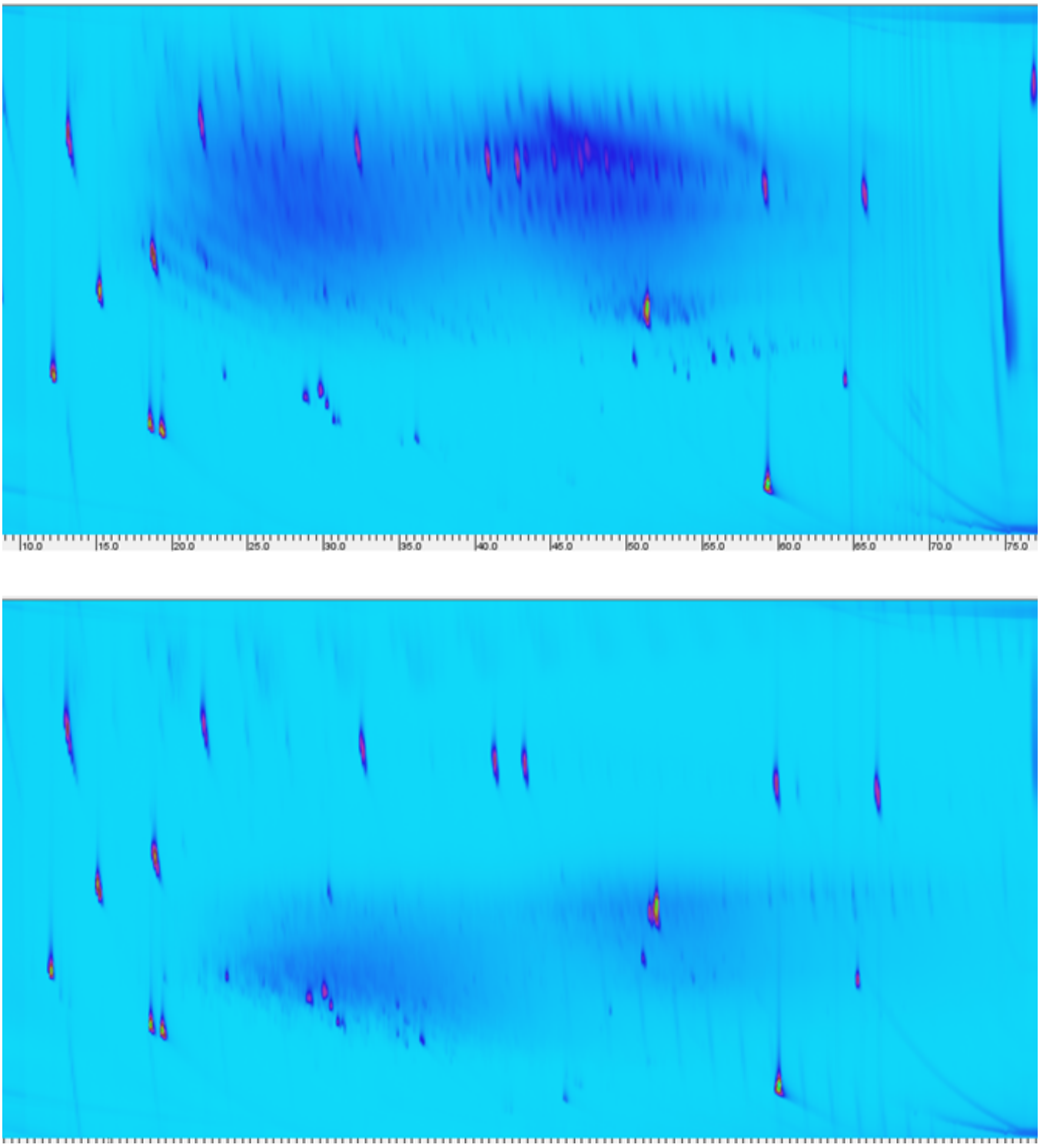


2D plot for linear alkanes C10-50 and aliphatic (black) and aromatic (red) markers

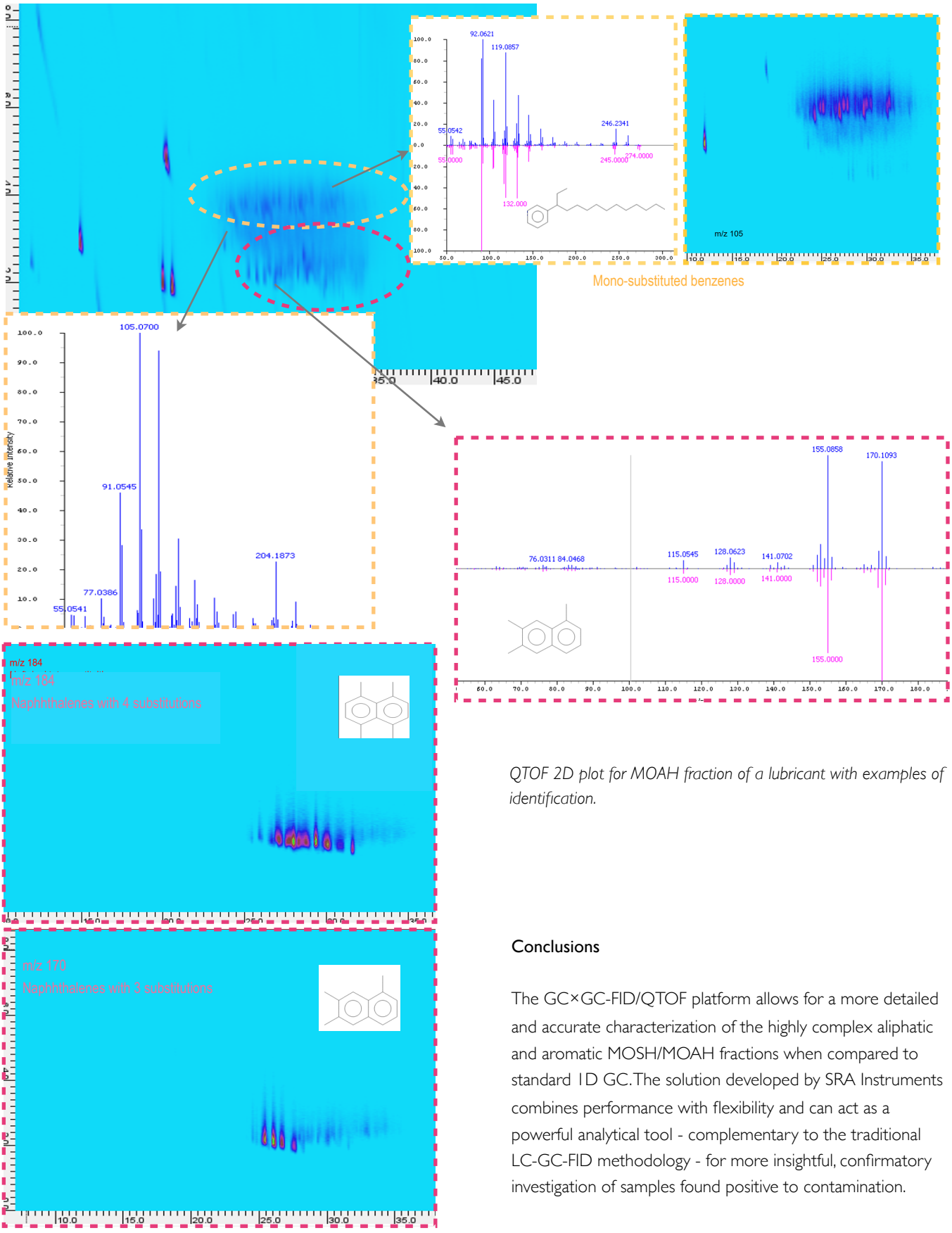


Discrimination test

The 2D plots obtained for real matrices, i.e. samples and lubricants possibly linked to contamination – show the value added by the two-dimensional approach. Compound and chemical classes are distributed across the separation space according to a chemical logic determined by chemical-physical properties and their affinity towards stationary phases. Separation is much more detailed and insightful, allowing for more accurate characterization.
The high complexity degree of these hydrocarbon mixtures makes the chromatographic patterns highly specific thus ideal for mapping. This can be particularly useful for fingerprinting analysis aimed at a quick, reliable identification of contamination origin.



FID 2D plots of MOSH and MOAH fractions separated by HPLC and spiked with alkanes and markers



QTOF 2D plot for MOAH fraction of a lubricant with examples of identification.

Conclusions

The GC×GC-FID/QTOF platform allows for a more detailed and accurate characterization of the highly complex aliphatic and aromatic MOSH/MOAH fractions when compared to standard 1D GC.The solution developed by SRA Instruments combines performance with flexibility and can act as a powerful analytical tool - complementary to the traditional LC-GC-FID methodology - for more insightful, confirmatory investigation of samples found positive to contamination.

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