# Analysis of FAME's by GCxGC-TOFMS

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## 1. Introduction

Four samples were analyzed by GCxGC-TOFMS for fatty-acid methyl esters (FAME's). Advanced software features were utilized in data processing.

## 2. Instruments and Methods

In this study, measurements were made with a LECO Pegasus® 4D GCxGC-TOFMS system. This system consists of an Agilent 6890 gas chromatograph equipped with a LECO dual-stage, quad-jet thermal modulator between the primary and secondary columns, and a LECO Pegasus HT Time-of-Flight Mass Spectrometer (TOFMS) as a detector. For this study, the primary analytical column was a 30.0 m x 0.25 mm ID x 0.25 µm df RTX-1. The secondary column was a 1.50 m x 0.10 mm ID x 0.10 µm df DB-17ms for samples #1 and #2. For samples #3 and #4, the secondary column was a 1.0 m x 0.10 mm ID x 0.10 µm df DB-17ms. Helium was used as the carrier gas at a constant flow of 1.0 mL/min. The transfer line to the TOFMS consisted of the last 20 cm of the analytical column. The detector was optimized to have a S/N ratio of ≥10 for a 2 pg/µL injection of hexachlorobenzene with a detector voltage of 1650 volts. For these non-quantitative analyses, each FAME sample was diluted in iso-octane. Injections were 1 µL using the splitless mode of the split/splitless inlet.

## 3. Results

All four of the samples were data processed for a S/N ratio of greater than or equal to 250:1. A contour plot of sample #1 is shown in Figure 1.

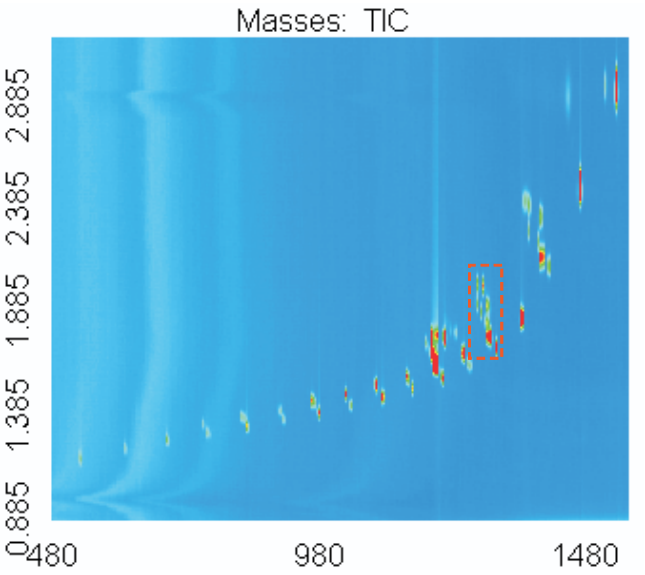


***Figure 1: A contour plot of FAME sample #1. The X-axis is retention time on the primary column. The Y-axis is retention time on the secondary column.***

The area indicated by the orange-dashed box in Figure 1 is shown as a 3D surface plot in Figure 2.

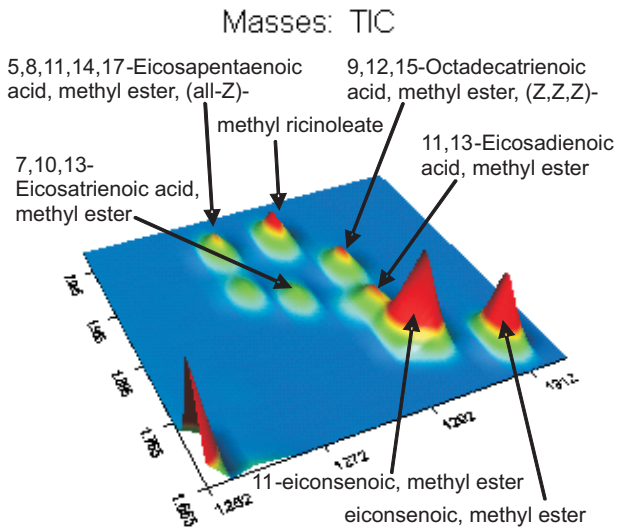


***Figure 2: A 3D surface plot of the area indicated by the orange-dashed box in Figure 1. Identifications of select peaks are included.***

A contour plot of sample #4 is shown in Figure 3. Sample #4 contains cholesterol-related compounds.

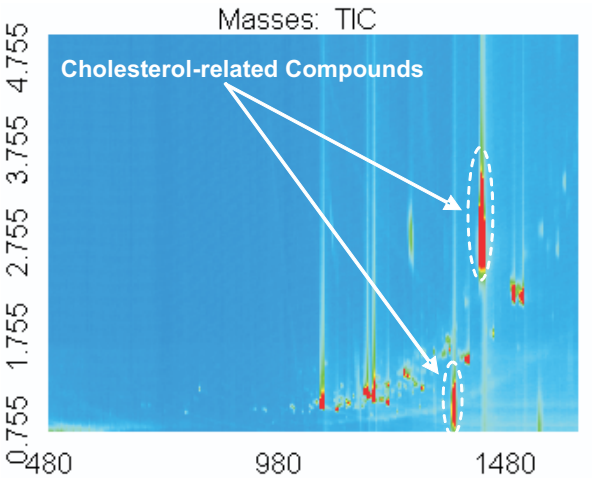


***Figure 3: A contour plot of sample #4 with regions of cholesterol-related compounds***

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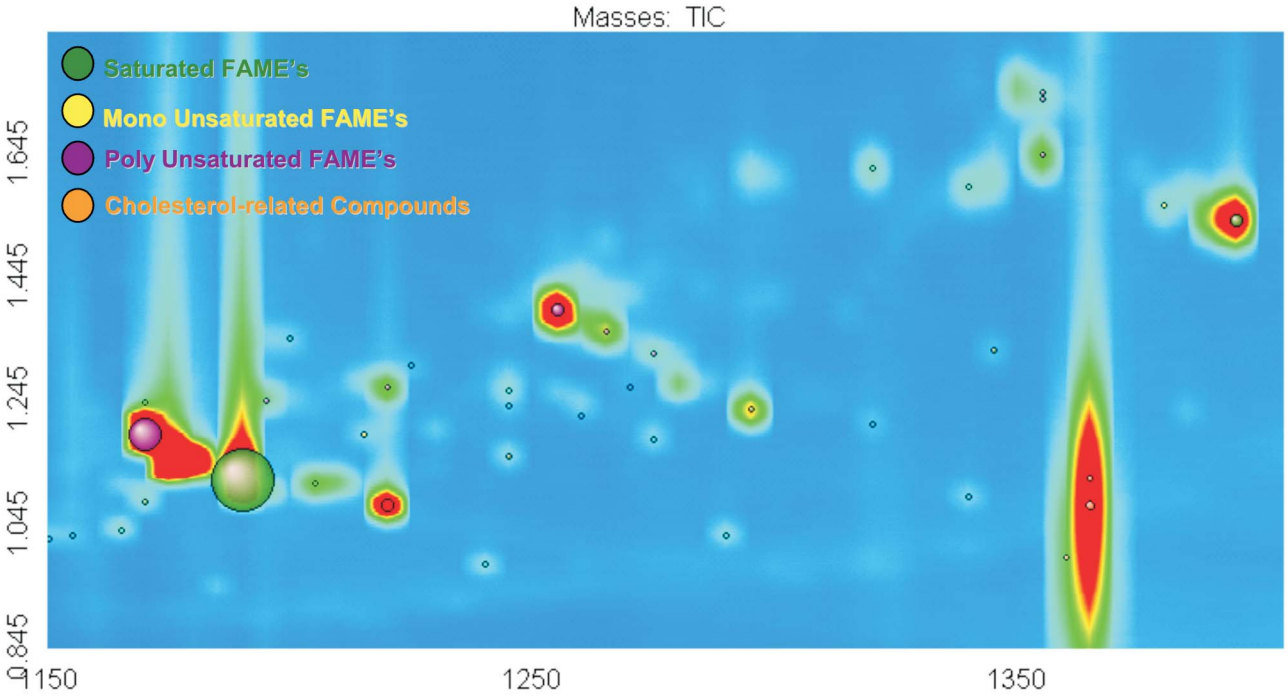


***Figure 4: A zoomed in region of sample #4 showing "peak bubbles w/ classifications".***

A useful feature of ChromaTOF® software, when combined with GCxGC data, is the use of classifications. A classification takes advantage of the structured nature of a GCxGC chromatogram. User-specified regions can be assigned to different classes. Another feature is called "peak bubbles". Peak bubbles are a different way to visualize information relating to a peak. Instead of a peak marker (a black dot) identifying the peak apex; a circle—centered on the peak apex and its radius, corresponding to the intensity of the ion being displayed—is used. For TIC and AIC, the intensity of the Unique Mass is used to determine the bubble radius. These peak bubbles can also be filled in with a color relating to the classification it falls in. Peak bubbles of peaks that do not fit into a classification are left uncolored. When looking at a GCxGC contour plot, use of the color-coded peak bubbles allows the analyst to quickly observe patterns in the chromatogram. An example of a region of sample #4 displaying peak bubbles and Classifications is shown in Figure 4. Four different classifications are visible in this region. These classifications are as follows: the saturated FAME region (green), the mono-unsaturated FAME region (yellow), the poly-unsaturated FAME region (purple), and the cholesterol-related compounds region (orange). Peaks can potentially belong to more than one classification zone. In that case, the peak bubble will appear striped in the colors of the classification zones it is associated with. Use of peak bubbles and Classifications are not detector dependent. They may be used (in ChromaTOF) for both GCxGC-TOFMS and GCxGC-FID\ECD data. An advanced feature called Scripting can also be used with Classifications on GCxGC-TOFMS data to further sort peaks in a classification zone by processing peaks, within a classification zone, by their mass spectral data.

## 4. Conclusions

GCxGC is well-suited to the analysis of samples containing fatty acid methyl esters. The structured nature of GCxGC chromatograms allows for visual identification of the different classes of these compounds. When combined with data processing by ChromaTOF software, patterns within the data are easier to observe. Use of Classifications and peak bubbles features within ChromaTOF allow the analyst to determine which classification region a peak belongs to, as well as being able to make an estimate of peak intensity, which is related to the radius of the peak bubble. The use of the Scripting feature, when used with GCxGC-TOFMS data, would enable the analyst to rapidly obtain and sort more information about the analytes in the sample.

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