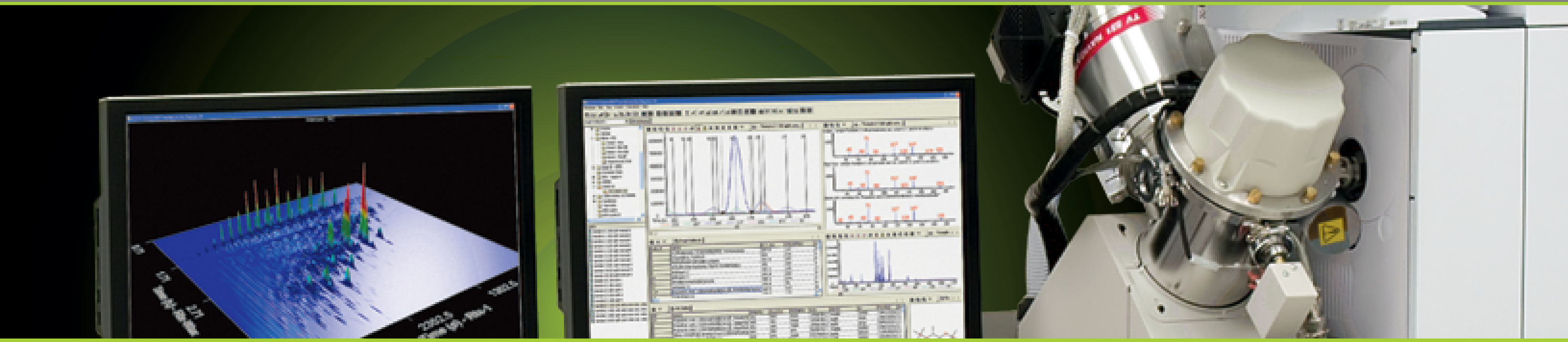
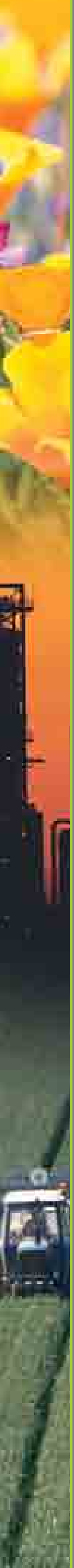


PEGASUS® 4D GCxGC TOFMS

Delivering the Right Results



LECO's PEGASUS® 4D GCxGC-TOFMS

The Ultimate Analytical Instrument

Pioneering GCxGC technology, combined with the Pegasus TOFMS and ChromaTOF® software provides today's researchers with Four Dimensions (4D) of analytical resolving power for more complete sample analysis.

1) 1st dimension chromatographic separation
2) 2nd dimension chromatographic separation
3) Mass Spectral data from the Pegasus TOFMS
4) True Signal Deconvolution® only from ChromaTOF

ChromaTOF®

Introducing the first-ever low maintenance, consumable-free thermal modulator

For complete patent information, see specification sheet 209-183-001.

Complex Sample Analysis

What is GCxGC?

Comprehensive two-dimensional gas chromatography (GCxGC) is the answer for complete analysis and characterization of complex samples. **As opposed to heart-cutting**, GCxGC passes all effluent from the primary column through the secondary column, whereby maximizing sample resolution throughout the entire analysis. Heart-cutting can only accomplish this in a narrow, pre-determined time window. Hundreds-to-thousands of individual heart-cutting analyses would be required to accomplish what LECO's GCxGC delivers to you in one analysis.

GCxGC adds a second dimension of chromatographic resolution to the sample analysis. This is accomplished by using two orthogonal separation phases (such as non-polar and polar) within a single analysis. The use of these two separation mechanisms expands the chromatographic plane—thus creating additional peak capacity in which peaks can be resolved.

LECO's GCxGC Thermal Modulator is the key to the enhancement of chromatographic resolution obtained by the Pegasus 4D system. The modulator, placed between the two columns, consists of a robust quad-jet system that creates two distinct trapping zones that ensure all of the effluent from the first column is properly focused prior to thermal release into the second column. A secondary oven is used for optimization of the second dimension separation.

Diagram of GCxGC-TOFMS Instrument

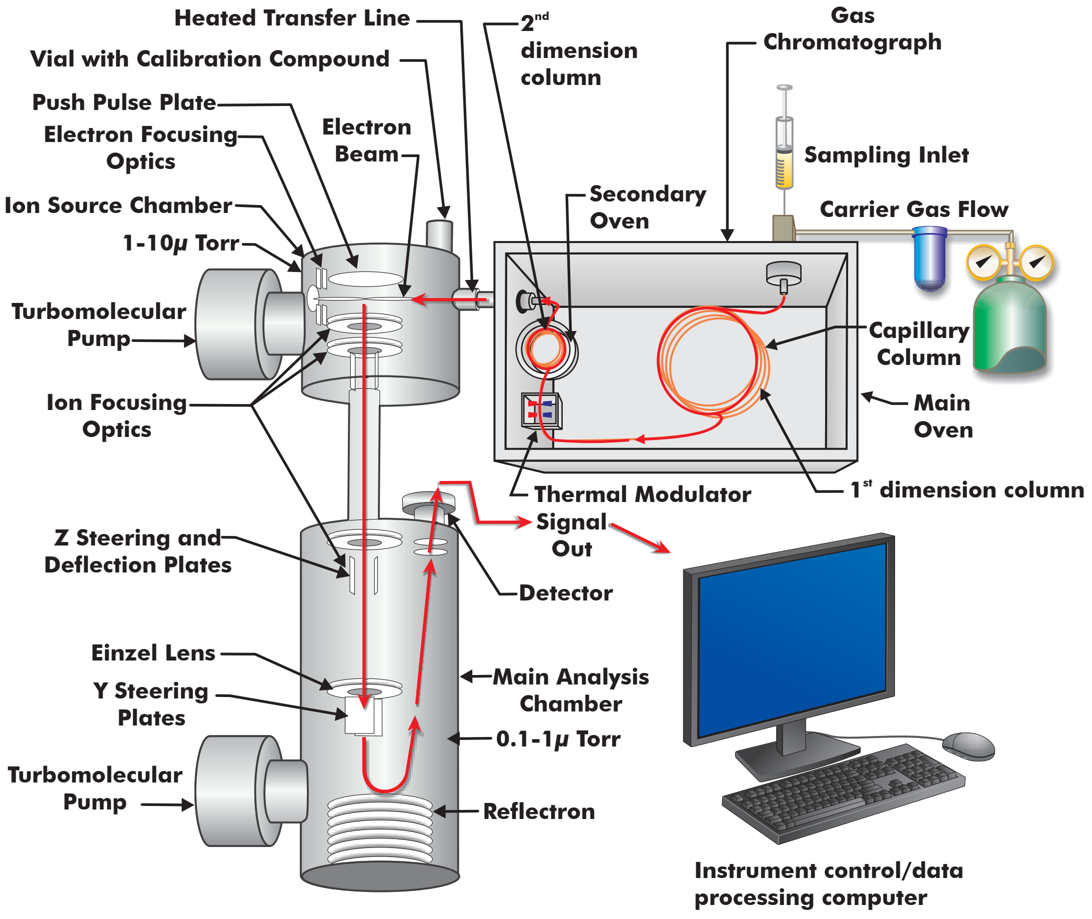
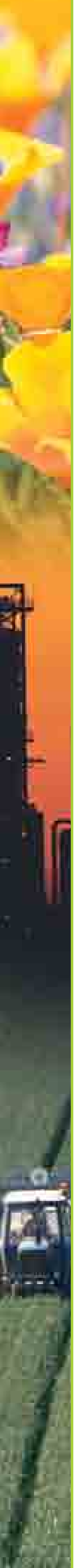


Figure 1. The entire modulator is mounted inside the primary GC oven. Control of the GC autosampler, GC, LECO's GCxGC Thermal Modulator, and the Pegasus TOFMS is fully integrated within a single computer using LECO's ChromaTOF software.



Complex Sample Analysis

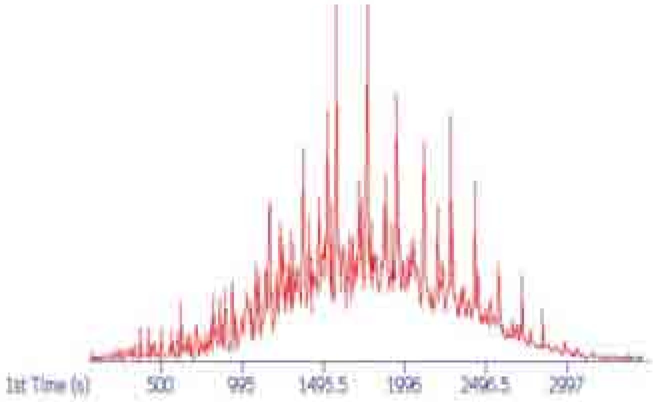


Figure 2. Traditional GCMS analysis of petroleum (678 analytes detected).

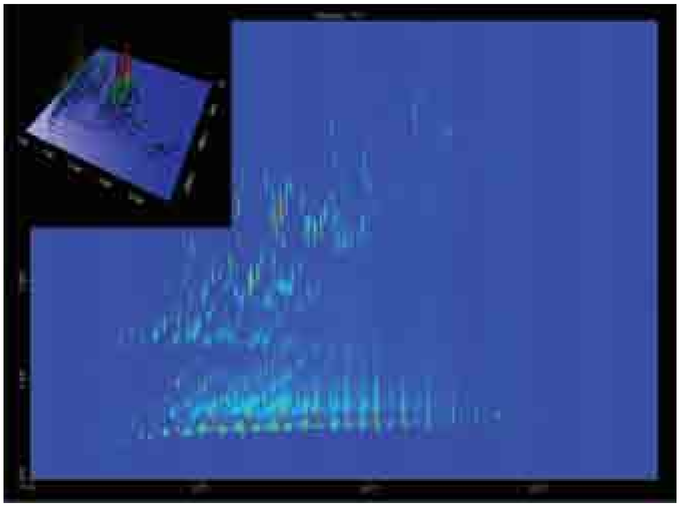


Figure 3. GCxGC-TOFMS analysis of petroleum (1,246 analytes detected).

The GCxGC Advantage

The demands of complex samples (such as the petroleum sample shown in Figure 2) quickly overwhelm traditional GCMS analysis techniques.

LECO provides the solution for complex sample analysis with the Pegasus 4D, the pre-eminent MS detector for Comprehensive Two-Dimensional GC (GCxGC). The High-Definition sample resolving capabilities of the Pegasus 4D, and the data processing features of ChromaTOF software offer every laboratory the ability to characterize and quantify even the most challenging samples.

Figures 2 and 3 are petroleum samples analyzed by both GC-TOFMS and GCxGC-TOFMS. Figure 2 clearly shows the complexity of the sample. Without the resolving capabilities of the Pegasus 4D, accurate characterization of the mixture would be impossible.

- Spectral collection rates up to 500 full-range mass spectra/second (500 Hz)—the Pegasus is the only MS detector capable of true Comprehensive multi-dimensional chromatography.
- Cryo-focusing prior to release on secondary column provides up to a ten fold increase in analyte detectability.
- True Signal Deconvolution®—no other MS manufacturer in the world can match LECO’s deconvolution experience and success.
- Automated Peak Find
- Wide dynamic range (4 orders of magnitude)
- Maintenance-free ion source (no cleaning required)
- A robust quad-jet, dual stage Thermal Modulator
- Secondary oven for enhanced selectivity

New Variable Modulation

An essential principle of GCxGC research states that the use of the shortest possible modulation period maximizes chromatographic resolution in the first dimension. Historically this has meant chemists have had to employ a modulation period identified by the latest eluting analyte in the 2nd dimension. Now available with ChromaTOF, beginning with version 4.30, variable modulation allows chemists to incrementally increase the modulation period only as needed (older systems may require an additional hardware upgrade). This revolutionizing capability allows for the incredible separations of complex mixtures expected from the leaders in GCxGC technology.

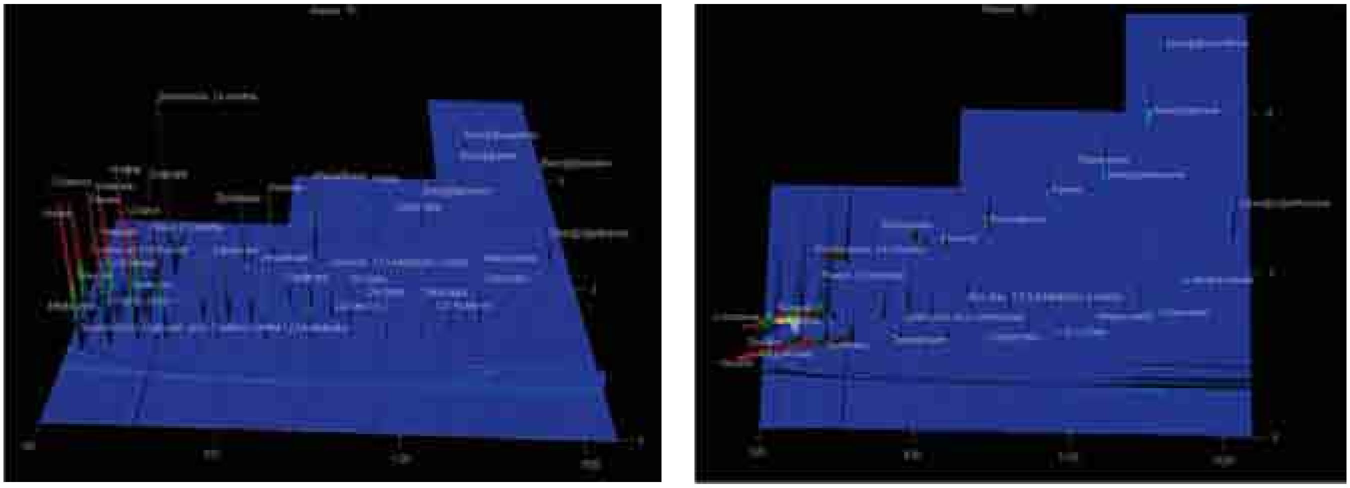


Figure 4. Contour plots of a single test mixture displaying the use of variable modulation from multiple views.

The Power of Time-of-Flight Mass Spectrometry

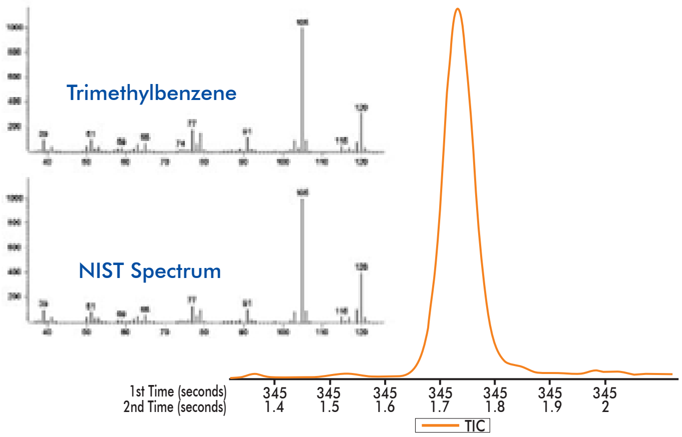


Figure 5. The 150 millisecond-wide trimethylbenzene GCxGC peak is easily defined and identified at a mass spectral acquisition of 200 spectra/second.

TOFMS—A Must-Have for GCxGC

As part of the cryo-focusing process in the GCxGC modulator, analyte bands that elute from the first column are significantly sharpened prior to being released into the second column. As a result, peaks ranging from 50 to 200 milliseconds wide are produced (Figure 5). These narrow peak widths require a detection system that is capable of collecting data at rates of 100 Hz or more in order to adequately characterize the shape of the chromatographic peak.

Only the Pegasus TOFMS, with continuous full-range mass spectral acquisition rates at up to 500 Hz, offers MS data with sufficient data density to address the requirements of any GCxGC separation. Spectral quality at these high acquisition rates is maintained in the Pegasus TOFMS as seen in Figure 5.

Qualitative Benefits of TOFMS

The power of the Pegasus TOFMS can also be seen in the qualitative benefits that GCxGC provide. By nature of the orthogonal separation system used in GCxGC, very structured chromatograms are produced showing distinct bands of analytes grouped by specific chemical characteristics (Figure 6). Using these chemical characteristics as a guide, more accurate individual analyte identification can be obtained from the Pegasus TOFMS.

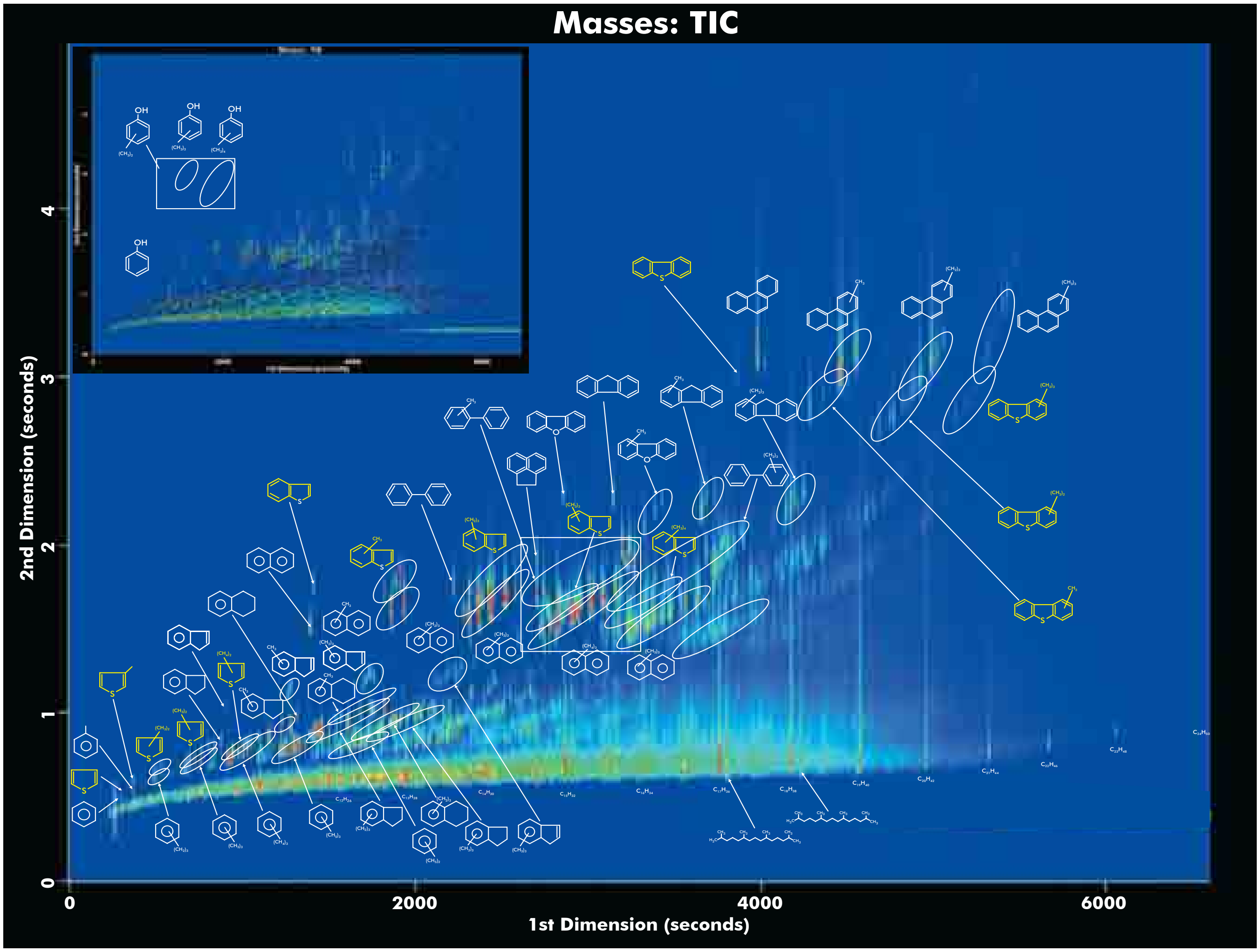
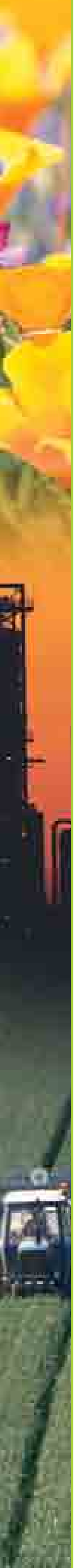


Figure 6. Complex sample characterization of diesel fuel is significantly simplified due to the structured GCxGC chromatogram that produces distinct bands for analyte chemical classes and the specific analyte identification provided by the TOFMS.



ChromaTOF® Software

ChromaTOF—Automated GCxGC Data Processing

In addition to offering fully integrated system control from a single computer and software package, LECO's exclusive ChromaTOF software provides fully automated processing of GCxGC-TOFMS data. From easily prepared Data Processing Methods, a complete sample assay can be obtained—including qualitative characterization of the sample and quantitative data for specific analytes of interest.

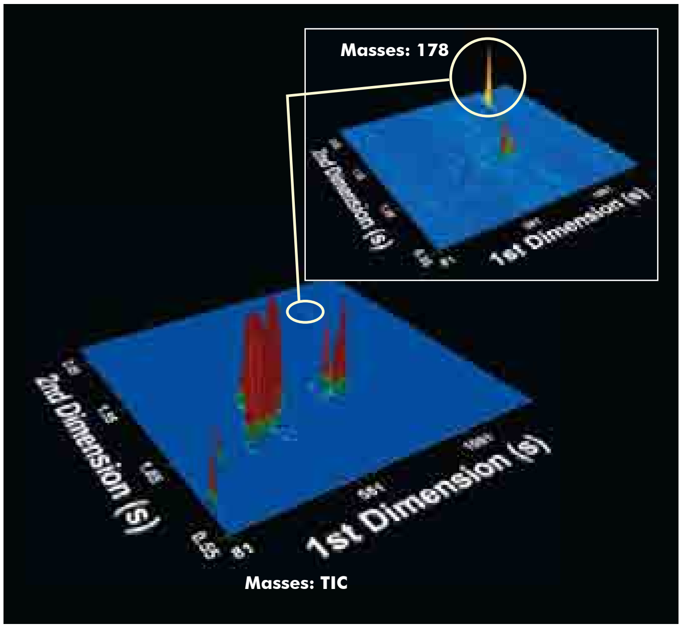


Figure 7. TIC surface plot for lime oil shows no peak in the gray highlighted region, however, viewing unique mass 178, the presence of a trace level compound is observed. In order to find the Citrapene peak, you must be able to look beneath the baseline—only ChromaTOF can deliver this to you automatically.

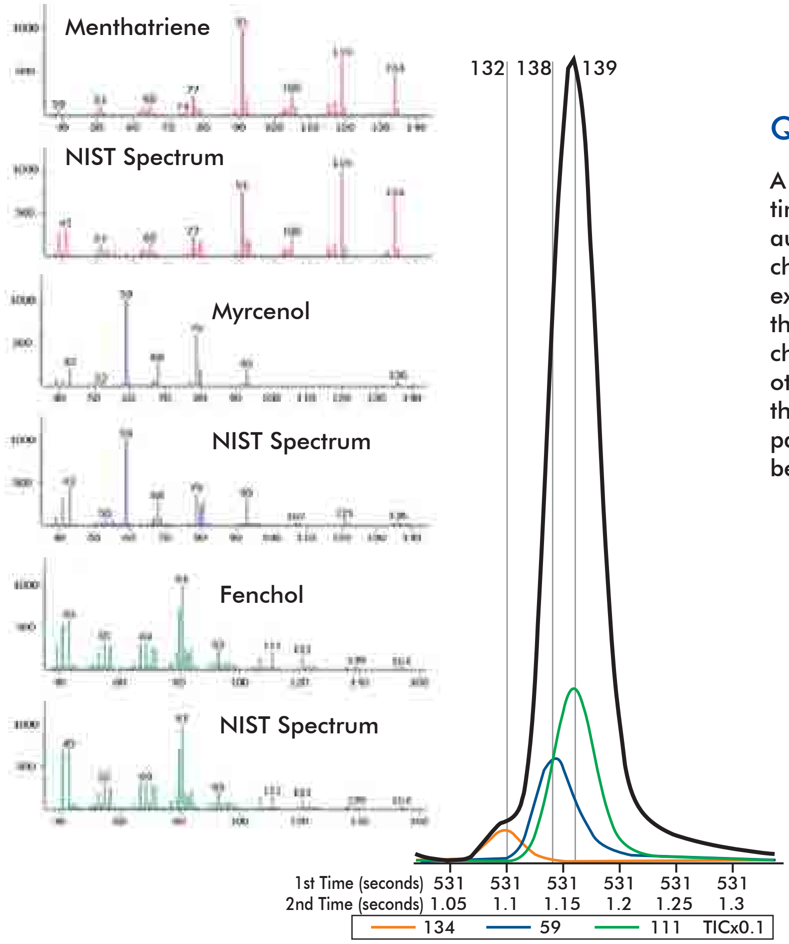


Figure 8. Automated Peak Find of three coeluting analytes in a 0.3 second portion of a second dimension chromatogram.

Qualitative Sample Characterization

A good software package reduces the amount of time it takes to decode your analyses. ChromaTOF automatically finds all peaks throughout the entire chromatogram and deconvolutes them in order to extract a clean mass spectra. These peaks include those that are hidden beneath the total ion chromatogram (TIC) and those that coelute with other compounds in the sample. Compare that to the manual requirements of other software packages and the advantage of ChromaTOF becomes undeniable.

- Automated Peak Finding
- True Signal Deconvolution to produce extracted mass spectra free of interfering signal
- Sample Compare feature automatically detects differences in unknown samples relative to a known reference sample

ChromaTOF® Software

Automated Quantitative GCxGC Analysis

LECO's ChromaTOF software offers fully automated quantitative analysis for use with GCxGC-TOFMS data.

- Unlimited number of Calibration Points
- Unlimited number of Internal Standards
- Multi-order calibration curves
- Individual point weighting factors
- Extended Range Calibrations
- Retention Index Probes
- Ion Ratios

A variety of electronic reporting and exporting functions are available for distribution of the quantified results.

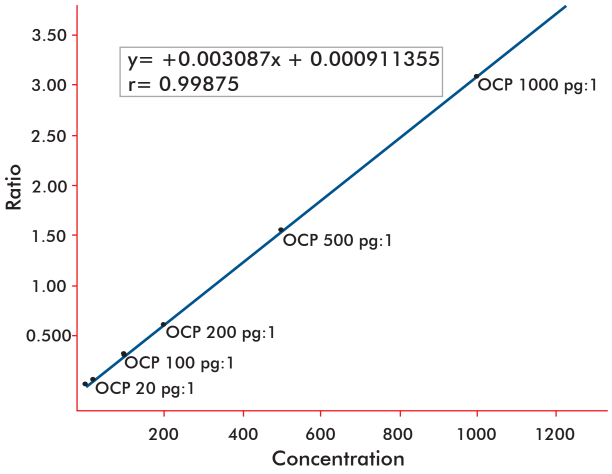


Figure 9. 4,4′-DDE calibration curve from 0.2 pg/μL to 1000 pg/μL.

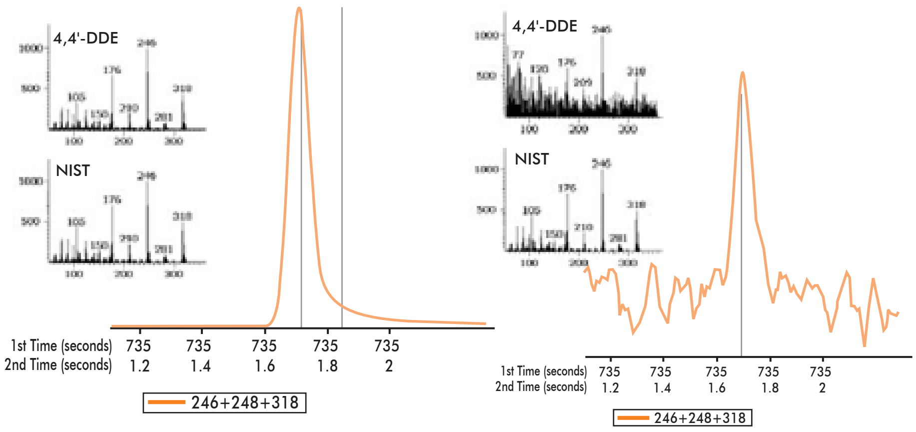


Figure 10. 1000 pg (left) and 0.2 pg (right) 4,4′-DDE Standards.

Analyte Calibration

Figure 9 depicts a GCxGC-TOFMS calibration curve for the organochlorine pesticide degradate 4,4′-DDE. The calibration ranges from 0.2 pg to 1000 pg on column to address the requirements for pesticide analysis in food matrices. The high and low level 246u extracted ion profiles are displayed, along with the mass spectrum for the 0.2 pg/μL and 1000 pg/μL standards in Figure 10.

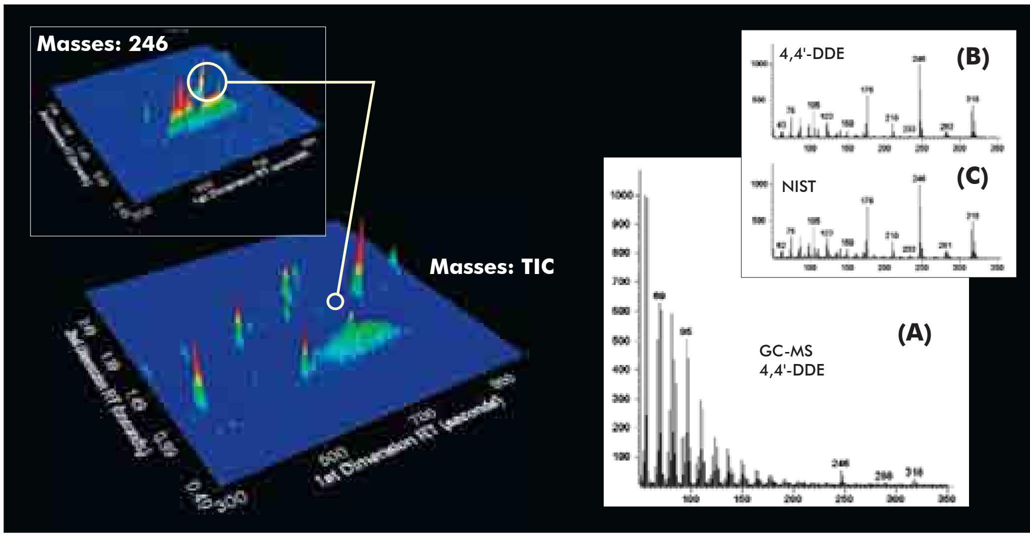


Figure 11. Analysis of 2 pg of 4,4′-DDE in a cucumber extract. No peak is observed in the surface plot TIC. Without GCxGC separation the 4,4′-DDE peak is difficult to identify among the cucumber matrix interferences (A). With GCxGC separation the 4,4′-DDE peak is easily identified (B) by a NIST library search (C).

Sample Quantification

Trace level quantification is frequently complicated by matrix interference. In Figure 11, the matrix components of a cucumber extract are clearly displayed in the surface plot. The added resolving power of GCxGC successfully moves the 4,4′-DDE peak away from the congested matrix region allowing for easier and more accurate identification and quantification.

ChromaTOF® Software

Leading the Way in Advanced GCxGC Data Processing

LECO's pioneering efforts in GCxGC data processing have resulted in the most comprehensive software package available. Even the most demanding analytical samples will be easily characterized, resulting in increased component identification and laboratory productivity.

- User-defined classifications—peak grouping based on proximity in chromatographic plane
- Bubble plots—peak intensity represented by circle radius
- Mass Spectral Scripts (visual basic programming) for enhanced spectral data-mining
- Custom-generated reports
- Sample Comparison feature
- Built-in spreadsheets for advanced data-mining
- Data exporting for secondary software analysis

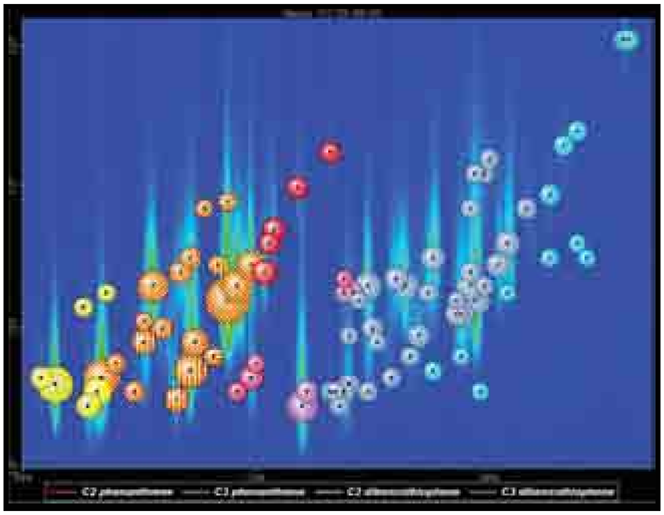


Figure 12. Bubble Plots are easily displayed to distinguish classifications as well as peak intensity.

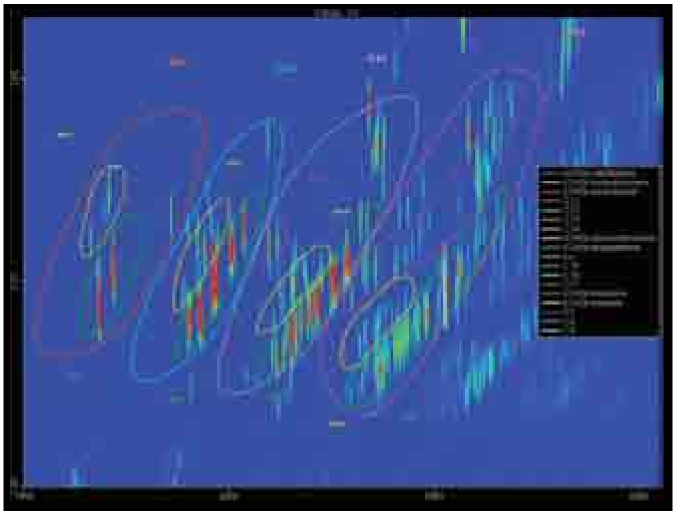


Figure 13. User-defined classifications identify regions containing sulfur and non-sulfur compounds found in this complex petroleum sample.

Statistical Compare

ChromaTOF's new Statistical Compare option answers the increasing demand for additional statistical analysis tools. The Statistical Compare feature allows a complete comparison of samples to be generated by aligning the analytes for each sample and providing statistical evaluations for each. Fisher Ratios can then be generated, and the grouping of different samples based upon the analytes found reported. Statistical compare can be extremely helpful for complex metabolomics samples.

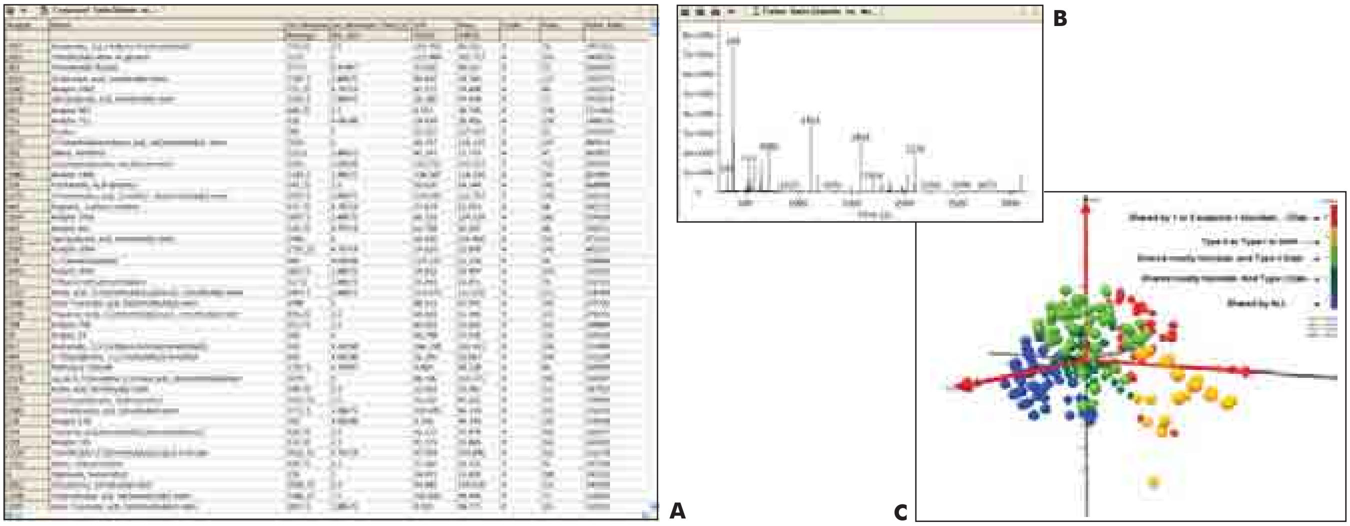


Figure 14. With Statistical Compare, the user can obtain a complete comparison for all analytes within a sample (A) as well as a Fisher Ratio for the sample run (B). The data can then be exported into an external software program such as Miner3D for PCA plotting (C).

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Delivering the Right Results

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