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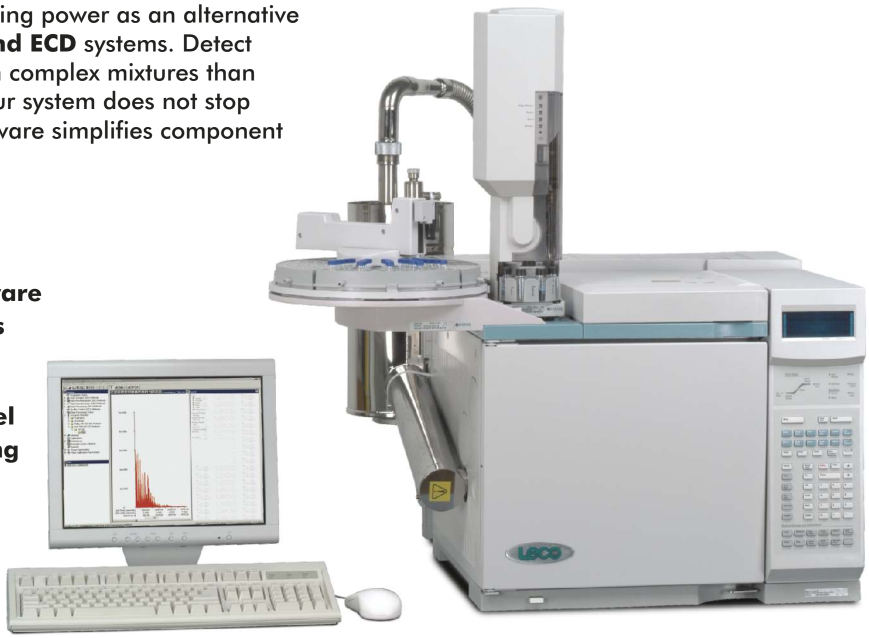
GCxGC FID/ECD

LECO GCxGC FID/ECD

Comprehensive two-dimensional gas chromatography (GCxGC) is a technique that multiplies the resolving power of a single gas chromatograph (GC) by that of another, resulting in an order of magnitude increase in the number of chemical compounds that can be separated in a given amount of time.

LECO now offers you this enhanced separating power as an alternative to conventional GC with our **GCxGC FID and ECD** systems. Detect hundreds-to-thousands more compounds in complex mixtures than previously seen. However, the strength of our system does not stop there. Easy-to-use ChromaTOF®-based software simplifies component identification like no other.

- **Ideal for quality control, production, and research**
- **Easy-to-use ChromaTOF-based software**
- **Classification software that simplifies component identification**
- **Ideal for**
  - **Petroleum samples, including diesel**
  - **Environmental compounds including pesticides, PCBs, and PBDEs**
  - **Flavors and fragrances**
  - **And more!**



The GCxGC Advantage

The demands of complex samples, such as detailed analysis of petroleum, overwhelm traditional GC systems. Using GCxGC FID analysis, the chromatogram may be spread across two dimensions, as shown with the sample of raw diesel fuel, shown in Figure 1.

GCxGC adds a second dimension of chromatographic resolution by using two distinct stationary phases (such as polar and non-polar) within a single analysis. The use of two separation mechanisms results in the separation of peaks across a plane, not just a line, and results in a dramatic increase in peak capacity.

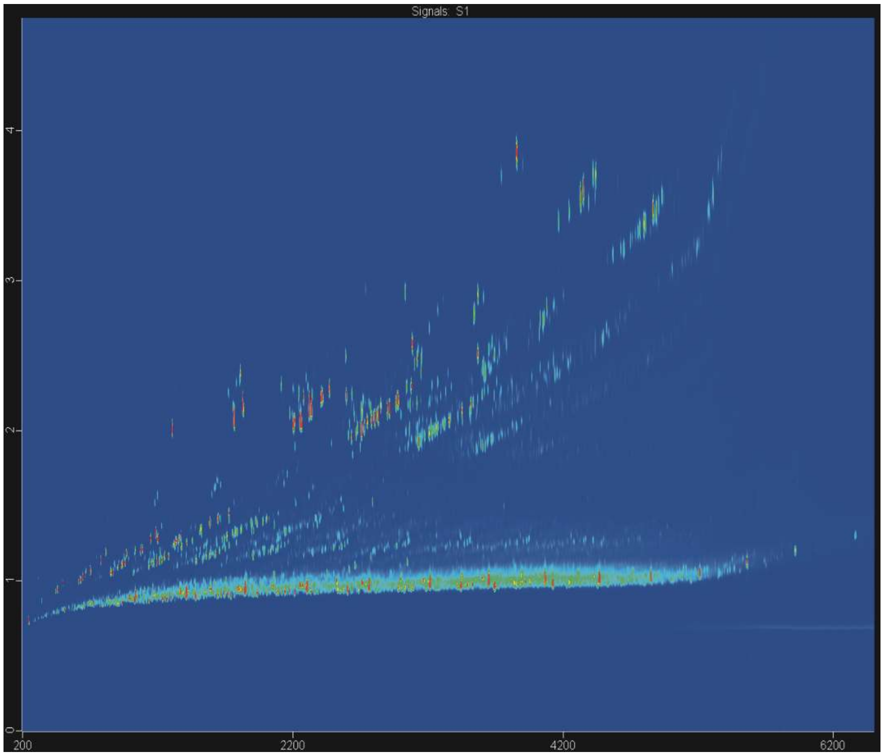


Figure 1: GCxGC FID contour plot showing the chromatogram of a diesel sample before sulfur-containing components were removed.

With GCxGC analysis, peaks are sufficiently resolved so that trace components, like organic sulfur compounds in diesel fuel, can be easily detected and quantitated. A surface plot showing the presence of these trace components in raw diesel fuel, and the absence in finished diesel fuel is shown in Figure 2.

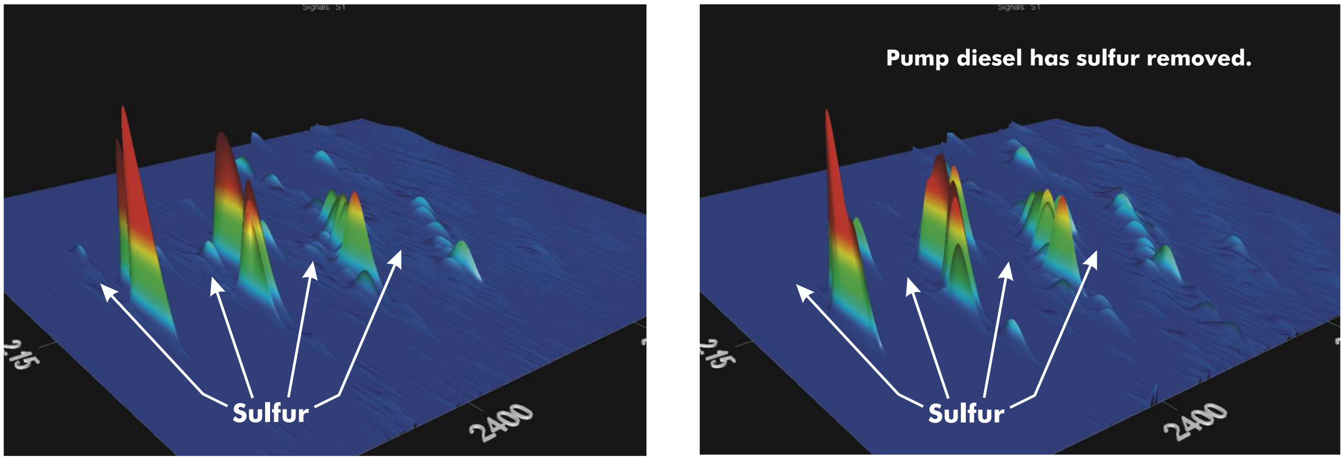


Figure 2: Detection of sulfur components in raw and finished diesel fuel.

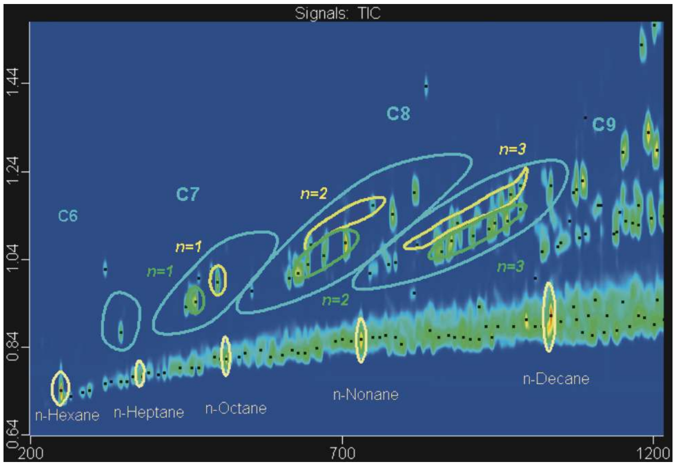


Figure 3: Petroleum sample, showing classification regions for n-paraffins, benzenes, and thiophenes. The blue circles indicate regions for substituted benzenes and are marked by carbon number. Yellow and green circles show regions containing compounds that are thiophenes and alkyl benzenes, respectively. Total response for peaks in any designated region may be reported.

Analysis by Classification

Some samples, such as petroleum, may be analyzed for sum totals of compound classes. GCxGC tends to organize compounds according to their chemical structure. With the Classification capability of the ChromaTOF-based software, chromatographic regions containing compounds may be easily marked for automatic selection during data processing, as shown in Figure 3. The total response for all peaks within each region may be reported. So, for a detailed petroleum analysis, it is possible to obtain a total response for each region of n-paraffins, isoparaffins, cyclic compounds, substituted benzenes, and other classes, with the data also characterized by carbon number.

Other Detectors

The LECO GCxGC system works with the Agilent micro ECD to allow for applications such as the identification and quantitation of pesticides in difficult sample matrices. Using the GCxGC ECD system, pesticides that would provide chromatographic difficulties because of coelution with other pesticides or with interferences in the matrix, can be separated (Figure 4). With the superior separation provided by GCxGC ECD, linear calibrations for analytes may be obtained over several orders of magnitude, even where coeluting interferences give rise to detection and linearity problems in one-dimensional chromatographic systems. A GCxGC ECD calibration curve for DDT is shown in Figure 5. The high sensitivity of the GCxGC ECD system allows for a split injection. With the split injection, less material is introduced to the column, extending column life and reducing inlet maintenance. In this figure, the samples are being run with a 20:1 split ratio. At this ratio, only 10 fg of DDT are introduced to the column for the low calibration point, still with excellent response for quantitation.

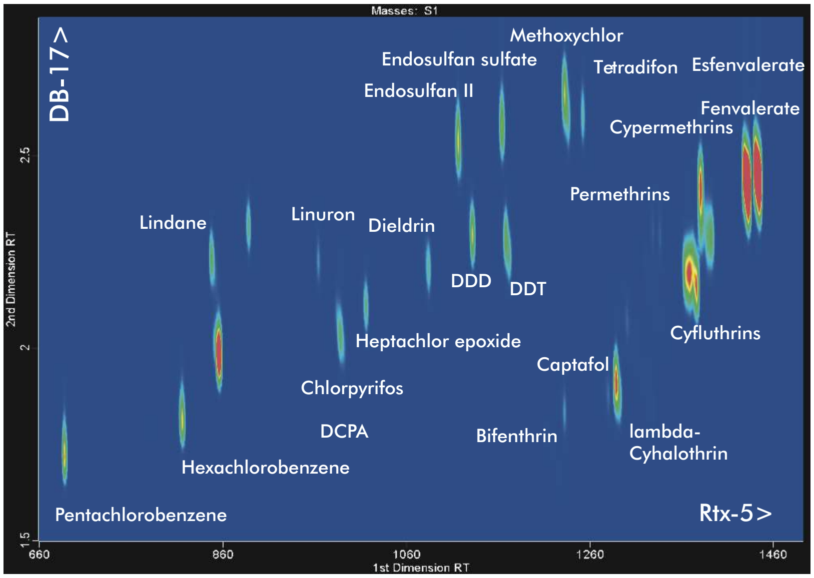


Figure 4: GCxGC ECD contour plot of halogenated pesticides.

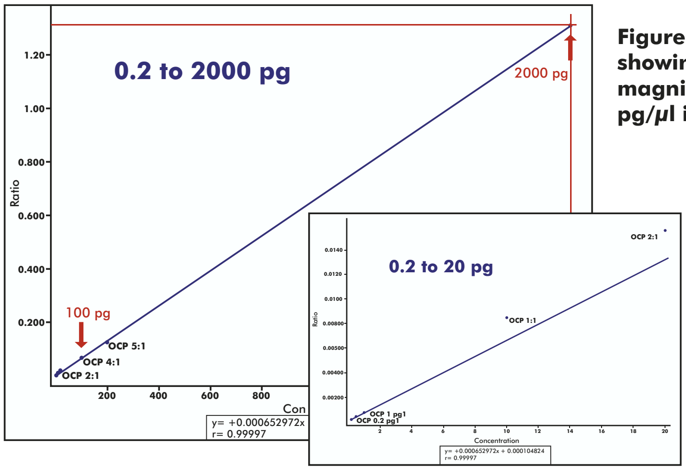


Figure 5: GCxGC ECD calibration curve for 4,4'-DDT, showing highly linear response over four orders of magnitude. The samples range from 0.2 pg/μL to 2000 pg/μl injected with 20:1 split ratio.

Unraveling Complexity

Real-life environmental samples have many coelutions between analytes, and between analytes and compounds in the matrix. These coelutions may be congeners, which are often extremely difficult to separate, particularly in an analytical method that is to find and quantitate a large number of compounds. The increased peak capacity of the GCxGC FID and GCxGC ECD systems provide separation between PCBs, PBDEs, and other environmental contaminants, with separation between congeners, all in a single analysis (Figure 6). This eliminates the need for the difficult separation of these compounds during sample preparation.

High-Powered QC

Critical ingredients of flavor and fragrance mixtures are often some of the more dilute compounds in the mixture. These compounds are often masked by the presence of other compounds when analyzed with one-dimensional chromatography. Even the use of high-powered detectors, such as mass spectral detectors may not be adequate for detecting and quantifying these compounds. Yet, the high peak capacity of GCxGC provides the ability to resolve and quantify these compounds. In the rosemary oil sample shown in Figure 7, over 400 chromatographic peaks can be quantified. With the ability to distinctly and reproducibly measure components at part-per-million levels and below in these complex mixtures, the GCxGC FID and GCxGC ECD systems are ideal for QC applications such as flavor analysis.

The GCxGC Instrument

GCxGC occurs by the repeated focusing and re-injection of effluent from one chromatographic column into a second column. The cycle of refocusing and re-injection is matched to the time required for compounds to elute from the second GC column, resulting in separation of compounds across a plane, rather than just along a line. With the LECO GCxGC system, this is accomplished with a dual-stage, quad-jet thermal modulator positioned between the two columns (Figure 8). The modulator and an independently controlled oven for the second chromatographic column are installed inside the oven of an Agilent 6890N GC.

The ChromaTOF-based software controls instrumental and data acquisition functions, as well as providing for automated data processing, all in a fully integrated package.

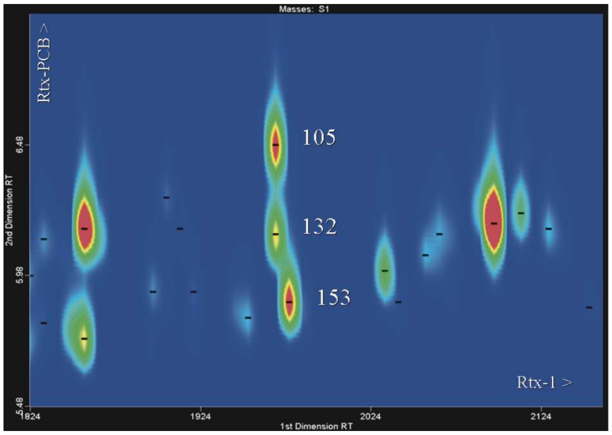


Figure 6: GCxGC ECD separation of PCB congeners in Aroclor 1254.

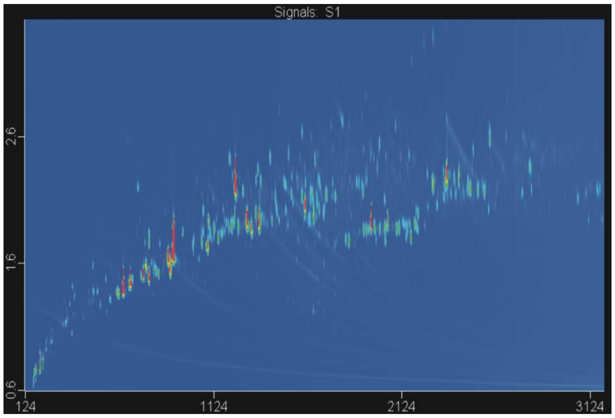


Figure 7: GCxGC FID chromatogram of Rosemary Oil showing over 400 quantifiable peaks.

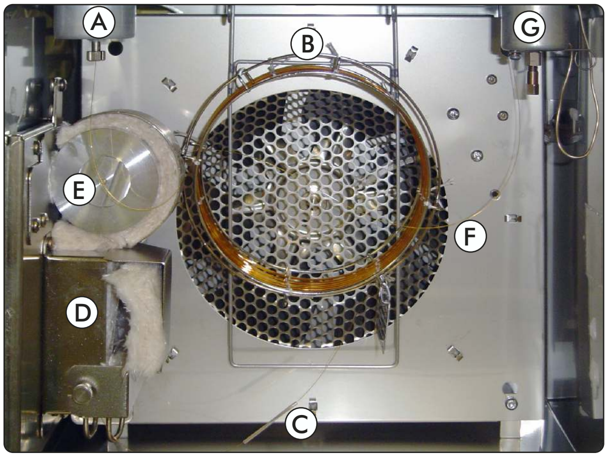


Figure 8: Inside of GC oven, showing GCxGC components and connections: (A) GC inlet, (B) Primary GC column, (C) Pressfit connection between primary and secondary GC columns, (D) Quad-jet modulator, (E) Secondary oven (containing the secondary column), (F) Transfer line (typically uncoated capillary) connecting secondary column in secondary oven to detector, (G) Detector.

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Form No. 209-157 R2.58-REV1