

Comprehensive Two-Dimensional High-Speed Gas Chromatography with Chemometric Analysis

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High-speed comprehensive two-dimensional gas chromatography (GC \times GC) is performed, in which a polar second column performs separations every half second on portions of the effluent from a nonpolar first column. Chemometric techniques that are traditionally used on chromatographic separations with multichannel detection are applied to two-dimensional chromatographic data, for the purpose of quantifying incompletely resolved peaks. Generalized rank annihilation method (GRAM) is evaluated in the quantification of varying amounts of selected overlapped analytes in a GC \times GC analysis of modified white gasoline. GRAM requires a sample and standard data set for quantification, and the high retention time precision arising from use of shortened GC columns aids in the analysis. Results from GRAM analysis of GC \times GC data are compared with a reference GC method. The test analytes ethylbenzene and *m*-xylene, existing in various proportions in white gasoline samples, were successfully deconvoluted despite having resolutions of 0.46 and 0.20 on the first and second dimensions of separation, respectively. Like other second-order techniques, GRAM was able to reliably quantify *m*-xylene despite the presence in the analytical sample of an overlapping compound not present in the calibration standard. Because GRAM can be successfully applied to GC \times GC data, full resolution of all the analytes of interest is not necessary. As a result, GC \times GC run times can be dramatically shortened, which has significant implications for analyses in which short cycle times are critical, such as in process analysis.

Gas chromatographic (GC) analysis of multicomponent samples can be a time-consuming process. To reduce analysis times, many separations are done using multicolumn GC. In traditional multicolumn GC (GC-GC), the first column performs a partial separation of a sample, and a fraction, or heartcut, of the eluent is injected into the second column for final separation. The first column effectively performs sample preparation, reducing the number of compounds injected into the second column. The second column must have a different chemical selectivity than the first column in order to optimize the GC-GC resolving power. The difference in the chemical selectivity between the two columns allows compounds that are poorly separated in the first dimension to be more fully resolved in the second dimension. Although this technique is proven to have numerous practical

value,¹ it can be a time-consuming process. Heartcuts from the first column range from seconds to minutes in length, and the separation that the first column has performed on the compounds within that heartcut is generally undone during the injection onto the second column. The lengthy runtimes of the second column limit the number of heartcuts that can be taken from the first column.

Traditional GC-GC is not a true "hyphenated" technique. Hyphenated chromatographic techniques, such as liquid chromatography with diode array absorbance detection, offer the advantage of being able to identify eluting compounds through inspection of the spectral information. For hyphenated techniques, a vector of data is obtained at short time intervals during a separation, producing a two-dimensional data array for each run. The ideal GC-GC instrument is one that can function as a hyphenated instrument, for it would preserve all the chromatographic information from both dimensions of separation. This ideal system would perform a second dimension separation of an infinite number of infinitely thin heartcuts from the first column. Under these circumstances, the separating power of the ideal system is proportional to the product of the separating powers of the individual columns.¹² There is a practical limit to how frequently a sample can be cut to the second column, so the actual separating power is less than that theoretically calculated. This mode of operation is termed comprehensive GC-GC, or GC \times GC,¹³ with the multiplicative term emphasizing the enhanced separation power of this technique. Because GC \times GC can provide much larger peak capacities, difficult separation problems are more easily handled with GC \times GC. In addition, if a separation problem requires a given peak capacity, GC \times GC is capable of generating it much more rapidly than an existing multiple-column GC using a few heartcuts. This article investigates how chemometric techniques can be applied to GC \times GC to get the most information from the data and reduce run times, thus broadening the scope of GC \times GC.

In fast GC, peak width, and thus separation efficiency, is frequently limited by the performance of the injector. To reduce separation times, the injected sample pulse width must be as short in time as possible.² GC \times GC is most useful when the second

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