An Executive Summary

Ultra-Fast GC: Useful Tool or Just Another Gimmick?

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Ultra-fast gas chromatography (UFGC) offers analysis times 5–20 times faster than conventional gas chromatography (GC), comparable sample capacity and resolution. These factors along with greatly reduced energy consumption make UFGC a valuable approach to explore in a number of application areas. Phillip James, Managing Director at Ellutia Ltd., describes the principles and practical uses of ultra-fast GC and the key considerations in method development using this technique.

What is Ultra-Fast GC?

Definitions of the basic forms of GC are instructive in understanding the dynamics of ultra-fast GC.

Conventional GC uses 15–60 m columns with internal diameters up to 0.53 µm or, sometimes, packed columns. Heating rates typically are from 1 °C/min to 40 °C/min, and analysis times run from 15 min to 90 min or more.

Fast GC uses columns with very small diameters, typically 0.1 mm, and short column lengths, such as 10 m. Fast GC yields excellent resolution and improved speeds over conventional GC at the cost of sample capacity. Run times average just a few minutes.

Ultra-fast GC uses much shorter columns with larger internal diameters, which helps overcome the capacity problems experienced with 0.1-mm columns and allows for faster ramp rates. The column is usually heated in a column compartment without the use of a column oven with the objective of heating as little mass as possible so that ramp rate increases and cool-down can be accomplished quickly. Cycle times for UFGC are 5 to 20 times faster than for conventional GC. Ramp rates of 1000 °C/min can be achieved, but for practical reasons, ramp rates in the range of 60°C/min to 200 °C/min are usually used. Run times are less than a minute to a few minutes.

The column can be heated to much higher temperatures with the columns upper temperature being the limiting factor, but the temperature inside the column compartment is typically only about 90 °C, which allows for cool-down in 30 s to 90 s. Rapid cool-down in UFGC requires significantly less energy than in conventional GC, which can be a factor when large numbers of samples are being analyzed.

Types of Ultra-Fast GC Systems

The bundled heater system for UFGC consists of heating wire and a sensor bundled within a fused silica column. The heating wire resistively heats the column by close approximation to the column itself. This system has a fair amount of mass to be heated.

The EZ Flash system uses a fused-silica capillary column placed inside a metal tube that is rapidly heated to heat the column. The heating mechanism is parallel to the column and is heated from one end to the other.

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Metal columns, unlike fused silica columns, can be heated directly, which involves heating the minimum possible mass. Ellutia has been exploring this approach for several years.

Applications of Ultra-Fast GC

UFGC is particularly useful for applications requiring rapid and repeated analysis. A few examples are:

- *Screening analyses.* UFGC offers significant advantages for the screening analysis of environmental, petrochemical, and pesticide samples because results can be turned around rapidly, even same day, and analyses can be repeated quickly for confirmation of results.
- *Reaction monitoring.* UFGC is used in motor racing to analyze sump oil for fuel breakthrough to help determine engine wear. The technique is also useful in the analysis of waste streams in pharmaceutical manufacturing and in testing permeation breakthrough of chemicals in warfare suits and other types of protective clothing.
- *Clean-up monitoring.* Industries such as essential oils manufacturing that require clean processing equipment use UFGC for fast determination and repeated analyses of possible residue materials.

Examples of Ultra-Fast GC Analysis

A very fast analysis of the Florida TRPH C8–C40 standard is shown in **Figure 1**. An analysis that typically requires about 2.5 minutes is accomplished in 28 seconds with baseline resolution for all peaks.

The fuel oil separations shown in **Figure 2** compare a conventional GC analysis at 35-min with a very similar analysis by UFGC in just 150 s.

Figure 1: Ultra-fast GC separation of Florida TRPH C8–C40 standard.

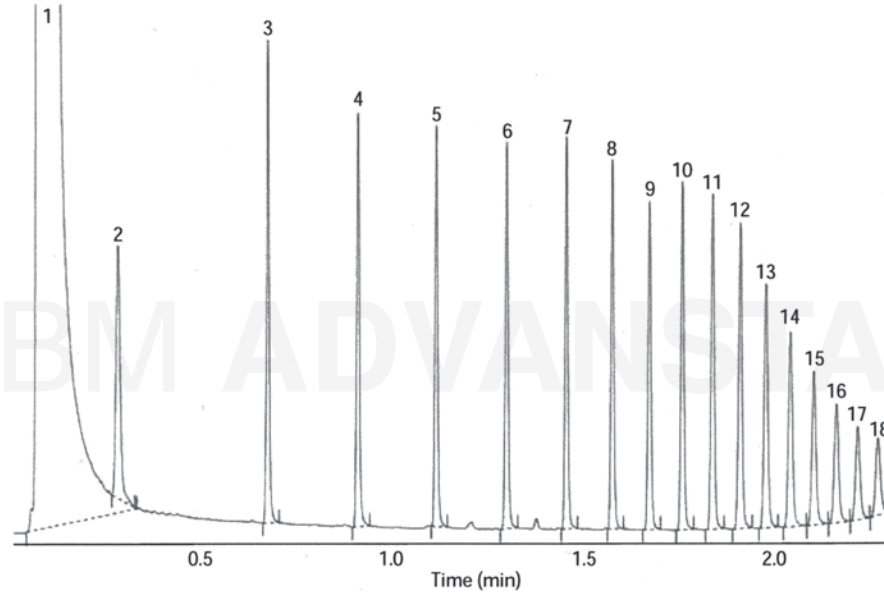
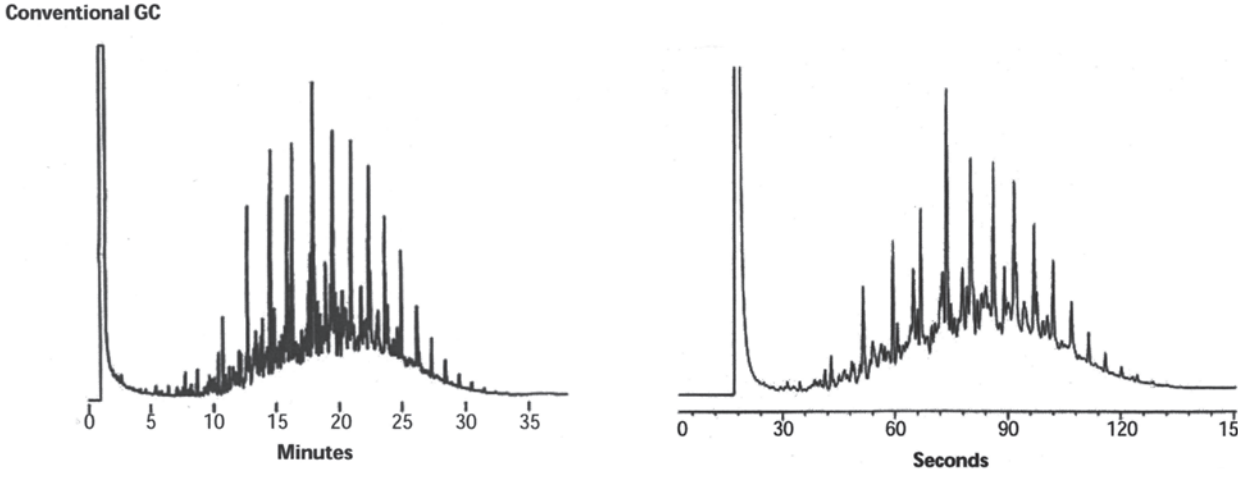
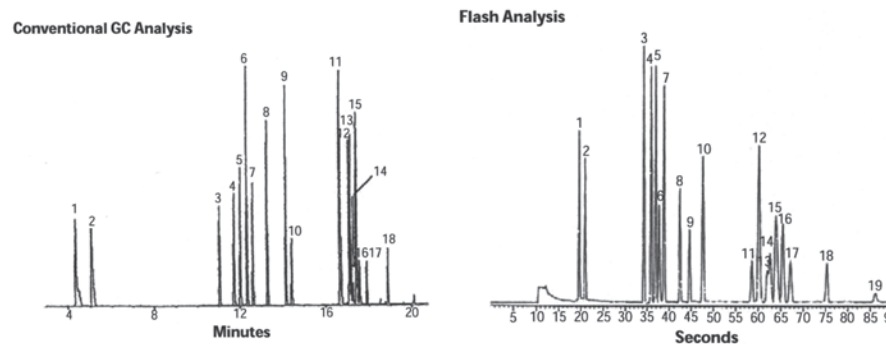


Figure 2: Fuel oil separation by conventional GC and ultra-fast GC.



In **Figure 3**, a sample of drugs of abuse is separated in 20 min using conventional GC and in 90 s using ultra-fast GC with resolution of all peaks

Figure 3: Drugs of abuse separation by conventional GC and ultra-fast GC.



Advantages of Ultra-Fast GC

The usual analytical requirements for chromatography are 1) the shortest possible run times, 2) large sample capacity, and 3) baseline resolution of all peaks of interest. Invariably, the analyst is faced with trade-offs among these factors. For example, a 10-m, 0.1-mm i.d. column (fast GC) (**Figure 4a**) can yield a fast analysis with good resolution, but sample capacity will be terrible. A 60-m, 0.25-µm i.d. column (conventional GC) (**Figure 4b**) has plenty of sample capacity and can produce good resolution, but separation times will be long. A 5-m, 0.25-µm i.d. column run with UFGC, however, can achieve separations with comparable runs times and sample capacities as a similar column using conventional GC (**Figure 4c** and **4d**) but with significant resolution gains as a result of rapid temperature ramping. Essentially, UFGC extends the separation triangle toward resolution.

Figure 4: Time, capacity, and resolution trade-offs for conventional, fast, and ultra-fast GC.

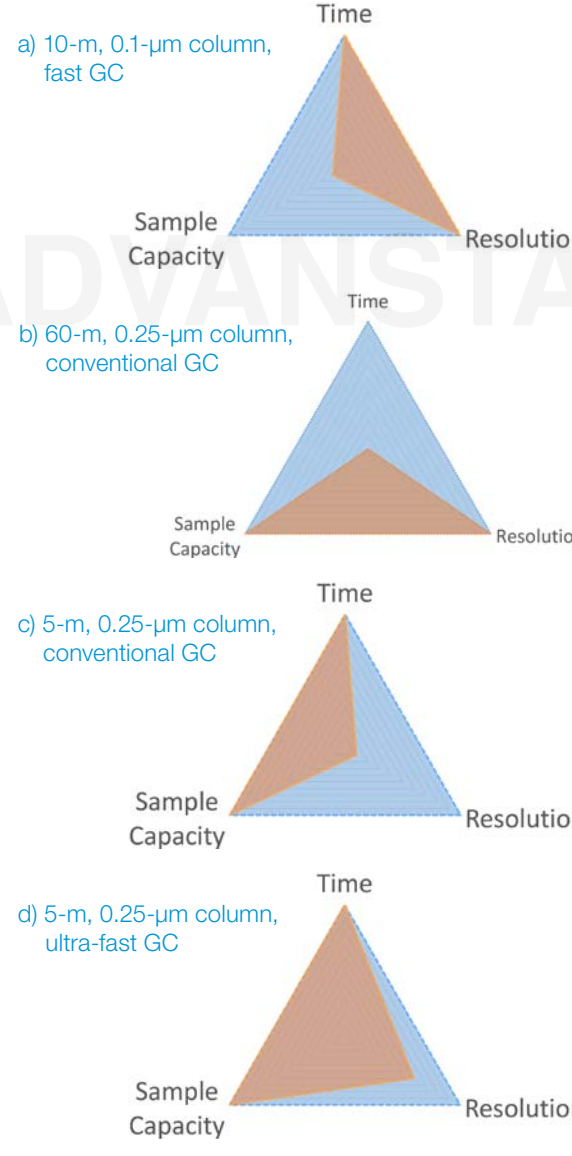


Figure 5 illustrates the peak sharpening that is possible using the fast ramp rates achievable with UFGC. The bottom (blue) chromatogram is run on a fast GC column at a ramp rate of 40 °C/min. Peaks are well separated, but broad, with a run time of about 4 min. In the middle (green) chromatogram, the run is started at 40 °C/min with a very quick ramp up to 120 °C/min across three peaks, which has the effect of sharpening them. The top (red) separation was ramped up to 150 °C/min for further peak sharpening with a much shorter run time while maintaining baseline separation.

Method Development for Ultra-Fast GC

Method development for UFGC can seem counterintuitive, so it's important to understand the basic principles. An application run on a 30-m column can usually be run with equivalent results via UFGC. If a method requires a 60-m column or extra thick films to achieve the required resolution, ultra-fast GC may not achieve the needed resolution, but, in that case, a screening method can be developed.

Start with a relatively slow ramp rate such as 60 °C/min using hydrogen as the carrier gas at either a constant pressure of 8 psi or a linear velocity of 50 cm/s and a 7-m thin-film 0.18- or 0.25-µm i.d. column. The column can be shortened, if needed, if all peaks do not elute in the required time.

The temperature ramp should be started about 10 °C above ambient. Adjust the temperature range to elute all components in the sample, which can usually be accomplished in 2–3 runs.

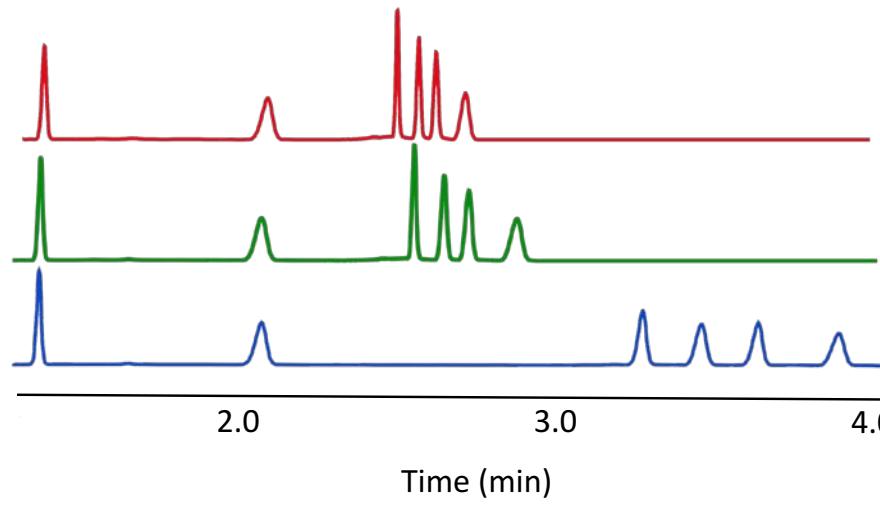
Strive for the top temperature to be as low as possible. Mark peaks of interest that will require good resolution.

Next, for the first single-ramp chromatogram, adjust the carrier velocity until the best separation of the peaks of interest is achieved (up to 120 cm/s). Use a constant pressure starting at 8 psi and then adjust up and down in small increments on each run to allow for achieving the best resolution in different parts of the chromatogram. This process typically requires about 10 runs.

Once the best pressure program is achieved, the temperature ramping program can be adjusted in areas of the chromatogram to speed up the separation as a whole. Look for areas of the chromatogram with gaps or with no peaks of interest — for example, if the main peaks of interest don't start until 150 °C, ramp up to 400 °C up to that point and then back off to lower rates.

Eventually, resolution will begin to deteriorate. When that point is reached, back the ramp rate down slightly. Finding the best ramp rate typically takes about 20 runs. Most successful UFGC methods are about 4 min with a 90-s cool-down. The entire optimization process might take about 35 runs of 5 min each.

Figure 5: Peak sharpening using the fast ramp rates achievable with ultra-fast GC.



The key points for UFGC method development are:

- Always elute on a ramp to sharpen peaks of interest.
- Use the lowest top temperature possible.
- Use multistage ramps, start slow and use faster ramps on compounds with higher boiling points (they have the greatest band broadening)
- Use thin films for better performance.
- Use 4- to 6-m columns for the best performance.
- Use rapid ramps to elute compounds that are not of interest.
- Clean the column by doing rapid intermediate runs.

Common problems in UFGC are:

- Baseline resolution can be difficult to achieve for some samples, such as PAH.
- Sometimes, sufficient ramp is difficult to achieve to get the peaks off the column. In this case, higher-temperature columns or shorter columns may help (7–4 m).
- Cold spots can result in peak broadening
- Detector speeds of a minimum of 20 Hz are required for UFGC; mass spec detectors are currently difficult to use.
- Retention time drift can be an issue; for example, a 1-s drift in a 120-s chromatogram is significant. Add stabilization time at the start of the separation to make the entire chromatogram more stable.
- Contamination of the front end of fixed-length columns is possible. If the front end is dead, the column must be replaced. The use wool in the liner, pre-heated retention gaps, guard columns or columns that can be cut down help address this.

Future Advances in Ultra-Fast GC

The next three to five years will see columns optimized for UFGC. Faster multichannel detectors are being developed that will improve detection capabilities, including time-of-flight mass spec, infrared absorption spectroscopy, atomic emission detection, and far-UV absorption spectroscopy. Advances in detection and fully integrated systems on the horizon will help move UFGC into common use.

Ellutia currently has the 500 Series GC available, which provides a convenient and cost-effective transition into UFGC. Conventional, fast, and UFGC are all possible using this instrument. A range of column configurations can be used with the system, and the conventional oven will automatically calibrate ultra-fast columns.