

Cool Technology

The new Clarus 600 gas chromatograph (GC) was developed to cut down analysis times and make the separation of highly volatile compounds easier. *The Column* spoke to Andrew Tipler, senior scientist at the chromatography business unit of PerkinElmer Instruments to find out more.

Why was the Clarus 600 GC developed?

The new platform aims to let chromatographers speed up their analysis without changing any of the existing chromatographic conditions. It can also separate highly volatile compounds at low temperatures (within 5 °C of ambient temperature, for example) previously not practical or even possible. To cool at such temperatures takes much longer to get the remaining residual heat out of the oven. In some instances, this cooling can take 20 to 30 minutes or even longer — this clearly has a detrimental effect on analytical throughput and may even make the analysis impractical. The newly designed oven is capable of cooling from temperatures of 450 °C to 30 °C in about 4 minutes and to 28 °C in about 9 minutes with an ambient temperature of 23 °C.

What are the advantages over traditional systems?

There are two main advantages to this new platform: speed and the ability to separate compounds at near ambient temperatures, which is useful for analysing highly volatile compounds. In terms of speed the new platform can match the heating rate of any other conventional air-bath GC on the market but, more importantly it can cool much faster. The fastest competitive GCs will take about 4 minutes to cool from 350 °C to 50 °C, the Clarus 600 will do the same in about 1.2 minutes. In both instances, a further 1 minute equilibration is normally applied to stabilize the column temperature before proceeding with the next analysis. The autosampler operation has also been enhanced to initiate loading of the syringe with sample before the next analysis.

Regarding operating at near ambient temperature, there are many compounds that elute from columns at near ambient temperature but with current commercial technology such a



separation is not practical. Traditional air bath ovens require unacceptable times to reach equilibrium at near ambient temperatures and in many instances may not even reach them. The new oven, because of its ability to rapidly and efficiently exchange heat, can now easily reach and maintain these temperatures so can separate compounds previously not practical, such as volatile organic carbon compounds (VOCs) in environmental analysis and gas stream monitoring.

What is new about this design?

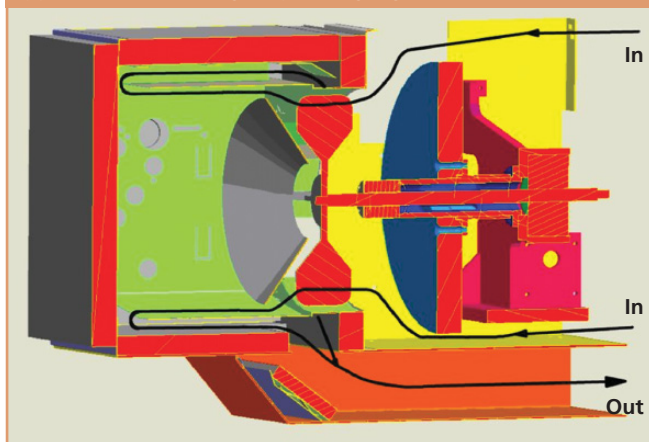
Similar to most modern GCs, the Clarus 600 uses an air-bath oven where a large fan is used to mix the air inside the oven during heating and to assist with the exchange of the hot air with ambient air during cooling. In this design (patents pending) a novel dual oven approach has been adopted. The inner chamber of the oven holds the GC column and injector and detector ports. The inner chamber is separated from the outer wall by a thin second wall that serves to reduce the heat loss from the inner chamber during heating and so improves the heating rate and minimizes temperature non-uniformity (gradients). During cooling, ambient air enters the inner chamber via a door, which is mounted behind and concentric to the fan. The hot air exists through the outer wall and out to a vent.

Thus, the exhaust air and the incoming ambient air have little opportunity to mix and so the exchange of air is rapid, which greatly assists the cooling process and near ambient temperature performance (Figure 1).

Another innovation is that the oven fan is driven by a programmable DC motor to enable dynamic control of its rotational speed. The fan speed is ramped up to increase the flow of ambient air through the oven during cooling.

A further design requirement is that the autosampler control

Figure 1: CAD drawing illustrating dual-walled oven design approach with cooling air path highlighted.



can now anticipate the time at which the GC has been cooled and is ready to accept the next sample injection and initiates the autosampler operation to load sample into the syringe before the GC actually becomes ready. This can save minutes for each analysis.

What problems did you have to overcome in its developments and how did you overcome them?

We wanted to give the users the benefits of a faster oven but retain the ability to use standard columns, injectors, detectors and pneumatics so that the GC could use existing methods without modification. This ability to combine both needs into a single instrument required careful review and evaluation of the various critical GC components to ensure that they performed under traditional and high-speed conditions.

A significant period of time was spent in optimizing the air flow within the oven during temperature programming. Chromatographic peaks are very sensitive to temperature gradients which, in extreme instances, give rise to fragmented peak shapes (known as the "Christmas tree effect"). By applying a combination of mathematical modelling, flow path studies and simple trial and error regimes, an optimum design was established to give artefact-free peak shapes.

During the development, we discovered a phenomenon that can actually be perceived as counter intuitive — columns can in fact be cooled too fast. With very fast cooling, as seen with this new oven, it's possible for the carrier gas inside the GC column to contract during cooling at a rate faster than the flow-rate of new carrier gas entering from the injector.

In such instances, gases would be drawn from the detector back into the end of the still-hot column with a consequential detrimental effect on the column integrity. Mathematical equations were developed to model and understand this behaviour. A technique called 'soft-cooling' was developed to limit the cooling rate so that ingress of detector gases back into the column was eliminated.

What disciplines do you see it being particularly useful for?

The new platform will be useful for any routine analysis where temperature programmed chromatography is used, such as petrochemicals, food and fragrances, environmental analyses and materials testing.

Because of the lower temperature oven operation, this new oven will be particularly suited to any application requiring a low temperature separation without resorting to liquid cryogen operation. Typical examples would be light hydrocarbons, permanent gases and halocarbons. The new oven would also be well-suited for air, water and soil analyses where a mass spectrometer is used to detect and identify highly volatile pollutants.

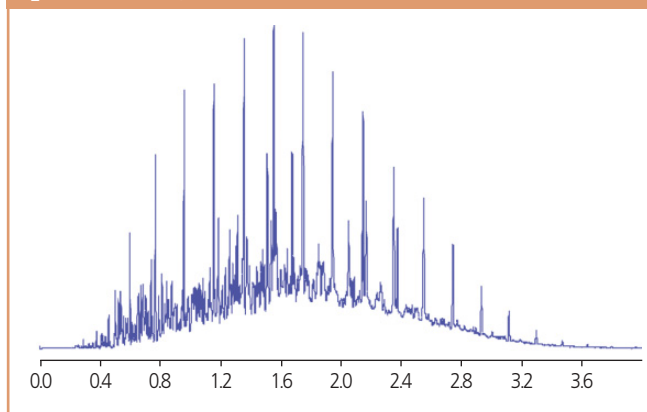
Can you illustrate this with a practical example?

An interesting example is an analysis we performed on diesel oil (Figure 2). This type of analysis is performed in the petrochemical industry for simulated distillation studies (ASTM method D-2887) or for environmental studies (soil analysis for leaking underground storage tanks). Traditionally, such an analysis may take 20–60 minutes for the chromatography with a further 6–10 minutes for the oven cooldown, temperature equilibration and autosampler preparation.

For this example, using the fast programming rates of the new platform with a short, narrow-bore column the chromatographic separation occurs in 4 minutes with a further 3.4 minutes for cooldown and autosampler operation, giving a total analytical cycle time of only 7.4 minutes — enabling 65 of these complex chromatograms to be run in a single 8 hour shift.

While this example demonstrates the benefits of both fast heating and cooling, more conventional applications using normal heating rates will also save time with this system.

Figure 2: Chromatogram of diesel oil. Injection: 0.3 μ L by autosampler in fast mode. Column: 5 m \times 0.050 mm \times 0.050 μ m PE-1. Oven: 50 $^{\circ}$ C then at 120 $^{\circ}$ C/min to 70 $^{\circ}$ C, then 90 $^{\circ}$ C/min to 115 $^{\circ}$ C, then 70 $^{\circ}$ C/min to 175 $^{\circ}$ C, then 50 $^{\circ}$ C/min to 300 $^{\circ}$ C, then 30 $^{\circ}$ C/min to 320 $^{\circ}$ C. Carrier gas: H₂ at 40 cm/s. Split: 400 mL/min (~4500:1)



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