

# Fast Micro-GC Capabilities Based on a Microintegration Technology Platform

Chromatography dates back to the early nineteenth century, when paper and cloth were used to spot-test dye mixtures and plant extracts. However, it was not until the mid-twentieth century that the first theoretical article on chromatography was published.<sup>1</sup> Chromatography is determined by many different parameters including, but not limited to, column efficiency or theoretical plate number, retention time, temperature and temperature ramp, volumes and flow rates, dead volumes, injection profiles and widths, and detector specifications. This article focuses on the impact of the instrument hardware on the chromatography.

The C2V-200 miniaturized fast micro-GC (**Concept to Volume B.V.**, Enschede, The Netherlands) is described here. The authors demonstrate why the right miniaturized design leads to a system that is smaller and faster and that demonstrates higher separation quality.

## Hardware theory

Chromatography instruments can be classified by the speed of analysis and thus by the mean width at half height of the chromatography peaks. In order to determine the limitations of the instrument, this article assumes almost perfect separation characteristics by the column, with the exception that the column length will impact the peak broadening. In the following, critical functional components are addressed.

The separation quality of a chromatography system is given by the number of theoretical plates, defined as:

$$N = \left( \frac{t_R}{\sigma} \right)^2 \quad (1)$$

where  $t_R$  is the retention time and  $\sigma$  is the peak width. This relationship shows

that decreasing peak width allows a reduction in analysis time while maintaining the same system efficiency.

In addition to diffusion broadening by the separation column, several other mechanisms contribute to peak broadening, such as injection peak broadening, dead or unswept volumes in the system, and detector volume and response time. The magnitude of these contributions depends on the chromatography hardware used, and should be minimized with respect to the injected sample volume in order to maximize system performance. In other words, if the above-mentioned items are minimized, a much faster analysis time can be obtained with equal separation quality.<sup>2</sup>

A basic gas chromatography system consists of an injector, a column, and a detector. The GC technology of the C2V-200 is built around columns up to 250  $\mu\text{m}$  i.d., which are manufactured either by means of silicon micromachining or glass capillaries. The injector creates the sample plug, which is launched into the separation column. The plug should ideally be as narrow as possible while containing sufficient sample to allow detection. This means that the injected plug should be a rectangular peak with a magnitude of 100%. To achieve this, the injector should meet the following criteria:

- Ultrafast switching capability
- Reproducible timing of the injection
- Negligible sample dilution
- Minimal dead volume.

The microtechnology of the C2V-200 enables the use of small valves with extremely low dead volume. The low mass of the valve membranes permits

switching times on the order of several milliseconds. Dead volumes are also minimized in the sample loop; therefore sample dilution is reduced.

The detector identifies the presence of substances emanating from the separation column. To allow accurate detection of fast, narrow sample peaks without introducing peak broadening, the detector should meet the following requirements:

- Low internal volume
- Fast response
- Negligible sample dilution
- Minimal dead volume.

The internal volume of the microtechnology-based microthermal conductivity detector (TCD) is on the order of several tens of nanoliters. With a typical flow rate of 1 mL/min, this means a peak broadening of several milliseconds. The thermal response of the TCD filaments is on the order of hundreds of microseconds, and the data acquisition must therefore be designed such that it does not contribute to the peak broadening.

## System integration

In addition to the use of properly designed components, system integration plays a major role in the perfor-



Figure 1 C2V-200 fast micro-GC. Cartridge dimensions: 90 × 50 × 15 mm.

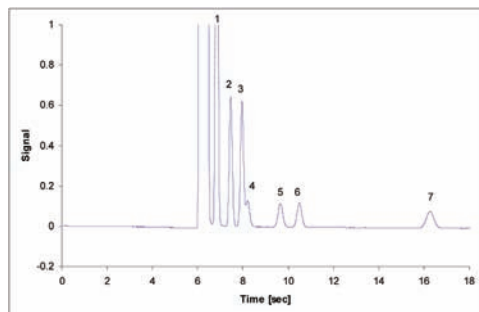


Figure 2 Typical separation of a C1–C6 mixture on a VB-1 column.

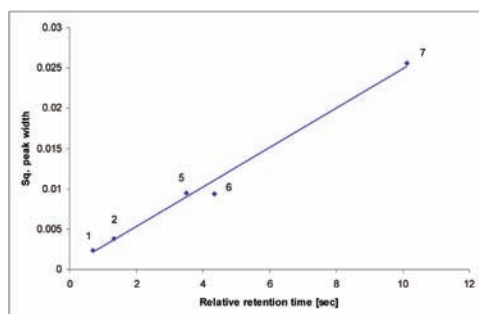


Figure 3 Squared peak width  $\sigma^2$  as a function of relative retention time.

mance of a micro-GC system. Dead volumes or temperature variations (i.e., cold spots) can dramatically deteriorate separation performance. To address this issue, the MicroDELTA™ platform was developed (**Concept to Volume B.V.**). The platform is a hybrid microfluidic integration technology based on conventional integrated circuit (IC) assembly equipment and technology in which components such as sensors and valves are assembled onto a fluidic channel plate. With IC-type bonding techniques, electrical and fluid connections between the channel plate and the components are achieved. The channel plate is fabricated from silicon and glass using micromachining techniques.

For fast GC, a channel plate was developed containing a microvalve-based

injector, an integrated TCD detector, and a flow sensor for system diagnostics. In this way the entire critical fluidic path of the GC system is optimally integrated, resulting in negligible dead volume in the system. Combined with the low internal volume of the micro-components used, this results in minimal peak broadening due to dilution and dead volume.

Combined with a column and low-heat-capacity heaters, the channel plate contains all the critical hardware for a GC system, assembled in a tiny and easily exchangeable GC cartridge with dimensions less than  $90 \times 50 \times 15$  mm (Figure 1).

## Results

Figure 2 shows a typical separation of a C1–C6 mixture on a ValcoBond VB-1 column (Valco Instruments Co., Houston, TX).

The peak width (full width at half maximum [FWHM]) ranges from 100 to 400 msec, and the number of plates,  $N$ , ranges from roughly 10,000 (hexane) to 20,000 (propane).

Plotting the squared peak width  $\sigma^2$  as a function of the relative retention time clearly shows that peak broadening is dominated by the inherent broadening of the chromatographic column, as demonstrated in Figure 3. The corresponding data are given in Table 1.

## Method integration

The above-described system is the most basic GC system. The integration method used enables the efficient integration of more complex GC layouts such as backflush, heart-cut, GC×GC, etc. In addition, the number of avail-

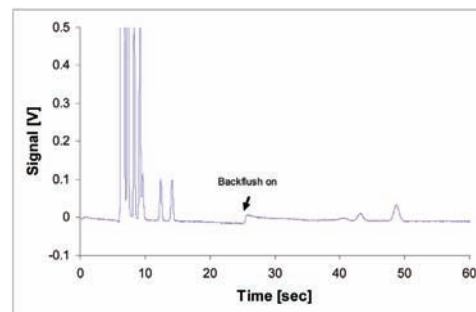


Figure 4 Very fast backflush chromatogram analysis performed by the C2V-200 micro-GC.

able GC microcomponents such as other detectors, component-selective sensors, concentrators, microcolumns, and pressure and flow sensors will grow. Due to the generic nature of the MicroDELTA platform, these can be integrated into a future system. As an illustration, Figure 4 demonstrates a very fast backflush chromatogram of a natural gas mixture.

## Conclusion

This article describes the impact of the chromatography instrumentation hardware and an optimal system approach to serve the very fast chromatography needs of today while exceeding today's separation standards. The MicroDELTA chip technology integration platform enables a new approach to GC instrumentation integration, more specifically, allowing integration of key functional parts such as injector, sample loop, detector, and columns. The technology platform permits a higher integration density of these components as well as further miniaturization of the system, leading to both faster and better chromatography than today's standard.

## References

1. Wilson, J.N. *J. Amer. Chem. Soc.* **1940**, *62*, 1583–91.
2. Grob, R.L.; Barry, E.F. *Modern Practice of Gas Chromatography*; John Wiley & Sons: New York, NY, 2004; 4th ed.; part one.

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**Table 1** Retention time data, peak FWHM, and number of plates for the analysis, as shown in Figure 3

Peak	Retention time	Peak FWHM	No. of plates
Propane (1)	6.8	114	20,000
Iso-butane (2)	7.5	145	15,000
Iso-pentane (5)	9.7	228	10,000
n-Pentane (6)	10.5	228	10,000
n-Hexane (7)	16.3	377	10,000