The FastGC

Innovators of High Resolution Chromatography Products

Fast GC Using Microbore Capillary Columns

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Reducing instrument & operator time for gas chromatographic analyses has become an important consideration for many laboratories.

The use of microbore (0.10mm ID) columns can significantly reduce analysis time without sacrificing resolution. The extremely high efficiency of microbore columns (-7000 plates/meter) can provide resolution of complex mixtures while using shorter lengths. Shorter columns are less expensive and reduce analysis times, resulting in a cost savings for the lab.

Some instrument companies have been promoting the benefits of fast screening columns, but the sacrifices required aren't always evident from their literature. The reduction of analysis time at the expense of resolution, sample capacity, and ease of use is not always an acceptable alternative. This article will discuss and demonstrate the benefits and limitations of 0.10mm ID columns.

Speed and Resolution

Table I compares the characteristics of microbore columns to conventional columns. This data holds the key to whether microbore columns are right for your analysis. The most striking difference of microbore columns is their high efficiency (plates/meter) compared to other diameters. **Table I** indicates that a 0.10mm ID column is 160% more efficient than a 0.25mm ID column. This high efficiency allows shorter columns to maintain excellent resolution and increase the speed of analysis. However, some of the other parameters in **Table I** illustrate limitations that may negate the usefulness of microbore columns in your laboratory. The effect of low flow rates, low sample capacity, and high operating pressures on your sample requirements will ultimately determine if microbore columns are an improvement for your laboratory.

Flow **Rates**

The low flow rates for microbore columns can be either an advantage or a limitation. Low flow rates are beneficial for GCMS users because the flow rates are well within the pumping capacity of most systems. In addition, the microbore prevents "pumping out the column" or operation below atmospheric pressure. This provides more efficiency

Table I:

Column Characteristics

Column ID	0.10mm	0.18mm	0.25mm	0.32mm	0.53mm
Theoretical plates/m	8,600	5,300	3,300	2,700	1,600
Effective plates/m	6,700	3,900	2,500	2,100	1,200
He flow @ 20cm/sec	0.lcc/min.	0.3cc/min.	0.7cc/min.	1.0cc/min.	2.6cc/min.
H2 flow @ 40cm/sec	0.2cc/min.	0.6cc/min.	1.4cc/min.	2.0cc/min.	5.2cc/min.
Sample Capacity	5-10ng	10-20ng	50-100ng	400-500ng	1000-2000ng
Operating Pressures	40.0psig	2l.0psig	12.5psig	7.5psig	3.0psig

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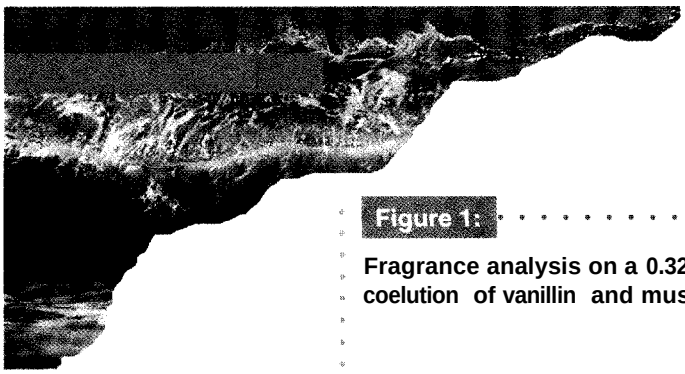


for the end user. However, low flow rates also translate into more flow path problems for the chromatographer. Unswept dead volume has disastrous consequences on the column performance.

Operating Pressures

Table I also shows that microbore columns require higher operating pressures which results in more ferrule leaks, septum leaks, and sample blow back through leaking syringe plungers. Connections need to be monitored for leaks more often. The pneumatic systems for older GCs are designed to operate at only 30psig and may need to be modified to handle higher pressures required for narrow bores. Operating microbore columns below optimum pressures will translate into poor resolution and poor performance.

Sample Capacity

A limiting factor of a microbore column is the amount of sample that can be injected onto the column. Table I indicates that the sample capacity of a microbore column is ten times less than a 0.25mm ID column. Therefore, the on-column injection should be at least ten times lower for a microbore column.

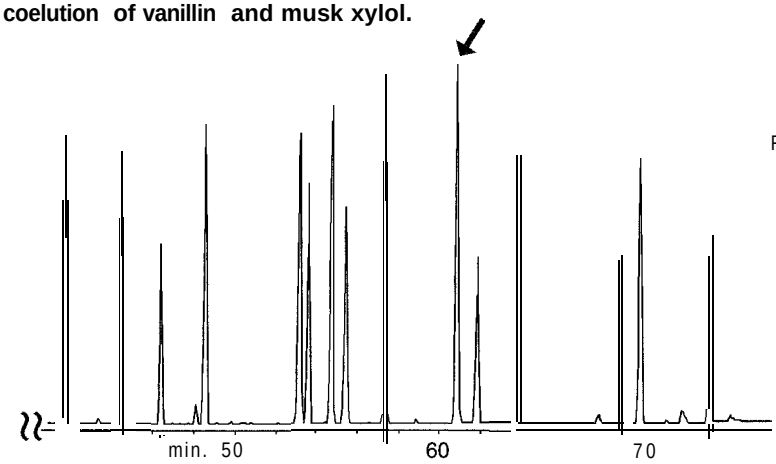
Sample cleanliness is another important factor to take into consideration when using microbore columns. Because the surface area of the 0.10mm ID columns is much lower than a conventional column, contamination will occur more rapidly when dirty samples are injected. This means that 0.25 or 0.32 mm ID columns will be more rugged and require less maintenance for dirty samples than microbore columns. Whenever possible, samples containing non-volatile residue should be avoided. If dirty samples are a must, extensive column and injection port maintenance is required. Otherwise, loss of resolution, ghost peaks, and a high background signal will result.

Injector Considerations

Direct and on-column injection modes are not recommended due to the required low flow rates and small bore size of these columns. Therefore, trace analyses are difficult to perform with microbore columns. Split and splitless injections are the best alternatives. However, since microbore columns require low flow rates, speed of sample transfer through the liner to the column is a concern. Due to the high dead volume, poor peak shape, and response, loss of resolution will occur when 2 or 4mm ID liners are used in conjunction with microbore columns. Thus, lmm ID inlet liners are a must for sharp, well resolved, and recovered peaks. Not only is the inlet liner a consideration when

Figure 1:

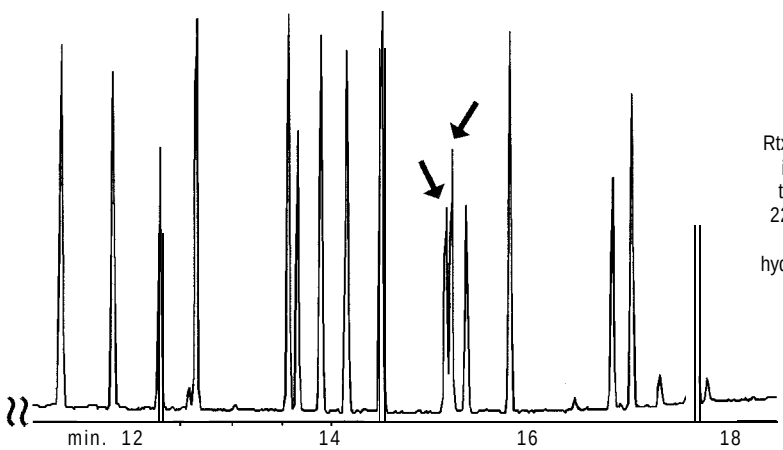
Fragrance analysis on a 0.32mm ID Rtx-Wax column takes 75 minutes with complete coelution of vanillin and musk xylol.



30m, 0.32mm ID, 0.25um Rtx-WAX (cat.# 12424). Splitless injection, 50mls/min. Oven temp.: 75C to 225C @ 70C min. (hold 15 min.). Inj. & det. temp.: 225°C. Carrier gas: helium, 30cm/sec. set @ 75C.

Figure 2:

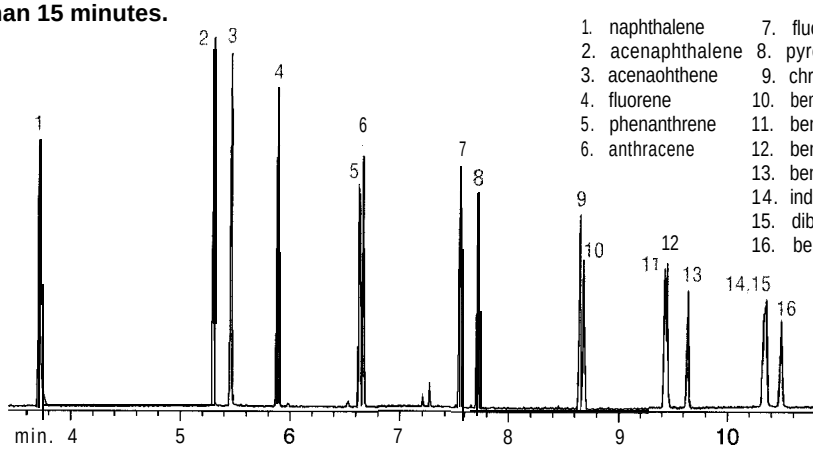
Fragrance analysis on the Rtx-Wax microbore column reduces run times by 75% with increased resolution between vanillin and musk xylol.



10m, 0.10mm ID, 0.20um Rtx-WAX (cat.# 41603). Splitless injection, 250mls/min. Oven temp.: 55°C (hold 1 min.) to 225°C @ l0C/min. Inj. & det. temp.: 225°C. Carrier gas: hydrogen, 70m/sec. set @ 55°C.

Figure 3:

Polynuclear aromatic hydrocarbons on an Rtx-5 microbore column are analyzed in less than 15 minutes.



l0m, 0.10mm ID, 0.10um Rtx-5 (cat.# 41201) 0.5ul splitless injection. 41psi initial pressure, hold 2 min. 8 psi/min. to 99psi (hold 1.87 min.). 275C, vent open @ 1 min. Oven temp.: 40C (hold 0.5 min.) to 90°C @ 70C/min. then to 100°C @ 5C/min then to 310°C @ 30C/min. (hold 2 min.).

using microbore columns for split or splitless injections, but other parameters specific to the type of injection method must also be optimized.

In a split injection, the choice of inlet liner and initial temperature will affect peak shape, response, and resolution the most. **Figure 1** shows part of a typical fragrance analysis on a conventional column (0.32mm ID). Under optimal conditions (4mm ID inlet liner and initial temperature of 75"(Z), the analysis time is more than 70 minutes and the separation of vanillin and musk xylol could not be achieved. By switching to a microbore column and optimizing run conditions (lmm ID inlet liner and initial temperature of 55C), we were able to reduce the analysis time to 18 minutes and attain 80% resolution of the vanillin and musk as shown in **Figure 2.** The 1 mm ID inlet liner improved the recovery and peak shape of the early eluting compounds.

Figure 3 illustrates a splitless PAH analysis on a 0.10mm ID, 0.10um Rtx-5 using an optimized inlet liner and inlet pressure. When a 2mm ID inlet liner was used, high molecular weight discrimination occurred. By changing to a lmm ID inlet liner, high molecular weight discrimination was eliminated. However, this change caused peak splitting of the early eluting compounds. The peak splitting was eliminated completely when pressure programming was applied in place of constant pressure.

Detector Considerations

Detector design and flows must be optimized when using microbore columns. Make up gas flows may need to be increased to minimize detector dead volume and compensate for the lower column fl w rates. Since peak widths are approximately half compared to conventional columns (< 1 second), fast integrator and detector electrometers must be used. Integrator sampling rates must be increased over rates used for 0.25mm ID columns since the peaks are much narrower with microbores. If the sampling rate is too slow, then poor integration and non-reproducible peak areas will result. Check with your instrument company and data system manufacturer to be sure your system is capable of handling microbore sampling rates.

Microbore columns can produce shorter analysis times, equivalent resolution, and provide cost savings. But remember, converting your conventional system to a microbore system isn't as easy as changing columns. Column capacity, sample purity, and injector and detector conditions must be considered and optimized for a successful analysis. Keep in mind that when switching from conventional capillaries to microbore columns, there may be the need to optimize inlet temperatures, liners, and GC run conditions.

Product Listing:

Microbore Capillary Columns			
0.10mm ID, 0.10µm			
Column	**temp. limits**	**10-meter**	**20-meter**
Rtx- 1	-60 to 330/350C	41101	41102
Rtx-5	-60 to 330/350C	41201	41202
Rtx-Wax	20 to 250C	41601	41602
0.10mm ID, 0.20µm			
Column	**temp. limits**	**10-meter**	**20-meter**
Rtx-Wax	20 to 240/250°C	41603	41604
0.10mm ID, 0.40µm			
Column	**temp. limits**	**10-meter**	**20-meter**
Rtx-1	-60 to 320/340C	41103	41104
Rtx-5	-60 to 320/340C	41203	41204

Contact Restek's GC experts to discuss the suitability of Microbore or other GC columns for your specific application.