Reduce GC Analysis Time Without Switching to Hydrogen by Using 0.15–0.18 mm ID Fast GC Capillary Columns

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Time is money, so labs are always interested in reducing GC analysis time so that more samples can be analysed in a given period. There are multiple ways to accomplish this, but care must always be taken to preserve critical separations between peaks. The only way to maintain resolution when speeding up GC analyses is to find a solution that supplies a theoretical plate count that is similar to the original method. Using hydrogen carrier gas is one way to do this, but safety concerns keep some labs from implementing this strategy. If converting to hydrogen is not an option, using smaller internal diameter (ID) columns is an effective solution. With smaller ID columns, the same efficiency can be achieved with a shorter column length. Shorter, smaller ID columns are faster and have a higher optimal linear velocity. Typically, 0.15–0.18 ID columns are used for fast GC due to their high efficiency and shorter analysis times. Also, these columns can be coated with films of up to 2 µm when loadability is a concern. Successfully converting to a smaller ID column is a simple way to achieve faster analysis times, but one must maintain efficiency, phase ratio, and elution temperature in order to obtain a similar separation.

Maintain Efficiency When Using a Smaller ID Capillary Column

Once the decision is made to use a smaller ID column, there are several choices available, varying from 0.1 mm ID up to 0.18 mm ID. Although the 0.1 mm ID columns have been available for a long time, their practical application is not as simple as is often claimed. The biggest challenges with 0.1 mm ID columns are sample introduction, loadability, and robustness. A 0.15 mm ID column is a good alternative that has been proven to work in nearly all existing GC systems. To keep the same efficiency, one uses a shorter length column and operates at a slightly higher linear velocity. Because a 20 m x 0.15 mm ID column provides, on average, 10% higher separation efficiency compared to a 30 m x 0.25 mm ID column, both columns will provide similar separation power. Table I shows typical replacement column dimensions. Reducing analysis time by a factor of two while still achieving comparable separation efficiency is a realistic outcome when using a smaller ID column and existing instrumentation.

Table I: Replacement column dimensions that will generate equivalent or improved efficiency

Current Column	Recommended Replacement
15 m x 0.25 mm x 0.25 µm	10 m x 0.15 mm x 0.15 µm
30 m x 0.25 mm x 0.15 µm	20 m x 0.15 mm x 0.15 µm
60 m x 0.25 mm x 0.15 µm	40 m x 0.15 mm x 0.15 µm
15 m x 0.32 mm x 0.15 µm	10 m x 0.15 mm x 0.15 µm
30 m x 0.32 mm x 0.15 µm	15 m x 0.15 mm x 0.15 µm
60 m x 0.32 mm x 0.15 µm	30 m x 0.15 mm x 0.15 µm

In addition to reducing analysis time while maintaining the original separation, moving to fast GC with a smaller ID analytical column also means the same sensitivity can be obtained using only half of the sample. This is because the eluting peaks are 2x higher, so one needs to inject only 50% of sample in order to get the same signal strength. Since less sample is introduced contamination of the column and liner will also be reduced, which translates into lower maintenance costs per analysis.

Choice of Stationary Phase and Film

When changing columns, getting the same peak elution order is preferred. To ensure this, it's important to use the same stationary phase in the smaller bore column as was used in the larger bore column. For most phases this is not a problem, but for "5" type columns, it can be a challenge as there are two kinds of phases available: 5% phenyl/95% methyl phases and their

Figure 1: Selectivity difference for xylenes between 5% phenyl and silarylene phases. (Columns: 60 m x 0.25 mm x 0.25 µm, Oven: 30 °C)

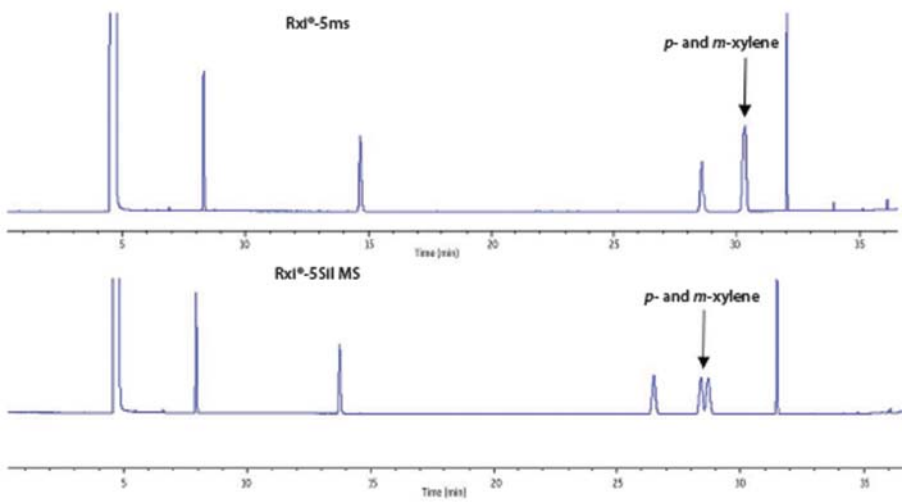
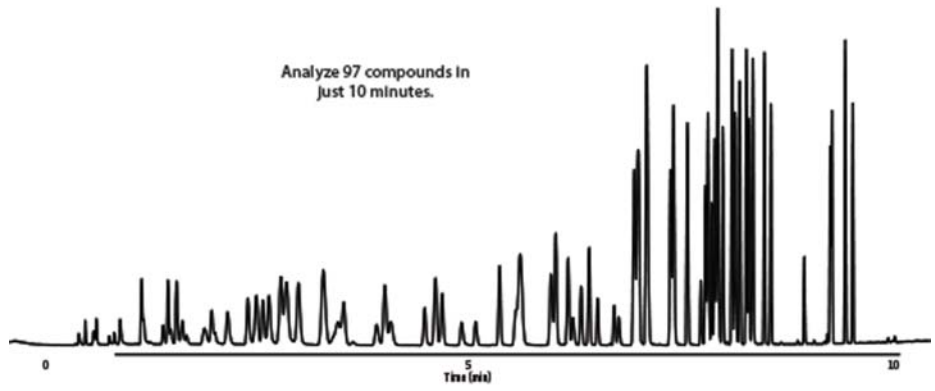
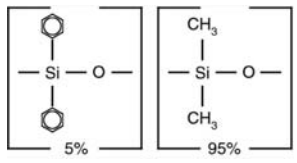
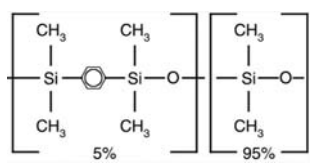


Figure 2: Separation of volatiles in a short analysis time using a thick film 0.18 mm ID capillary GC column



Column: Rtx®-VMS, 20 m x 0.18 mm x 1.00 µm; Sample: U.S. EPA Method 8260B compounds; Conc.: 5 ppb in 5 mL water; Injection: Split (1:40); Flow: 1.0 mL/min, constant flow; Oven: 50 °C (hold 4 min) to 100 °C at 18 °C/min to 230 °C at 40 °C/min (hold 3 min); GC: HP6890; Detector: 5973 MS, scan 35-260 amu; P&T: Tekmar 3100

Table II: Common "5" type columns and their respective Rxi® equivalents. Note that standard 5% phenyl columns and their silarylene-based counterparts differ in selectivity

Recommended Columns	Rxi-5ms	Rxi-5Sil MS
Structure		
Description	5% phenyl	Silarylene stabilised
Similar to:		
Restek	Rtx-5	Rtx-5Sil MS
Agilent	HP-5, HP-5ms, DB-5, Ultra-2	DB-5ms, DB-5msUI
Varian	CP-Sil 8 CB	VF-5ms, CP-Sil 8 CB-LowBleed/MS
Alltech	AT-5ms	--
Supelco	SPB-5, Equity-5	SLB-5ms
SGE	BP5ms	BPX5
Phenomenex	ZB-5, ZB-5ms	ZB-5msi
Macherey-Nagel	OPTIMA 5, OPTIMA 5 MS	OPTIMA 5MS Accent

silarylene-stabilised counterparts. These phases differ significantly in chromatographic selectivity. For example, using the same column dimensions, the peak elution order for a simple mixture containing xylenes is completely different (Figure 1). The Rxi®-5Sil MS (silarylene) column separates para- and meta-xylene, while the Rxi®-5ms (5% phenyl) column does not. With more complex mixtures these differences increase, making accurate identification and quantification quite difficult. To simplify conversion and to allow analysts to take advantage of the exceptionally inert and low bleed Rxi® column technology, Restek makes both phases available. For both types of phases, Table II lists commonly used columns and their respective Rxi® equivalents.

In addition to using the same phase type when changing to smaller ID columns, it is best to choose a column with a film thickness that gives a similar phase ratio. This ensures that the retention factor for all components remains the same, which simplifies method conversion. Film thicknesses range from 0.10 µm to 2.0 µm, depending on column ID, and the thicker film coatings are recommended when greater loadability is needed. Figure 2 shows an example using a very popular column that is already widely used, the 0.18 mm ID Rtx®-VMS column with a 1.00 µm film coating. This column is used for volatiles separation in existing GC configurations and it produces excellent results.

Figure 3: Similar separations of a complex mixture are achieved in half the time when analysed using fast GC on a smaller ID column. See Table III for method conditions

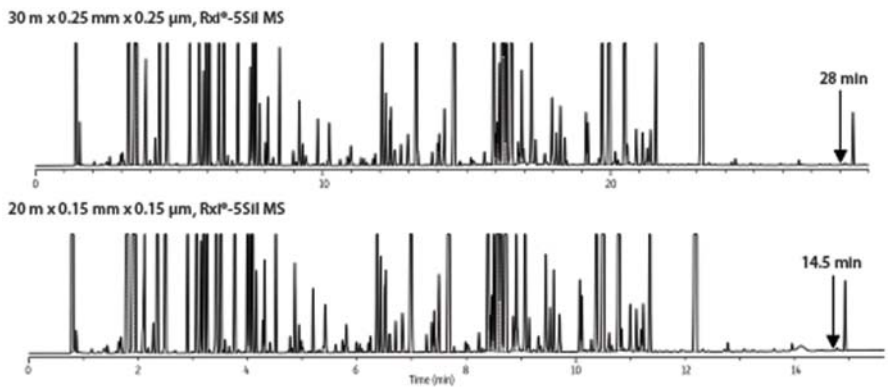
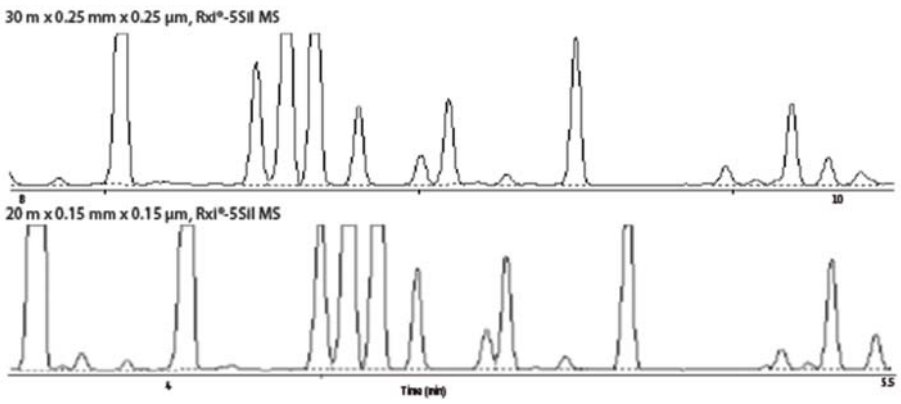


Table III: Conditions for Figures 3 and 4. Original method conditions were adjusted so similar elution temperatures and separations would be maintained when switching to a smaller ID column

	Original Method (0.25 mm ID)	Faster Method (0.15 mm ID)
Column	Rxi-5Sil MS, 30 m x 0.25 mm x 0.25 µm	Rxi-5Sil MS, 20 m x 0.15 mm x 0.15 µm
Carrier Gas	H_2, 1.2 mL/min, 36 cm/sec, constant flow	H_2, 0.84 mL/min, 50 cm/sec, constant flow
Injection	1.0 µL split (1:100)	1.0 µL split (1:100)
Oven	100 °C (hold 2 min) to 250 °C at 5°C/min	100 °C (hold 0.9 min) to 250 °C at 9.75 °C/min
Instrument	Agilent 6890	Agilent 6890

Figure 4: Zoom of separations obtained in Figure 3. Peak elution is very similar because similar elution temperatures were maintained. See Table III for method conditions



Changing the Oven Temperature Program

Besides using the same stationary phase and phase ratio, a similar peak profile can only be achieved by adjusting the oven temperature programming rate so that the peak elution temperatures are the same as when using the longer, larger ID column. To obtain the same elution temperature, the new conditions have to correct for the column length and the carrier gas linear velocity. For example, consider the analysis of a complex mixture shown in Figure 3. The same sample was analysed using a 30 m x 0.25 mm column and a 20 m x 0.15 mm column, both of which have a similar phase ratio. The chromatograms are nearly identical as the carrier gas flow and oven temperature program were modified in order to generate similar elution temperatures (Table III). Figure 4 shows a magnification of a section of these analyses, confirming that the elution is very similar. By using a properly modified method, resolution was maintained while cutting analysis time in half. For method conversion calculations, one always can contact the Technical Support group or download a "method translator", which is freeware developed by Leonid Blumberg (Hewlett Packard) many years ago.

Summary

If it is not possible to use hydrogen as the carrier gas, there is still a simple way to reduce analysis time and perform fast GC using existing instrumentation. Using a smaller ID capillary column will provide the necessary efficiency in a shorter analysis time. This is done routinely and works well with proper method conversion, which entails making small changes to inlet pressures, split ratio, and the oven temperature program. By using a similar phase and phase ratio along with properly converted method conditions, cutting analysis times by a factor of 2 can be easily achieved.