

High Performance RediSep Rf Gold® Columns on CombiFlash® Companion XL systems

Abstract

The 750 and 1500 gram columns packed with high performance RediSep Rf Gold silica show the expected reduction in run time and solvent usage when compared to standard RediSep® media on a CombiFlash® Companion® XL system while providing comparable purity. Using RediSep Rf Gold columns on a Companion XL system reduces total solvent usage by at least 42 percent and run time by at least 22 percent.

Results and Discussion

RediSep Rf Gold columns are known for having increased resolution and the ability to reduce run times and save solvent. The 750 and 1500 g columns were packed with standard Flash silica and RediSep Rf Gold spherical silica. Columns packed with the RediSep Rf Gold silica were able to run with a steeper gradient profile, thereby reducing run times. They also used reduced flow rates to accommodate the smaller particle size of the RediSep Rf Gold silica.

Gold Resolution

Figure 1 shows the purifications of catechol and resorcinol on a standard RediSep Rf column using a standard method and RediSep Rf Gold column using the Gold Resolution method. The sample load (7.5 g) was at 1% or less of the 750 g column media weight.

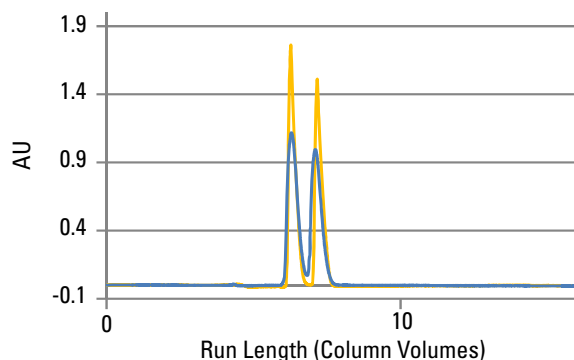


Figure 1: Gold Resolution comparison of 750 gram columns The high performance RediSep Rf Gold column and Gold Resolution method (gold trace) showed improved resolution over the standard column and method (blue trace).

The RediSep Rf Gold column exhibited baseline resolution of the two compounds. A TLC evaluation of the results confirmed the improved purity from the RediSep Rf Gold column (Figure 2).

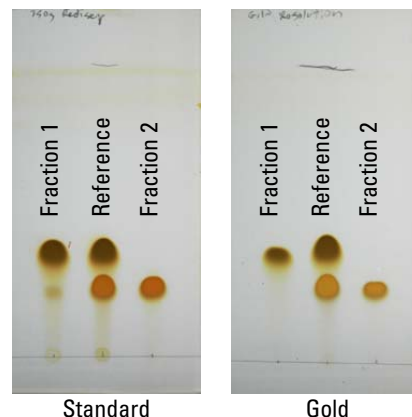


Figure 2: TLC evaluation of compound purity Fractions from the RediSep Rf Gold columns in Gold Resolution mode showed greater purity.

Gold Speed

A Gold Speed method is recommended for compounds exhibiting a $\Delta R_f > 0.1$. This method saves time and solvent while still producing pure materials.

Figure 3 compares a RediSep Rf Gold column run using a Gold Speed method to a RediSep Rf column using a standard method. The Gold Speed method still provides pure compounds as indicated by TLC (Figure 4).

Table 1 shows that the 750 g RediSep Rf Gold column using a Gold Speed method saved 42 percent of solvent (including equilibration) and 22 minutes (including equilibration and column air purge).

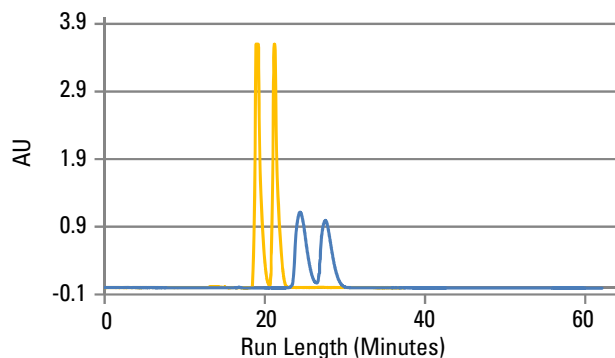


Figure 3: Gold Speed comparison of 750 gram columns The RediSep Rf Gold column and Gold Speed method (gold trace) resolved the closely eluting compounds at a greater speed than the standard column and method (blue trace).

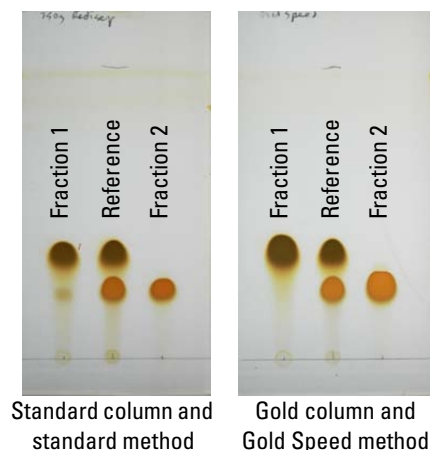


Figure 4: TLC evaluation of compound purity
Fractions from the RediSep Rf Gold columns in Gold Speed mode showed better purity.

Scaling up to a 15 g sample on a 1500 g RediSep Rf Gold column showed similar results (Figures 5 and 6).

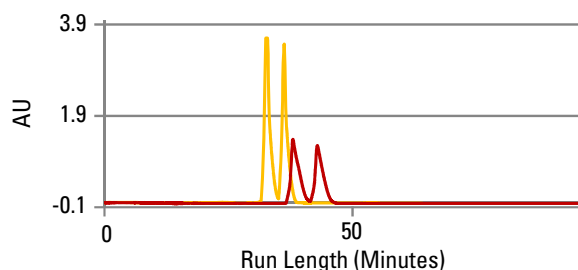


Figure 5: Gold Speed comparison of 1500 gram columns High performance RediSep Rf Gold column (gold trace), standard RediSep Rf column (red trace).

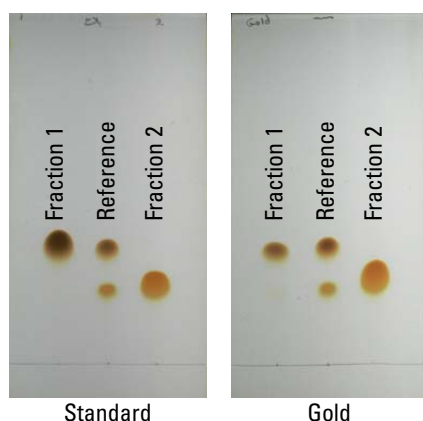


Figure 6: TLC evaluation of compound purity
1500 g RediSep Rf Gold columns in Gold Speed mode showed comparable purity while saving time and solvent.

Table 1: Gold Speed Time & Solvent Savings

Column Size	Total Solvent used	Total Run Time	Solvent Saved (Liters, %)	Time Saved (minutes, %)
1500 g	23.2 L	98 min	18.5 L 44%	28 min 22%
750 g	13 L	68 min	9.3 L 42%	22 min 24%
330 g	3.7 L	61 min	3.6 L 42%	35 min 36%
220 g	2.6 L	36 min	2.6 L 45%	26 min 42%
120 g	1.7 L	38 min	1.6 L 45%	26 min 40%

Methods

Tables 2 and 3 list the parameters that must be modified to update the Companion XL system's default methods to Gold Resolution or Gold Speed methods optimized for RediSep Rf Gold columns.

Table 2: Gold Resolution Method Changes

Part Num	Size	Run Length (CV)	Flow Rate (mL/min)	Threshold (AU)	Peak Width (min)
69-2203-428	1500 g	16	300	0.2	4
69-2203-427	750 g	16	250	0.2	4
69-2203-369	330 g	16	100	0.2	4
69-2203-359	220 g	16	100	0.2	4
69-2203-369	120 g	16	85	0.2	4

Table 3: Gold Speed Method Changes

Part Num	Size	Run Length (CV)	Flow Rate (mL/min)	Threshold (AU)	Peak Width (min)
69-2203-428	1500 g	8	300	0.25	2 min
69-2203-427	750 g	8	250	0.25	2 min
69-2203-369	330 g	8	100	0.25	2 min
69-2203-359	220 g	8	100	0.25	2 min
69-2203-369	120 g	8	85	0.25	2 min

Conclusion

The proven benefits of RediSep Rf Gold columns with Gold Resolution and Gold Speed methods can be duplicated on the CombiFlash Companion XL system for large scale purifications.

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