High Performance RediSep Gold® Columns

on CombiFlash® systems

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Chromatography Application Note AN73

Abstract

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

Results and Discussion

RediSep 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 Gold spherical silica. Columns packed with the RediSep 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 Gold silica.

Gold Resolution

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

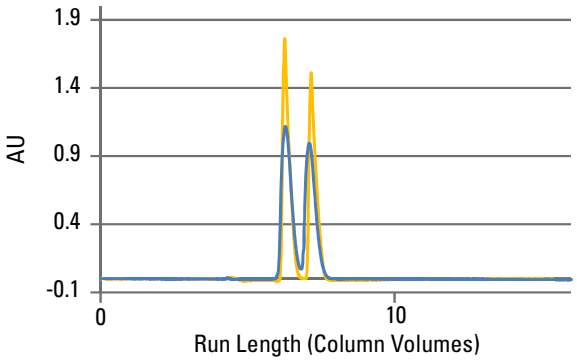


Figure 1: Gold Resolution comparison of 750 gram columns The high performance RediSep Gold column and Gold Resolution method (gold trace) showed improved resolution over the standard column and method (blue trace).

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

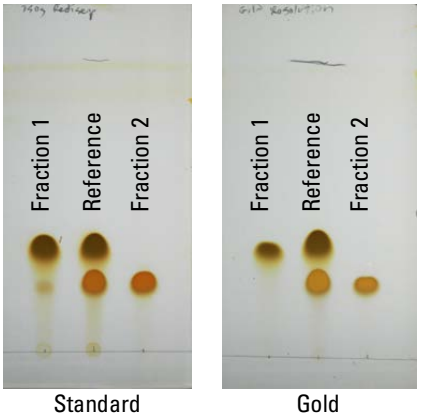


Figure 2: TLC evaluation of compound purity Fractions from the RediSep Gold columns in Gold Resolution mode showed greater purity.

Gold Speed

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

Figure 3 compares a RediSep Gold column run using a Gold Speed method to a RediSep 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 Gold column using a Gold Speed method saved 42 percent of solvent (including equilibration) and 22 minutes (including equilibration and column air purge).

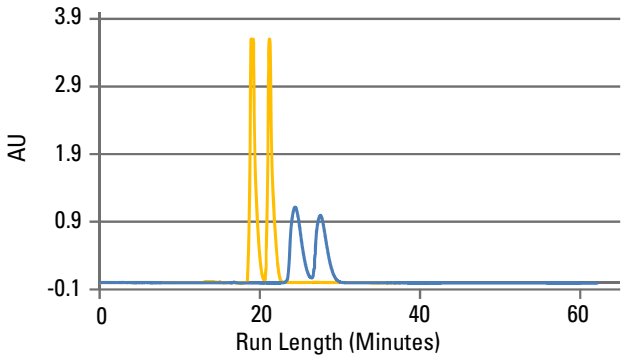
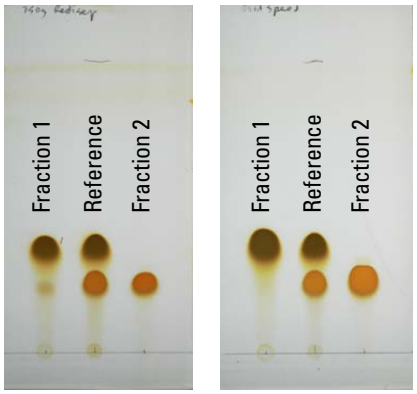


Figure 3: Gold Speed comparison of 750 gram columns The RediSep Gold column and Gold Speed method (gold trace) resolved the closely eluting compounds at a greater speed than the standard column and method (blue trace).



Standard column and standard method | Gold column and Gold Speed method

Figure 4: TLC evaluation of compound purity Fractions from the Redi*Sep* Gold columns in Gold Speed mode showed better purity.

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

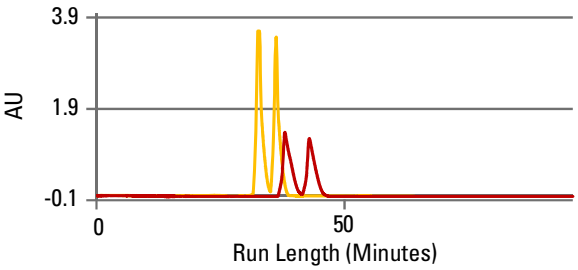
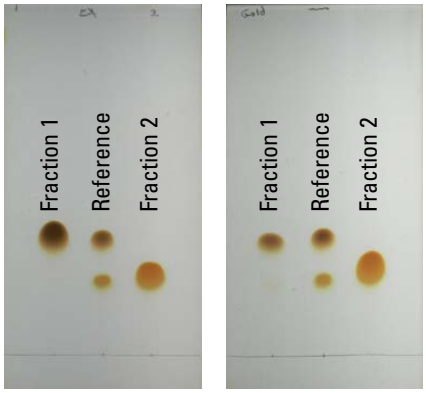


Figure 5: Gold Speed comparison of 1500 gram columns High performance Redi*Sep* Gold column (gold trace), standard Redi*Sep* column (red trace).



Standard | Gold

Figure 6: TLC evaluation of compound purity 1500 g Redi*Sep* 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

Updating the default methods of the Combi*Flash* system to Gold Resolution or Gold Speed methods optimized for Redi*Sep* Gold columns would have required modification of the parameters listed in Tables 2 and 3.

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
69-2203-349	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
69-2203-349	120 g	8	85	0.25	2 min

Conclusion

The proven benefits of Redi*Sep* Gold columns with Gold Resolution and Gold Speed methods can be duplicated on a Combi*Flash* system for large scale purifications.

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