Improved Productivity and Savings

with Redi*Sep* Gold® Columns

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Chromatography Application Note AN72

Abstract

The Redi*Sep* Gold® high performance columns now provide organic chemists with more resolving capability in their flash chromatography purification through the utilization of smaller, spherical media. Finer particle sizes (20–40 µm) easily purify compounds that were previously unresolved with classic flash grade silica (40–60 µm, 60Å).

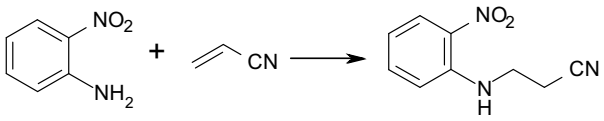
Redi*Sep* Gold high performance columns are filled with spherical media that enhance resolution while lower back pressures allow higher flow rates and shorter run times. The properties of the Redi*Sep* Gold high performance columns used on the Combi*Flash*® system will result in an average of 30% or greater laboratory savings of time and solvent.

When used on the Combi*Flash* system, RFID tags recognize the purification power of the Redi*Sep* Gold high performance columns and provide the option of separating with "Gold Resolution" for compounds with a ΔR_f of ≤0.1 or with "Gold Speed" for compounds with a ΔR_f of >0.1 which shorten the run time and increase flow rate. Shorter runs also help purify compounds that are sensitive to silica.

Examples are shown to compare the faster purification times of Redi*Sep* Gold high performance columns to standard 40–60 µm silica gel flash columns and also 20–40 µm irregular particle, high performance columns.

Results and Discussion

Fast purification of 3-(2-nitrophenyl amino) propionitrile



3-(2-nitrophenyl amino) propionitrile was synthesized and the reaction product adsorbed onto celite. This was purified on a Combi*Flash* system using both a standard flash column and Redi*Sep* Gold high performance silica gel column. Both columns gave baseline purification of the product, but the Redi*Sep* Gold column provided improved resolution.

Table 1: Run conditions for Figure 1

Column size:	40 g
Load:	0.4 g (1% load)
Solvents:	Hexane and acetone
Gradient:	0 to 100% acetone
Detection wavelength:	229 nm
Flow rate:	40 mL/min
Run time:	19 min

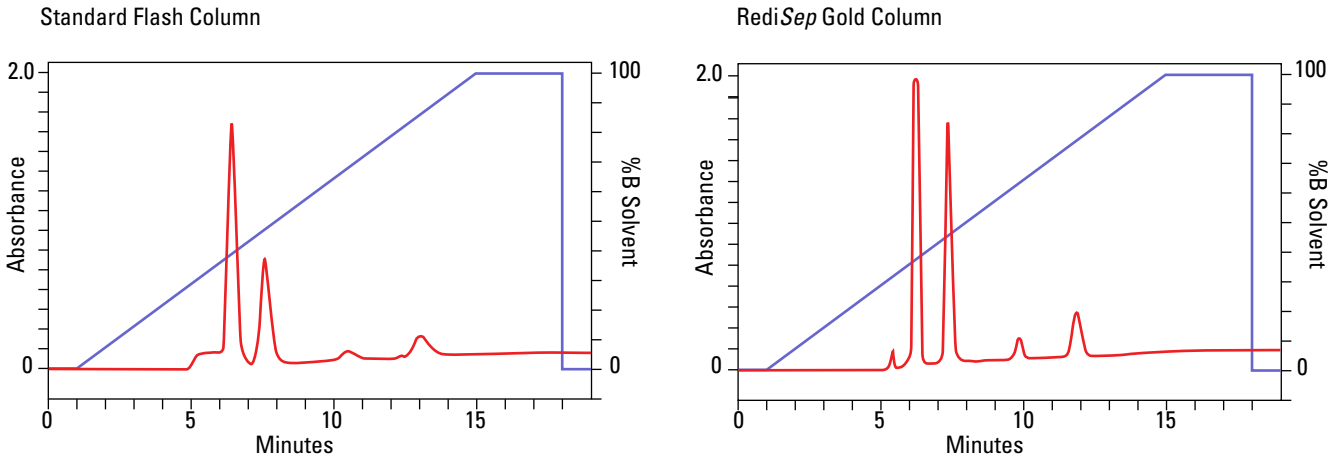


Figure 1: Purification of 3-(2-nitrophenyl amino) propionitrile on a 40 gram standard flash column (left) and a Redi*Sep* Gold column (right) using the standard Combi*Flash* parameters.

This higher, "excess" resolution was employed to reduce the run time of the purification. The run time was shortened by adjusting the gradient slope and increasing the flow rate. The resulting run time of Gold Speed optimized method for Redi*Sep* Gold column is only 5 minutes versus 18 minutes with a standard flash column. Figure 2 shows purification on the Redi*Sep* Gold columns with dramatically less time and solvent. Because of the spherical packing of the Redi*Sep* Gold column, the backpressure was only 65 psi even at the high flow rates of 80 mL/minute. The backpressure of smaller particle size, irregular particle columns exceeded the pressure capabilities of the columns and system.

Entire purification time from start to finish is 29 minutes for the standard method, including the column equilibration and post-run air purge. This is reduced to only 10 minutes by using Gold Speed method optimized for the Redi*Sep* Gold high performance columns. Actual run time was 18 minutes versus 5 minutes. Reducing the run time saved 430 mL of solvent compared to the standard method. The Combi*Flash* system recognizes the Redi*Sep* Gold high performance column through RFID technology and loads the Gold Speed parameters. At a click of a button labs can save 30–50% on their solvent usage and greatly reduce purification time. Greater savings are seen with larger columns.

Table 2: Run conditions for Figure 2

Column size:	40 g
Load:	0.4 g (1% load)
Solvents:	Hexane and acetone
Gradient:	0 to 100% acetone
Detection wavelength:	229 nm
Flow rate:	40 mL/min : 80 mL/min
Run time:	18 minutes for the standard flash column; 5 minutes for the Redi*Sep* Gold high performance column

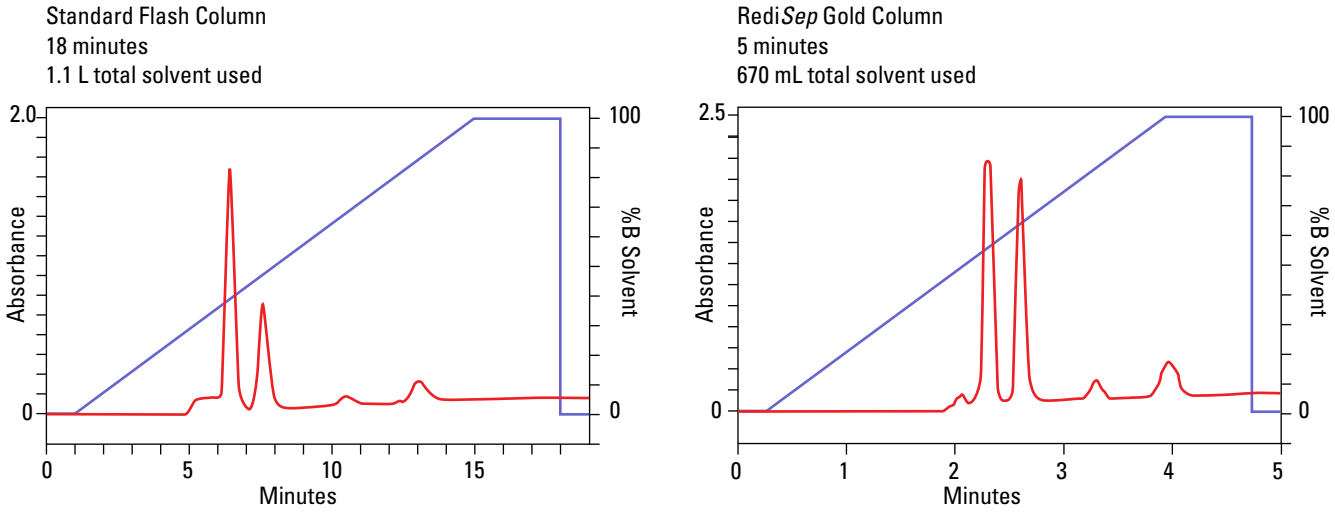
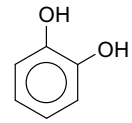


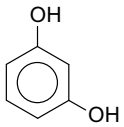
Figure 2: Gold Speed Purification of 3-(2-nitrophenyl amino) propionitrile on a 40 gram standard flash column (left) and a Redi*Sep* Gold high performance column (right) with Gold Speed parameters

Comparison to Irregular High Performance Silica

Spherical silica shows higher resolution when compared to similar size irregular silica when run with the Gold Speed methods. Figure 3 shows a purification of catechol and resorcinol using a column packed with irregular high performance silica gel (20–40 µm) compared to a Redi*Sep* Gold high performance column.



catechol



resorcinol

Under the same conditions, the Redi*Sep* Gold high performance column achieved near baseline resolution of both compounds. The irregular particle, high performance column has unresolved products and runs under conditions that are in excess of recommended pressure capabilities of the column and system.

Table 3: Run conditions for Figure 3

Column size:	400 g
Load:	0.40 (1.0% load)
Solvents:	Hexane and ethyl acetate
Gradient:	0 to 100% ethyl acetate
Detection wavelength:	275 nm
Flow rate:	40 mL/min
Run time:	10 minutes (both columns)

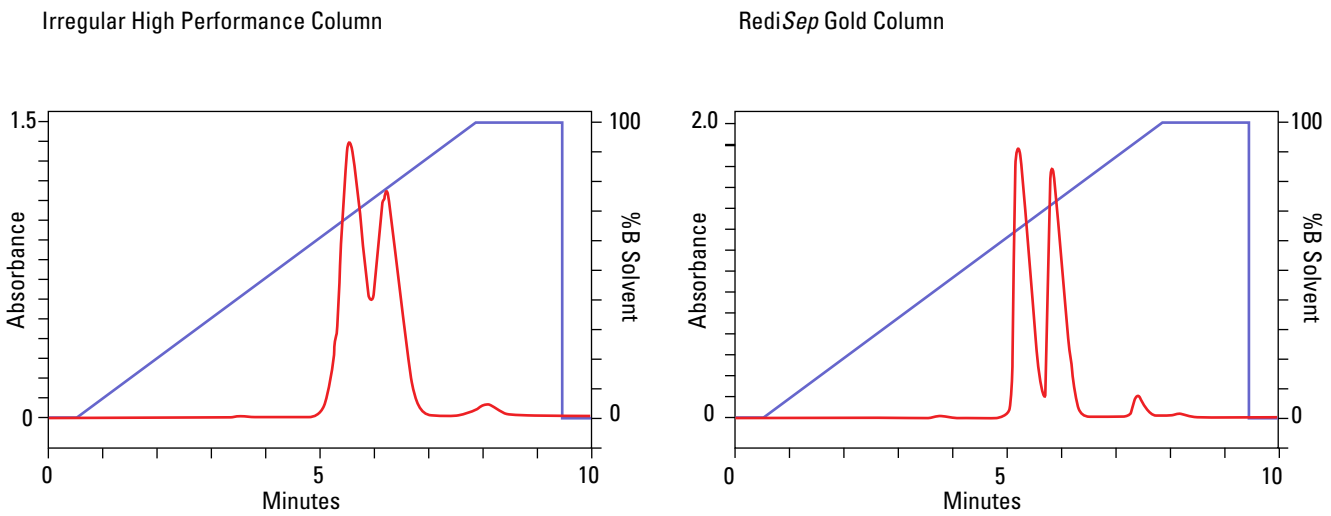


Figure 3: Gold Speed Comparison of irregular, high performance column (20–40 µm, left) to Redi*Sep* Gold high performance column (right) purifying catechol and resorcinol.

Conclusion

The Redi*Sep* Gold high performance columns provide superior resolution compared to standard silica flash columns and irregular particle high performance flash columns. This enhanced resolution can be used to purify closely eluting compounds using the Gold Resolution methods on the Combi*Flash* system or reduce the total run time with the Gold Speed methods. The Combi*Flash* system allows easy setup and choice between the performance options via the RFID tags.

Reducing the total run time shows 30–50% savings in time and solvent usage. Indirect savings from the use of Redi*Sep* Gold columns include reduced solvent waste and time savings due to the reduced number of fractions that need to be processed. Since the peaks are sharper, fractions are collected in fewer test tubes. This reduced the time to evaporate the solvent from these fractions by almost two-thirds.

Redi*Sep* Gold Ordering Information:

Part Number	Description
69-2203-344	Redi*Sep* Gold Column, 4 g, pkg of 14
69-2203-345	Redi*Sep* Gold Column, 12 g, pkg of 14
69-2203-346	Redi*Sep* Gold Column, 24 g, pkg of 10
69-2203-347	Redi*Sep* Gold Column, 40 g, pkg of 10
69-2203-348	Redi*Sep* Gold Column, 80 g, pkg of 6
69-2203-349	Redi*Sep* Gold Column, 120 g, pkg of 6
69-2203-359	Redi*Sep* Gold Column, 220 g, pkg of 4
69-2203-369	Redi*Sep* Gold Column, 330 g, pkg of 3
69-2203-427	Redi*Sep* Gold Column, 750 g, pkg of 3
69-2203-428	Redi*Sep* Gold Column, 1500 g, pkg of 2

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