

Higher Resolution Results with RediSep Rf Gold™ Silica Columns

Flash chromatography purification using smaller, spherical particles

Abstract

The RediSep Rf Gold silica columns now provide organic chemists with more resolution capability in their Flash chromatography purification. Through smaller particle sizes, compounds that were previously unresolved with classic Flash grade silica (40–60 μm , 60Å) are now easily purified.

RediSep Rf Gold silica columns are distinguished in the market by an exclusive smaller, spherical particle (20–40 μm) that enhances resolution while minimizing back pressure. Proprietary end user purifications are used as examples.

Results and Discussion

Example 1: Enhanced Resolution – RediSep Rf Silica vs. RediSep Rf Gold Silica

The separation of various α -substituted bromotoluenes, purified on a CombiFlash Rf system, is compared in Figure 1, courtesy of an end user. All run conditions were constant for both columns.

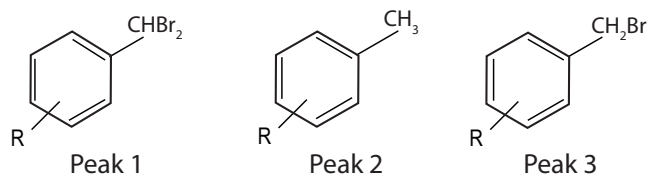


Table 1: Run Conditions for Example 1

Column size:	40 g
Load:	500 mg (on 5 g cartridge)
Solvents:	Hexane and Ethyl Acetate
Gradient:	0-20%
Flow rate:	40 ml/min
Run time:	15 min.
Wavelength:	254 nm

The RediSep Rf Gold columns provided baseline resolution of all three compounds. The ΔR_f between any two adjacent compounds was 0.08. Purification of 3 distinct compounds was obtained on the RediSep Rf Gold silica column.

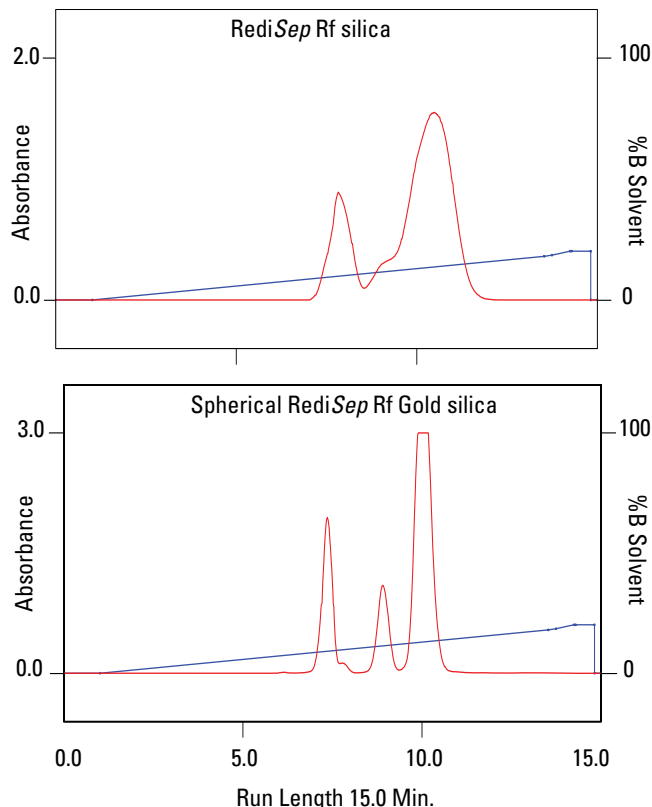


Figure 1: CombiFlash Rf system chromatogram of 500 mg purification of various bromotoluenes

using a regular RediSep Rf silica column (top) and a RediSep Rf Gold silica column (bottom)

Example 2: High Resolution with Low Back Pressure on RediSep Rf Gold Silica

A comparison between the purification on a CombiFlash Rf system using a standard RediSep Rf silica column, the new RediSep Rf Gold silica packed column, and an alternative smaller particle size with irregular media, is shown in Figure 2, courtesy of an end user. All run conditions were constant for both columns.

The standard RediSep Rf silica and RediSep Rf Gold silica columns both ran at a relatively low back pressure (17 psi). The RediSep Rf Gold achieved purification of the desired compound, where the standard RediSep Rf did not resolve the intermediate and desired compound.

The spherical silica media in the RediSep Rf Gold columns provide a better separation at lower back pressures than a column packed with irregular silica. The higher back pressure of irregular media would also limit the flow rate range available on lower pressure

Flash chromatography systems (*i.e.*, CombiFlash Companion), reduce the resolving capability of the column, and increase the purification time.

RediSep Rf Gold silica columns are able to purify compounds with better resolution at much lower back pressure, with no increase in purification time, than any other option in Flash chromatography columns.

Table 2: Run Conditions for Example 2

Column size	40 g
Load	333 mg (on 5 g cartridge)
Solvents	Hexane and Ethyl Acetate
Gradient	0–100%
Flow rate	40 mL/min.
Run time	19.4 min.
Wavelength	254 nm

Conclusion

RediSep Rf Gold columns, packed with smaller, spherical particles, provide higher resolution than conventional Flash columns without the higher back pressure of irregular material. The columns also allow purification of very difficult compounds ($\Delta Rf < 0.1$) with no change in method parameters. The reduced back pressure allows these columns to be used on all models of Flash chromatography systems.

Table 3: RediSep Rf Gold Columns

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

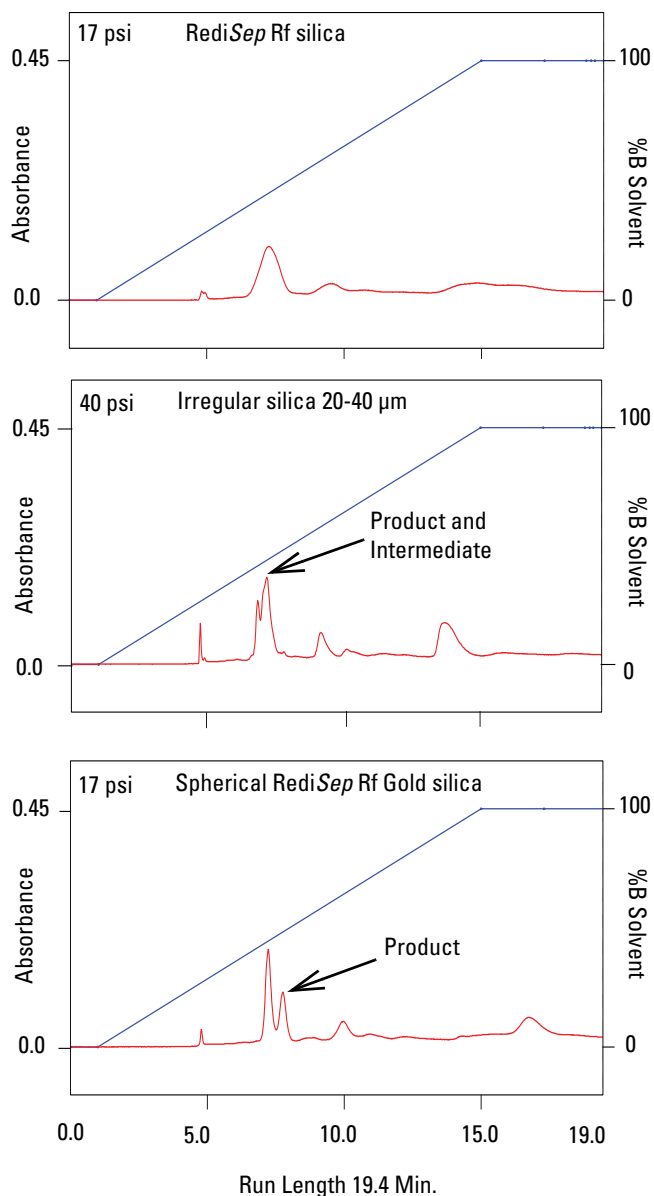


Figure 2: CombiFlash Rf system chromatogram of 40 g column using a regular RediSep Rf column (top), irregular silica (middle), and RediSep Rf Gold spherical column (bottom)

CombiFlash, RediSep, and Companion are registered trademarks of Teledyne Isco, Inc. All other trademarks are the property of their respective holders.

Last modified March 20, 2009

Teledyne Isco, Inc.

P.O. Box 82531, Lincoln, Nebraska, 68501 USA
 Toll-free: (800) 228-4373 • Phone: (402) 464-0231 • Fax: (402) 465-3091
 E-mail: iscoinfo@teledyne.com