

Resolving Minor Compounds with RediSep Gold® Columns

Flash chromatography purification using smaller, spherical particles

Chromatography Application Note
AN71

Abstract

In drug discovery, it is advantageous to collect compounds synthesized in side reactions from desired compounds. These minor compounds are collected for the purposes of screening and patent protection. Pharmaceutical companies are concerned that similar compounds may show activity in a therapeutic area and should be separated from the desired compound. The minor compounds may show activity similar to the desired material. Collection of minor compounds is also important to demonstrate that these materials do not affect the efficacy or toxicity of the pharmaceutical product. Minor compounds are often difficult to resolve from the main compounds since they are generally an isomer of the main compound.

RediSep Gold columns use a smaller particle size that enhances resolution, while minimizing increases in back pressure due to the spherical particle shape. Proprietary end user purifications are used as examples.

Results and Discussion

Resolution is proportional to the square root of the number of theoretical plates (N). $N \approx 1/d_p$, where d_p is the average particle size. The RediSep Gold column derives its improved resolution from smaller particle sizes which increases the number of theoretical plates. This enhanced resolution allows easier collection of minor compounds.

Table 1: Run Conditions for Example 1

Column size:	40 g
Load:	500 mg (1.1% loading)
Solvents:	Hexane and Ethyl Acetate
Gradient:	0-100%
Flow rate:	40 mL/min
Run time:	23 min.
Wavelength:	254 nm

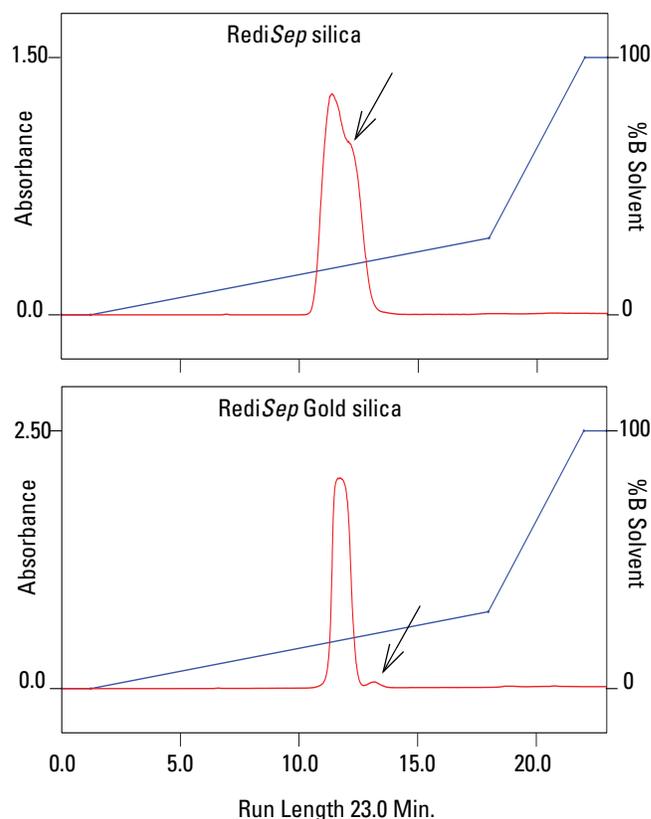


Figure 1: CombiFlash system chromatogram of a 450 mg separation using a RediSep silica column (top) and a RediSep Gold silica column (bottom). Arrows denote minor compound.

Near baseline resolution was obtained with the RediSep Gold silica column. The ΔR_f between the two compounds was 0.1 on TLC.

The second example is again provided courtesy of the end user. Here, two minor compounds can be purified from the main peak, compared with only one on a conventional Flash column.

Table 2: Run Conditions for Example 2

Column size:	40 g
Load:	500 mg (1.1% loading)
Solvents:	Hexane and Ethyl Acetate
Gradient:	20%
Flow rate:	40 mL/min
Run time:	19 min.
Wavelength:	254 nm

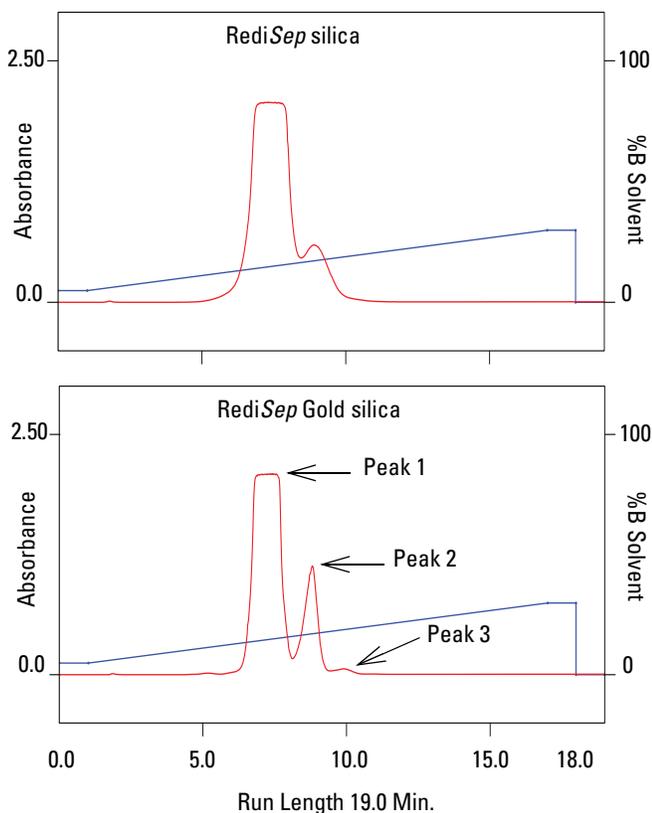


Figure 2: CombiFlash system chromatogram of a minor compound separation using a RediSep silica column (top) and a RediSep Gold silica column (bottom)

Conclusion

RediSep Gold columns, packed with a smaller, spherical media provide higher resolution than conventional Flash columns without the higher back pressures. This enhanced resolution can be used to purify minor compounds, which may be screened for activity.

Table 3: RediSep Gold Silica Columns

Part Number	Description
69-2203-344	RediSep Gold Silica Disposable column, 4 gram, pkg. of 14
69-2203-345	RediSep Gold Silica Disposable column, 12 gram, pkg. of 14
69-2203-346	RediSep Gold Silica Disposable column, 24 gram, pkg. of 10
69-2203-347	RediSep Gold Silica Disposable column, 40 gram, pkg. of 10
69-2203-348	RediSep Gold Silica Disposable column, 80 gram, pkg. of 6
69-2203-349	RediSep Gold Silica Disposable column, 120 gram, pkg. of 6
69-2203-359	RediSep Gold Silica Disposable column, 220 gram pkg. of 4
69-2203-369	RediSep Gold Silica Disposable column, 330 gram, pkg. of 3
69-2203-427	RediSep Gold Silica Disposable column, 750 gram, pkg. of 3
69-2203-428	RediSep Gold Silica Disposable column, 1.5 kg, pkg. of 2
69-2203-529	RediSep Gold Silica Disposable column, 3.0 kg, pkg. of 1
69-2203-921	RediSep Gold Silica Disposable column, 7.0 kg, pkg. of 1

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