

# INCREASED SAMPLE PURITY WITH HIGH RESOLUTION FLASH CARTRIDGES

The demand for fast and easy purification techniques is increasing.

Flash Chromatography can be utilised to achieve quick preparative separations - however, increased resolving power is often desired to purify target compounds from closely eluting contaminants. To satisfy the need for flash cartridges with better resolution and higher loading capacity, high-resolution flash cartridges have been developed such as GraceResolv™ from Grace Davison Discovery Sciences.

The silica packed in these newer cartridges has been specifically designed to increase cartridge efficiency (plates/meter) and thereby increase resolution of closely eluting compounds. This results in an increase in the target compound recovered at the desired purity level. The increase in resolution also enables a significant increase in loading capacity, over two times greater in some cases, versus traditional flash cartridges. By purifying greater amounts on a given cartridge size, users can reduce purification costs and increase productivity.

In order to demonstrate the advantages of higher resolution flash cartridges we compared GraceResolv™ cartridges to standard 50µm flash chromatography cartridges. Experiments were designed to test sample resolution, loading capacity, recovery, and throughput.

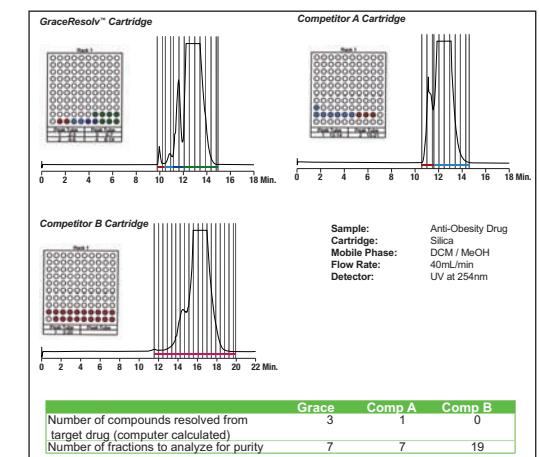


Figure 1. Stage 1 Anti-obesity compound purified from synthetic byproducts (Data courtesy of Cambridge Biotechnology Ltd, part of the Biovitrum Group).

## RESULTS AND DISCUSSION

Figure 1 demonstrates the ability of the GraceResolv™ cartridge to deliver superior resolution of closely eluting impurities, thereby separating two additional impurity compounds from the target drug. The main impurity is better resolved when compared to standard 50µm silica cartridges.

This can dramatically increase the yield of high purity fractions as demonstrated in Figure 2 which shows

## Chromatography Focus

Flash chromatography, also known as medium pressure chromatography was popularised several years ago as an alternative to slow and often-inefficient gravity-fed chromatography.

Today, flash chromatography has advanced considerably by introducing automated instruments using pre-packed flash chromatography cartridges thereby eliminating the time-spent manually packing silica into glass columns. The one area of flash chromatography that has seen little advancement is the heart of the separation, the silica.

Most cartridges today are packed with the same 40-60µm irregular shaped silica that was used for self-packed glass columns in the 1970s. The benefit of this type of silica is that it is inexpensive and generates low backpressures, however it only provides a marginal level of resolution.

Silica producers are now applying new technologies to the field of flash chromatography. For example, the silica incorporated into the GraceResolv™ cartridges is not only smaller in particle size (centered on 40µm), but also narrower in particle size distribution than the standard flash silica traditionally packed in flash cartridges.

This smaller particle size increases the efficiency of the cartridge resulting in higher resolution, while the narrow particle size distribution (Figure A) reduces fines (small particles less than 5µm), which keeps backpressure low.

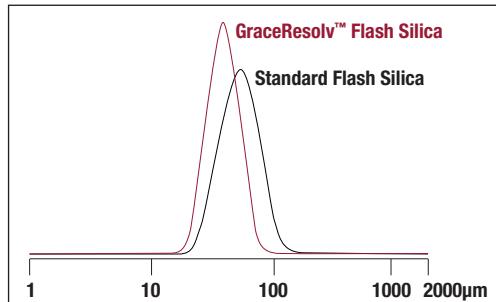
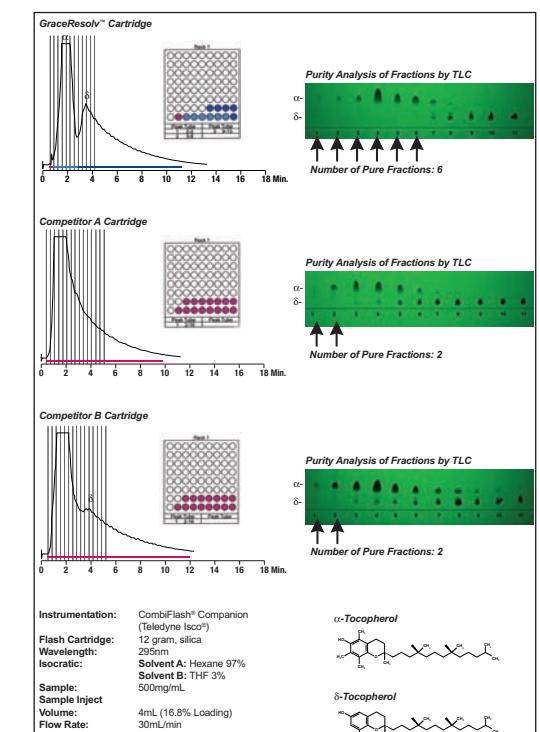


Figure A. A smaller particle size increases resolution. Tighter particle size distribution and minimal fines (small particles) keep backpressure low. Measured by Malvern® Mastersizer® 2000.

the purification of alpha from delta tocopherol. This increase in resolution also allows for higher loading capacity while maintaining base-line separation.

Chemists can then use smaller cartridge sizes to purify the same amount of material that would typically be purified on larger standard cartridges leading to a reduction in costs from the smaller tubes, the solvent used and the solvent disposal. (Figure 3/Table 1).

The combination of higher resolution and loading capacity can alternatively enable chemists to increase flow rate whilst still maintaining baseline resolution, therefore increasing throughput and saving time (Figure 4).



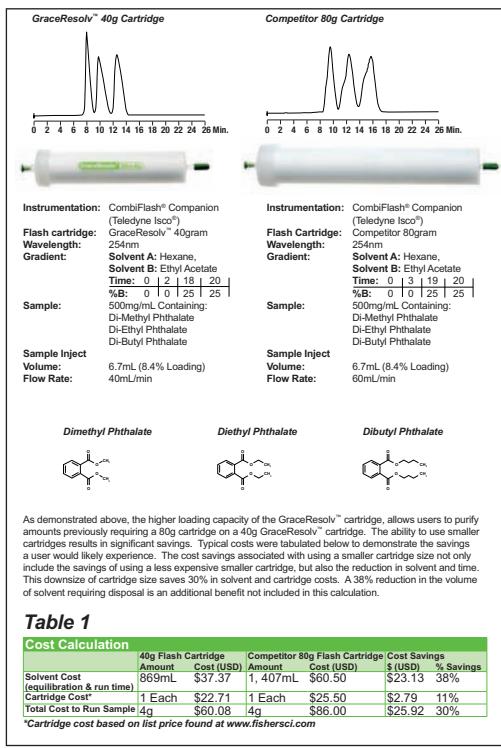


Figure 3. Loading comparison of a 3.35g Phthalate mixture: GraceResolv™ 40g cartridge vs Competitor 80g cartridge.

## CONCLUSION

The GraceResolv™ higher resolution flash cartridges are packed with specially developed silicas that result in superior resolution and loading capacity when compared to the traditional 50µm cartridges.

This leads to the following advantages for the chemist:

- Significant increase in pure compound recovered
- Increased productivity via Faster purification without losing baseline resolution
- Reduction in overall costs – less solvent, less waste

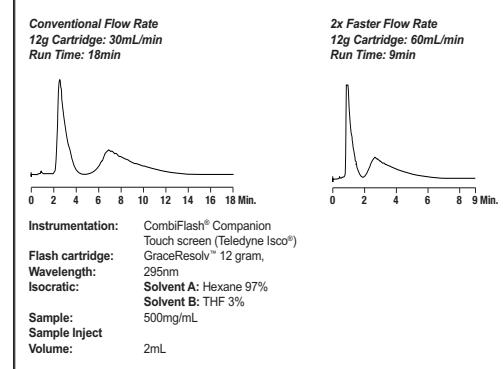


Figure 4. Flow rate comparison in the purification of  $\alpha$  from  $\delta$ -tocopherol.

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## ADVERTORIAL

### The Safe Hydrogen Carrier Gas Solution for GC



The benefits of switching GC carrier gas from helium to hydrogen such as faster analysis and improved resolution have long been understood.

Now, financial motives driving the change are gaining momentum due to the rising market price of helium amid supply shortages and the rising costs, both financial and environmental, of cylinder deliveries. Unfortunately, concerns with heightened safety risks when using hydrogen via high pressure cylinders cannot be completely allayed.

But now, the Parker domnick hunter H-MD hydrogen generator with highly sophisticated software control and alarm capabilities presents the GC user with the opportunity to reap all the benefits offered by hydrogen, at an affordable price without the need to worry about safety and environmental factors associated with cylinder-based supplies. H-MD generators are available in three models to deliver varying flow rates of ultra high purity hydrogen (1 ppm total contaminants) to GC and GC/MS carrier gas and fuel gas applications. Sizes include 160, 250 and 500 ml/min.

### One Size Does Not Always Fit All. Bespoke LC/MS Nitrogen Generators Facilitate Better Analysis

Modern LC/MS instruments use high flows of nitrogen for nebulisation and drying in the interface. However, the gas demands of different interfaces vary extensively depending on design, with some instruments even employing multiple gases. One standard gas supply therefore may not necessarily meet all the demands of the instrument. Parker domnick hunter however offers the widest range of LC/MS nitrogen generators which most effectively and efficiently match instruments' gas requirements. Bespoke models, supplying multiple gases, have been fully researched, designed and developed specifically for use with Agilent, Varian, Bruker and now Applied Biosystems LC/MS instruments to offer optimum performance to the laboratory user.

The AB4000TGS and AB5000TGS models have been recently designed in close collaboration with Applied Biosystems to provide the three gas supplies required for their API range of instruments from a compact, low noise and low vibration unit. The LCMS6410 also represents the first all-in-one gas solution for Agilent's 6410 triple quadrupole and 6510 Q-TOF LC/MS instruments. All Parker domnick hunter nitrogen generators employ robust and totally regenerative carbon molecular sieve technology to guarantee optimum performance, durability and lifetime cost effectiveness. A range of tailored, preventative maintenance contracts are also available via a dedicated global aftermarket network which, in turn, offers complete peace of mind.

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The benefits of using hydrogen as a GC carrier gas are proven; faster analysis, improved resolution and longer column life. And, with the price of non-renewable helium increasing at a rapid rate, hydrogen is now a more cost-effective and environmentally friendly choice of carrier.

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