Simplify Flash Chromatography and Organic Chemical Purifications using Strata™ Giga Tubes

Patrick Boyce
Phenomenex Limited, Macclesfield, UK

Introducing Strata Giga Tubes

For many years, synthetic chemists have packed silica into glass columns to run gravity and flash chromatography. Over the past ten years, the use of pre-packed, disposable columns has become increasingly popular. Phenomenex offers a complete line of pre-packed syringe barrel tubes for flash chromatography.

- Selection: available with reversed, normal and ion exchange sorbents. The 12, 20 and 60mL tubes are packed with a bed mass ranging from 500mg-20g.
- Quality: packed with high purity sorbents, which reduces peak tailing of acidic and basic compounds. The very narrow particle size distribution leaves less interstitial volume and improves peak width, leading to better resolution.
- High analyte capacity: high and reproducible separation yields of samples ranging in size from milligrams to grams.
- Fast and simple: three easy purification steps that requires about 20-30 minutes.
- Convenience: pre-packed, single use tubes that are ready to be used. You won't be handling loose sorbent to pack your own tubes. These tubes can be easily processed with a home made apparatus, the Argonaut FlashMaster system, or the Phenomenex vacuum manifold.

How to use Strata Giga Tubes

There are three simple steps: condition, load, and elute. Detailed practical guidelines are presented overleaf, but first let's look at each of these steps in detail.

1. Conditioning

This step eliminates of air from the cartridge, ensuring your sample has full access to all the stationary phase. The conditioning solvent should have weak eluting strength. For instance, Strata Si-1 silica columns are conditioned with non-polar solvents such as hexanes.

2. Sample Loading

Ideally your sample should be retained on the top of the column in a tight band. Two important practical points: first, if excess sample is loaded, it will bleed through the column length, leading to what chromatographers call peak distortion. This will reduce the yield of purified material. Secondly, the type and volume of sample solvent must be carefully selected. In the case of silica columns polar organic solvents, like methanol, will prematurely elute your sample. If possible, load your sample in a non-polar (weak eluting strength) solvent such as hexanes.

What if your sample can only dissolve in a polar solvent? The ability of the column to handle a strong solvent is improved by switching to a larger mass of sorbent. In other words, if you were considering using a 5g silica tube, but have to load your sample in methanol, peak distortion can be minimized if a 10g or 20g tube is used instead. An alternative strategy would be to select a different sorbent chemistry. For instance, SCX, NH2 or SAX sorbents allow you to load your sample in very polar solvents. These are particularly useful for the extraction of amines or acidic compounds.

3a. Elution - What is the optimal flow rate?
If the mobile phase is passed through the tube too fast, you will not allow your sample, or the eluting solvent to equilibrate properly with sorbent, and therefore the resolution will suffer. On the other hand, if the mobile phase passes through the tube too slowly, the compound(s) will start to diffuse through the sorbent, and again lowering the resolution. The optimal flow rate will depend on the diameter of the tube. Optimum flow rates are given overleaf.

3b. Elution - How long should you run a column?
Chromatographers normally aim for a capacity factor between 2-10. In brief, the capacity factor is a measure of how many column volumes will be required to elute the compounds of interest. Capacity factors <1 (the compound elutes with less than 2 column volumes) indicates your sample hasn't effectively interacted with the sorbent, and therefore you get little resolution. For capacity factors between 2-10, resolution increases significantly. Above 10, (your compound elutes with >11 column volumes) any incremental increase in resolution is offset as peaks become wider.

Flash Chromatography on Strata Giga Tubes

Sorbent	Strata Si-1	Strata SCX	Strata NH_2 & SAX
Compound class	Neutrals, Acids, and Bases	Bases	Acids
Recommended Flow rate (use within 33% of these values)	12mL tubes: 10mL/min 20mL tubes: 14mL/min 60mL tubes: 28mL/min	12mL tubes: 10mL/min 20mL tubes: 14mL/min 60mL tubes: 28mL/min	12mL tubes: 10mL/min 20mL tubes: 14mL/min 60mL tubes: 28mL/min
Column conditioning (use 1 column volume)	hexanes (or nonpolar solvent)	same as sample solvent	same as sample solvent
Selecting the sorbent mass	For difficult separations (similar R_f values by TLC), load no more than 1% by mass of the sorbent bed mass. (e.g. purify 50mg of crude material using a 5g or larger bed mass.) For separations where there is a large difference in R_f value by TLC, up to 5% loading can be reached (e.g. purify 250mg using a 5g sorbent.	Load no more than 1-5% by mass of the sorbent bed mass. (e.g. purify 50mg-250mg of crude material on a 5g sorbent mass.) Weak amines such as aromatic amines will be less loadable than strong amines.	Load no more than 1-5% by mass of the sorbent bed mass. (e.g. purify 50mg-250mg of crude material using a 5g bed mass). Weak acids will be less loadable than strong acids. Consider using SAX for weak acids.
Sample solvent	Preferably non-polar (e.g. hexanes). If a polar solvent is needed for solubility, use a larger tube and load less than 1% of the sorbent bed by mass with a gradient mobile phase.	Any (spike with 0.1% acetic acid to ensure bases are protonated)	Any (note: if it is a weak acid, use SAX instead of NH_2 and spike with ammonia)
Isocratic mobile phase	Use the solvent system that gives R_f values between 0.15 and 0.35 on silica TLC plates.	Elute acids and neutrals with 1-3 column volumes of methanol. Elute all amines with 1-2 column volumes of 10% ammonia in methanol.	Elute bases and neutrals with 1-3 column volumes of methanol. Elute all acids with 1-2 column volumes of 10% ammonia in methanol, or 5% formic acid in methanol (SAX).
Gradient mobile phase	Take the solvent system that gives R_f values between 0.15 and 0.35 on silica TLC plates. Divide the amount of polar solvent in this mobile phase by 4. Run a gradient from this new value of polar solvent to 100% over 10 column colums.	Elute acidic and neutral compounds with 1-3 column columes of methanol. Elute different amine classes with 2 column volumes of 0.1% ammonia in methanol, followed by 2 column volumes of 0.5% ammonia in methanol, followed by 2 column volumes of 2% ammonia in methanol, followed by 2 columns volumes of 10% ammonia in methanol.	Elute amines and neutrals with 1-3 column volumes of methanol. Elute different acid classes with 2 column volumes of 0.1% formic acid in methanol, followed by 2 column volumes of 0.5% formic acid in methanol, followed by 2 column volumes of 5% formic acid. For NH_2 cartridges, formic acid can be subsituted with ammonia.
Generic gradient	Solvent A: hexanes + 0.1% diethylamine. Solvent B: 80/20 dichloromethane/ methanol + 0.1% diethylamine. Run a gradient from 100% A to 90% B over 10 column volumes.	As above.	As above.
Run time	5 to 10 column volumes	5 to 10 column volumes	5 to 10 column volumes
What is a column volume?	1 column volume (mL) = 2 x mass of of sorbent (g)	1 column volume (mL) = 2 x mass of of sorbent (g)	1 column volume (mL) = 2 x mass of of sorbent (g)

Questions? Please contact your Phenomenex Technical Representative

Ordering information

	500mg/12mL	2g/12mL	5g/20mL	10g/20mL	10g/60mL	20g/60mL
Strata Si-1 Silica	8B-S012-HDG	8B-S012-KDG	8B-S012-LEG	8B-S012-MEG	8B-S012-MFF	8B-S012-VFF
Strata SCX	8B-S010-HDG	8B-S010-KDG	8B-S010-LEG	-	8B-S010-MFF	8B-S010-VFF
Strata SAX	8B-S008-HDG	8B-S008-KDG	8B-S008-LEG	-	8B-S008-MFF	8B-S008-VFF
Strata NH_2	8B-S009-HDG	8B-S009-KDG	8B-S009-LEG	-	8B-S009-MFF	8B-S009-VFF

Other Strata phases are available in the Giga Tube format. Contact Phenomenex for more information.

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This method is designed as a convenient starting point for further investigation.
Phenomenex makes no guarantee regarding the accuracy or completeness of the method.