Flash Chromatography Products

TELOS®

Separation of Basic Organic Compounds using TELOS Flash NH2 Columns – without a modifier

The Challenge

Basic organic compounds, in particular primary, secondary or tertiary amines, do not chromatograph as sharp symmetrical or discrete peaks when using silica flash columns – the compounds exhibit poor chromatography, often tailing or eluting very slowly from the column when using standard normal phase eluents such as cyclohexane/ethyl acetate or DCM/methanol. Some basic compounds do not elute at all under such conditions from silica.

The Common Practice

The addition of a small amount of amine (0.1 - 2% v/v), e.g. TEA (or ammonia), to the mobile phase is a common way to improve the chromatography and elute these compounds from silica. The drawback with this approach is threefold: (a) the modifier has to be removed from the collected fractions (hydrolysis can be a problem during evaporation), (b) the retention time of the compounds of interest is greatly reduced and (c) the loading capacity of the flash column is reduced compared to a method without modifier.

Our Approach

TELOS Flash NH2 Columns are packed with aminopropyl modified silica gel (bonded phase). The immobilised amino group provides a slightly basic surface (pK_a value of the bonded moiety is ~9.8) and acts similar to a "dissolved modifier" in the mobile phase, displacing the basic compounds from the silica gel surface. The surface of the chromatographic support is slightly basic, with basic compounds "rejected" (or repelled) from the surface. Their interaction with the silanols, and therefore their retention is decreased in a controlled and reproducible way.

Developing a Method on TELOS Flash NH2

Kinesis provide TELOS NH2 TLC Plates allowing a reaction mixture "pre-test" with common solvents and solvent systems (cyclohexane, ethyl acetate, DCM and methanol etc). The plates use the same NH2 media as the columns, allowing easy and rapid transfer of the selected solvent system. Adjust the mobile phase to ensure the separation occurs within the R_f range: 0.1 – 0.4. Calculate $1/R_f$ for the main compound. This will provide the number of bed volumes required to use to elute the compound using an isocratic method.

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Example:

- R_f = 0.25 with 5/95 v/v methanol/DCM
- $1/R_f$ gives 4 bed volumes
- A TELOS Flash NH2 12g Column has a bed volume of ~14ml
- Elution of the required compound will start after ~48ml of mobile phase is pumped through the column

We recommend isocratic systems should not be used as standard; linear gradients provide faster separations and allow the use of the high performance and capacity of TELOS Flash Columns. Start with few minutes of a non-eluting mobile phase (using the example above, start with 100% DCM (or DCM with 0.5% v/v methanol), then run a linear gradient to 5% v/v methanol using ~10 bed volumes. Finally, continue with these conditions for a further 3 - 5 column volumes. If necessary, increase the methanol concentration further to remove all compounds from the flash column.

It is recommended TELOS Flash NH2 Columns are not re-used; they will be deactivated when using polar elution solvents.

Sample Loading

Samples can be applied pre-adsorbed using TELOS NH2 or NM (diatomaceous earth) bulk media using a TELOS Dry Loading Cartridge or directly onto a syringe barrel (SPE) type column. Do not use standard silica to dry load the sample - it will delay elution onto the main separation column, causing poor separations/chromatography.

For liquid injections, dilute the sample (after dissolving in a small volume of a polar solvent) with a non-polar solvent to ensure a peak compression effect during the injection/sample loading process.

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